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Assessment of mechanical properties of metallic thin-films through micro-beam testing

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ABSTRACT

Microelectronic industry is driven by the continuous miniaturization process conducing to the introduction of materials with better performance. These materials are subjected to stresses mainly due to thermal mismatch, microstructural changes or process integration which can be in the origin of mechanical reliability issues. To study these phenomena and even electromigration a good mechanical characterization of the materials is needed. This work aims at developing tests to assess fracture and elastoplastic behavior of thin Cu films. The tests developed are based on the deflection of microbeams (micromachined using a focused ion beam) using a nanoindenter. Different test geometries for microbeams have been evaluated and quantitative data have been obtained combining experimental results with analytical or numerical models, depending on the property under study. Microbeam response shows a strong dependence on the orientation of the grains close to the fixed end. Grain orientation has been measured by electron backscatter diffraction and the plastic behavior has been modeled by the finite element method using an in-house crystal plasticity subroutine. The effect of film thickness on fracture energy has been determined from tests of notched beams.

1. Introduction

As the microelectronic industry advances, the materials used in integrated circuits are mainly optimized from their electronic and optic point of view but the mechanical properties cannot be disregarded. As residual stresses, that appear as a consequence of the deposition processes, and stresses during packaging and during in-service thermal cycling increase [1–3], the low-*k* dielectric materials [4,5], with poor mechanical properties, can compromise the reliability of the chip. Therefore many different techniques have been developed to characterize the mechanical properties of the thin films and the properties of their interfaces [6], but these techniques have severe limitations as the thickness of the thin films is reduced.

Four-point bending (introduced by Charalambides et al. [7] and developed and extensively used by the group of Dauskardt [8,9]) and Cross Sectional Nanoindentation [10–12] are tests currently in use in the industry for the characterization of adhesion of interfaces between different layers. Concerning cohesive fracture, several efforts have been made. “Channel cracking”, developed by Huang et al. [13], is a technique extensively used in industry. Indentation techniques have also been used to measure fracture toughness of brittle thin films [14,15].

As for the characterization of ductile materials, many different techniques have been investigated. Bulge test, proposed by Vlassak and Nix [16] to calculate the Young's modulus and residual stresses of thin films, was used by Xiang et al. [17] to study the mechanical behavior of freestanding Cu thin films of different thicknesses and the influence of microstructure on the yield stress.

Tensile tests have also been used to characterize the mechanical response of small volumes. For instance, Kiener et al. [18] developed in situ experiments of miniaturized single-crystal copper samples to investigate the size effects on the plastic deformation behavior. Works developed by Keller et al. [19] and Hirakata et al. [20] are examples of the use of micro-tensile tests to obtain information on the elastic–plastic behavior and fracture energy of Cu thin films.

Testing of micro-pillars has been widely used for the characterization of plastic behavior of metals at the micro- and sub-micro scale. A comprehensive review of some of the most relevant results obtained with this technique, with a discussion on the combined effects of intrinsic (i.e. microstructural) and extrinsic (i.e. sample size) sizes on the material deformation behavior was presented by Greer and De Hosson [21].

Bending of cantilever beams is an alternative test for material characterization. For instance Matoy et al. [22] used this technique to calculate the fracture toughness of silicon-based dielectric materials and found that toughness increased with decreasing cantilever thickness.

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In this work, the ability of microbeam testing to assess the mechanical response (elastoplastic behavior and fracture) of metallic (copper) thin films has been explored. Microsamples, machined samples using focused ion beam, have been tested using a nanoindenter. The advantage of this approach is that the Cu tested has the microstructure, and hence the mechanical properties, obtained from the actual process. A significant scatter in the load–displacement records is observed that has been linked to the different crystallographic orientations of the beams tested. The use of crystal plasticity implemented in a commercial finite element (FE) code makes it possible to assess the intrinsic mechanical properties of the tested Cu films. Fracture energy has been calculated from results obtained testing notched cantilever beams.

2. Materials and experimental procedure

The materials studied in this work are Cu blanket thin films (0.5 and 1 μm thick) deposited (electroplated on a seed deposited by PVD) on a {100} Si wafer. The beams have been machined using a dual beam focused ion beam QUANTA3DFEG, FEI.

Starting from small pieces (about 10 mm × 10 mm), cleaved from a wafer, a procedure for the beam machining has been established. In a first step, the top geometry of the beams is defined (Fig. 1a) using ion currents decreasing from 7 nA (rough machining) to 1 nA (finishing), to get a smooth surface. Positioning marks, which will help the positioning of the tip when testing each beam at the nanoindenter, are also machined in this step. In a second step, the sample is machined from its cross section to define the thickness of the beams and to leave some space under the features allowing for the beam deflection in the test. Fig. 1b shows a detail of the second step and the final geometry typically obtained. To assess fracture behavior, a third step is needed: a lateral notch is machined from the top at the fixed end (Fig. 6).

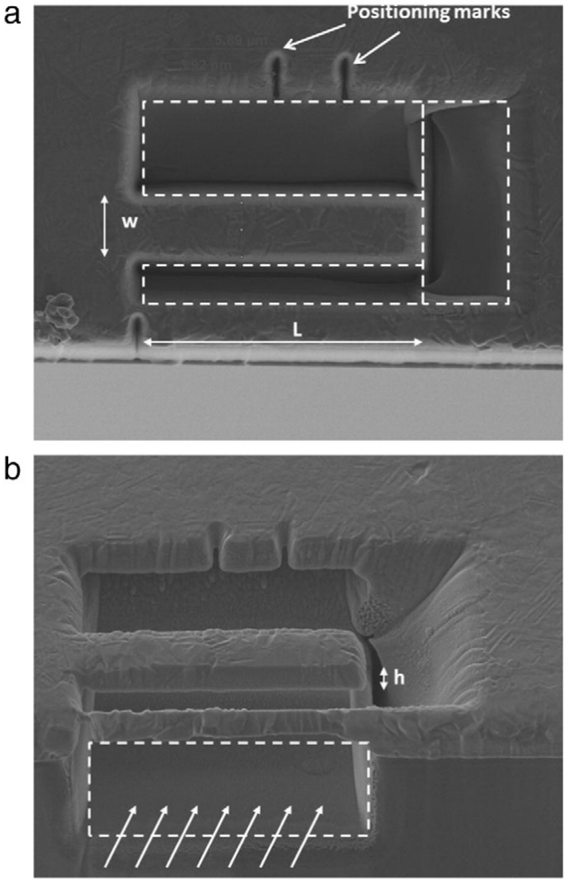

Fig. 1. SEM images showing details of the first step of the machining process for cantilever beams. The white boxes and arrows sketch the material removal process. a) Top view, showing the first step defining the width (w) and length (L) of the beam; b) image taken at 45° showing the second step defining the beam thickness (h) and the final geometry.

The microstructural analysis of the Cu film showed a columnar structure with an average grain size of about 1 μm. The grain orientations of Cu in the tested beams have been measured before testing by electron backscatter diffraction (EBSD) in a Jeol JSM-7000F microscope equipped with a HKL system.

The beams are tested using a TriboIndenter™ (Hysitron, USA) with a conical tip (tip radius 1.86 μm). In the testing process, the correct positioning of the application point of the load is of paramount importance. Hence a scanning probe microscopy (SPM) image of the beam is taken with the imaging mode of the TriboIndenter™. Once the aimed point is in the center of the SPM image, the test is carried out, assuring accuracy in the measurement of the effective length, L, of the beam in the tens of nanometers. All the samples are tested under displacement control, and after being deflected, a post test analysis is performed in the scanning electron microscope (SEM) to determine the final deflection and, in the case of notched samples, the propagation of the crack.

3. Results and discussion

3.1. Elastoplastic behavior

In order to study the elastoplastic behavior of 1 μm Cu thin film, beams were machined as described above and tested from the top. In all the cases, the resulting thickness was slightly smaller than 1 μm, making sure that the beams were made only of Cu. Fig. 2 shows the load–displacement curves obtained for one of the sets of beams tested and Fig. 3 shows an example of one of the beams before and after the test, where the permanent deformation introduced is apparent. A significant scatter in the response of the different beams can be noticed which cannot be explained in terms of the small geometrical differences among them (all the beams are about 1.7 μm wide, 1 μm thick and are loaded at 6 μm from the fixed end).

As mentioned in the previous section, the average grain size measured in the samples is about 1 μm. Consequently, the tested beams have 1–2 grains along the width in the fixed end, and a few more grains in the whole length, being the response of each feature strongly dependent on the crystallographic orientation (measured by EBSD) of the grains. Using the measured microstructure as an input, a FE model of each beam has been built. The actual microstructure has been replicated assuming columnar grains, that is, the grains as measured at the top surface are extruded through thickness as indicated in Fig. 4.

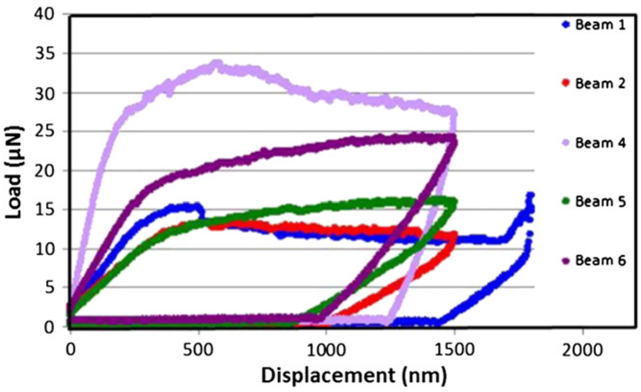

Fig. 2. Load–displacement records of 6 beams corresponding to the same lot. All of them have similar dimensions (1.7 μm × 6 μm × 1 μm) and the scatter in the mechanical response is apparent.