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Scripta Materialia 52 (2005) 873–879

Scripta MATERIALIA

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Intrinsic stress development and microstructure evolution of Au/Cr/Si multilayer thin films subject to annealing

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Received 5 October 2004; received in revised form 21 December 2004; accepted 5 January 2005
Available online 26 January 2005

Abstract

Au/Cr/Si microcantilevers were studied in their as-deposited condition and annealed state, with emphasis on a thermal treatment of 225 °C for 24 h. Change in beam curvature was monitored during isothermal hold as a function of time. Secondary grain growth was observed in the gold, which contained non-uniformly distributed twins and dislocation defects. Diffusional transport of the chromium layer was observed during annealing. Nodules arranged in a "rolling hill" topography were observed at the free surface, both before and after annealing. Nanometer thick coatings of alumina grown by atomic layer deposition improved the uniformity of both microstructure evolution and curvature evolution during high-temperature annealing.

Keywords: Thin metal films; Reliability; Material stability; MEMS

1. Introduction

Multilayer Au/Cr/Si structures were originally researched for use in hybrid (high frequency) circuits [1]. Such multilayer (multimorph) structures are also common in microelectromechanical systems (MEMS). Proposed applications include: reflective optical structures such as mirrors [2], optical detectors [3], DC electrical relays and contacts [4], RF or high frequency components including switches [5] or variable capacitors [6], chemical or biological sensors [7,8], and vertical [9] or lateral [10] actuators. In some instances, basic multimorph diagnostic structures might also prove applicable to understanding single layer structures [11], such as gold metallic traces or gold interconnect wire bond pads.

While early research focused on the electrical characteristics of Au/Cr/Si multilayers, there is still limited understanding of their mechanical and morphological stability, i.e. the long-term performance and reliability of such structures. The metallic layers often exist having a limited thermal history, i.e. the as-deposited structure, as necessitated in microsystems applications. Multimorph structures are, however, typically exposed to thermo-mechanical loading during their fabrication, subsequent post-processing, and application environment. Therefore, a single thermal cycle is often sufficient to invoke significant change in the shape of metastable gold thin film based structures [12]. Similar inelastic behavior has been observed in aluminum [13] and copper [14].

The curvature evolution for a multilayer beam subject to a thermal ramp, isothermal hold at maximum temperature, followed by return to room temperature appears in Fig. 1. The profile demonstrates that a

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doi:10.1016/j.scriptamat.2005.01.004

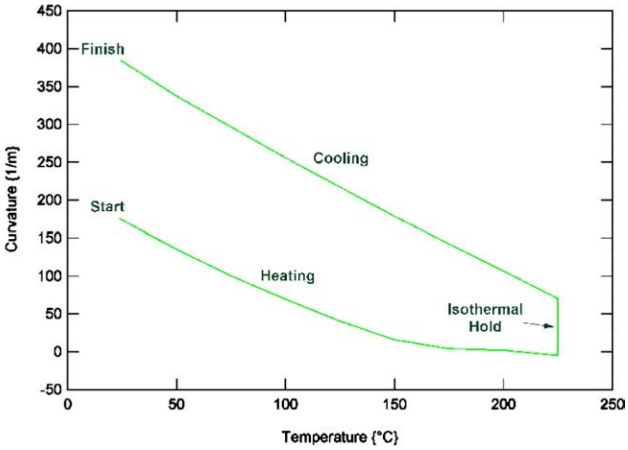

Fig. 1. Measurement of curvature for a microcantilever (polySi = 3.5 µm thick) subjected to an initial thermal cycle, with isothermal hold for 4 h at maximum temperature of 225 °C.

significant change in curvature (i.e. shape) exists when the specimen is heated to an elevated temperature and then returned to the ambient temperature. The curvature of a cantilever beam is in general proportional to the stresses in its component layers. The development of intrinsic stresses is believed to be related to changes in the microstructural evolution within the metals. The thermally activated inelastic mechanical change invoked during annealing also depends on time [16].

The thermo-mechanical behavior of multilayer microcantilevers often differs from that in traditional thin films owing to the comparable thickness of the various layers and the resulting magnitude and non-uniformity of stress distributions. The relatively large displacement, large curvature, and smaller stress in microcantilever devices makes them different from conventional thin film studies performed using metals on “thick substrates”. Their size renders microcantilevers extremely sensitive to microstructural scale phenomenon, which will serve to invoke readily observable changes in curvature. Also, for example, the popular Stoney equation [17], used to approximate a single stress value when a thick substrate is used is not valid for microcantilevers, which require a different mathematical representation.

Understanding of the complex behavior observed at elevated temperature motivated the present work, which focuses on the change in microstructure and morphology in the gold and chromium layers on thin-film polycrystalline silicon (polySi) microcantilevers. In particular, the beams demonstrate the development of constrained “intrinsic” stresses, which result from changes in the material microstructure rather than a change in external loading (applied stress) or temperature (thermal stress). Film morphology, chemical composition, and defect structure were studied in both the as-deposited state as well as after annealing at 225 °C. The goal of the research was to identify and correlate changes in morphology and microstructure to observed changes in curvature. To our knowledge this is the first study conducted to relate the microstructure evolution of metallic layers to the change in shape (curvature) of a multilayer microcantilever structure subject to thermo-mechanical loading.

2. Experimental

Multimorph cantilever beams were fabricated using the Multi-User MEMS Process (MUMPS), provided by the MEMSCAP Corporation [18]. Interposed arrays of fixed-free beams were fabricated having the width of 20 µm and length ranging from 80 to 280 µm, in 40 µm increments. After being annealed at 1050 °C, beams of polySi, either 1.5 or 3.5 µm thick, are coated on top with a gold film that is 0.5 µm thick. Gold is deposited using e-beam evaporation, with the substrate (host) wafer being unheated. Adhesion is promoted by using a 20-nanometer thick chromium layer located between the gold and polySi. Some beams were conformally coated with alumina using the atomic layer deposition (ALD) process [19], which can be used to grow material incrementally in monolayer thickness.

Separate MEMS chips featuring arrays of multilayer microcantilevers were individually placed within an INSTEC STC200 thermal chamber, capable of rapid thermal cycling. Duration time between automatically controlled steady state temperatures typically lasted for 1–3 min, depending on the size of the step between the temperatures. Full-field topography measurements were made in situ as a function of temperature and time using a white-light interferometric microscope. A Michelson type objective was used to perform measurements through a quartz window in the chamber.

The ZYGO New View 200 interferometric microscope used for shape characterization is capable of accurate surface profiling. For the microcantilevers studied, the measurement accuracy is estimated to be better than 3%. The full-field topography profiles were converted to curvature by fitting a second order polynomial to a user-specified one-dimensional cross-section of the scan and then taking the second derivative of the polynomial-fit. The sign convention assumed for the measurements is such that when the free end of the beam deflects upwards, away from the substrate, a positive curvature value will exist.

Transmission electron microscopy (TEM) was performed using an analytical Philips CM200 operated at 200 kV. Thin film specimens used for TEM work consisted of larger mechanically fixed MUMPS plate specimens and not the diagnostic microcantilever beams examined using other instrumentation. First, the free surfaces on the specimens were coated with platinum