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Mechanical and tribological properties of thin films under changes of temperature conditions

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ABSTRACT

Direct measurements of tribological and mechanical properties of thin films are important to predict the life-time of micro/nano-devices using thin films as a protective layer in order to reduce the stiction, adhesion and wear. This paper presents the analysis of mechanical and tribological properties of thin films at different temperatures using atomic force microscope (AFM) and nanoindentation. A thermal stage is used to control the temperature of investigated samples in the range of 20 °C to 100 °C. Thin films as SiO_2, polysilicon and dielectric films as Si_3N_4 are deposited on different substrates (silicon and silicon dioxide) and analyzed.

Nanoindentation is performed using a Berkovich indenter with diamond tip in order to analyze the variation of modulus of elasticity and hardness for different temperatures. Under the same indentation load, the indentation depth increases as temperature increases and the hardness and modulus of elasticity decrease, respectively. The thickness influence of these thin films on hardness and modulus of elasticity is also observed.

The aim of tribological investigations is to estimate the variation of the friction force for different temperatures using the AFM lateral mode. The adhesion force between the AFM tip and investigated thin films is measured using the spectroscopy in point of AFM. Decreasing of the adhesion force as temperature increases is experimentally observed.

Measuring mechanical properties like hardness and modulus of elasticity to evaluate their behavior at different temperatures can help designers to improve the reliability of the materials and components and to understand the strengthening and deformation mechanisms at small scales. The visco-elastic effect makes friction rate and temperature dependent. The friction forces increase as a function of temperature based on the change of the material strength. As the temperature increases, the material properties such as modulus of elasticity and hardness slowly decreased based on the material thermal relaxation.

1. Introduction

The evaluation of the mechanical and tribological properties of thin film is indispensable for designing reliable micro-nano devices, since their properties play the following roles:

- Accurate values of the tribomechanical properties are needed for obtaining the best performance;
- Because of their small size the nano devices (MEMS/NEMS) are intended to be used in harsh environments. In this case the reliability plays an important role.

The mechanical properties of micro-materials are strongly associated with the devices' performances as well as their mechanical reliability. The strong relationship between the performances and reliability exists and the reliability problem often limits the performances [1]. The mechanical shock causes two severe reliability problems in the structure of a device, which are stiction and fracture.

Stiction means that two structures bonded together and never part in controlled or unintentional contact between them. The stiction is crucial in smaller structures because it is caused by relatively large interfacial force compared to the restoring force. The control of these two forces is crucial. Since the reduction of interfacial force is done by mainly chemical processing [2], for stiction, the controls of the contact area and surfaces conditions are important [1].

Though a lot of mechanical reliability evaluations have been performed, it is difficult to discuss generalized theory about the reliability of the small structures because all of the evaluations are done by different methods and conditions. These are mainly caused by the lack of standard test methods and procedures (it is no doubt that it is difficult to establish the standard methods because of the variation of device properties).

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Various reliability evaluation methods have been employed in micro-nano systems and a lot of research papers have been reported [1–16]. However, the details for each evaluation were extremely different because the motivations of these evaluations are directed to each research's interest, for example: product development, material science etc.

To understand the reliability test structure for nano devices, they were categorized into three levels: specimen level, device level and product level [1]. Fig. 1 illustrates the schematic description of these three levels.

In this paper we have focused on the first level of test, and the test sample are different thin films deposited.

Thin films from MEMS point of view are strongly dependent on material properties, which in turn depend on the deposition conditions, film thickness, surface effects and post-process. This complexity makes it harder to know the exact material properties of a thin film without detailed information about the process condition, film dimensions and so on.

Evaluations of the mechanical and tribological properties of a micro and nano materials, used in the mechanical structures of MEMS/NEMS devices are significant.

Most often, when the mechanical and tribological properties are needed the bulk properties are adopted. However, when thin films started to be used for various mechanical structures the mechanical and tribological properties play important roles in the operation of MEMS devices. Therefore, the mechanical and tribological properties of thin film need to be accurately measured and this is our interest in this work.

There is little information regarding the dependence between the mechanical and thermal properties of thin films because the existing devices have been less demanding with respect to these properties [14]. Therefore, the most urgent need of MEMS engineers is for information about the mechanical and thermal properties of thin films [14]. This paper focuses on these properties.

The mechanical properties of thin films should be measured on the same scale as nano devices, since they are different from those of bulk materials. These differences occur for the following reasons:

- Thin film materials fairly often have different composition, structure and phase from the bulk materials, even if they are called from material with the same name. The processes of elaboration are deposition, thermal treatment, implantation and oxidation. For instance, bulk silicon nitride (a poly crystalline material) often contains impurities for improving the properties. On the other hand thin films of silicon nitride are deposited by chemical vapor deposition (are amorphous) and seldom add impurities.
- Considering the manufacturing process for bulk structure (mechanical process), it is rarely used for thin films, because is too fast for micro and nano scale (widely used are photolithography and etching). The surface finishing between two processes are complete different.
- On the other hand the size effect of the surface must be considered because the ratio of the surface area to the volume increases with the decrease in the dimension of the device structure. For instance the fracture of silicon (brittle) is initiated from surface defects and also the strength is dominated by the surface roughness.

2. Experimental details

2.1. Thin film (material) fabrication

Silicon wafers, n-type, <100> orientation has been used in order to obtain the experimental samples. After a standard cleaning in piranha ($H_2SO_4 + H_2O_2$) and 10% HF solution for 30 s at room temperature, the thin films have been deposited. Silicon oxide, polysilicon and silicon nitride, with different thicknesses, have been selected as test materials. These are ones of the most used films in micro and nanofabrication of MEMS devices.

Three different experiments were performed.

For the first samples the substrate has been thermally oxidized in order to grow the 90 nm thin film. For the second ones, the silicon nitride (Si_3N_4) obtained by LPCVD technique has been deposited from reaction between dichlorosilane ($SiCl_2H_2$) and ammonia (NH_3). For the last samples, we grew a thick silicon oxide (with 1.7 μm thickness) using thermal wet oxidation. Finely a polysilicon layer obtained by LPCVD technique has been deposited from silane (SiH_4) decomposition. The LPCVD deposition was performed at two different temperatures: 580 °C and 630 °C. In order to evaluate the mechanical and tribological properties of the polysilicon films, two different thicknesses have been chosen. The deposition (processes) method and thickness of the test materials are summarized in Table 1.

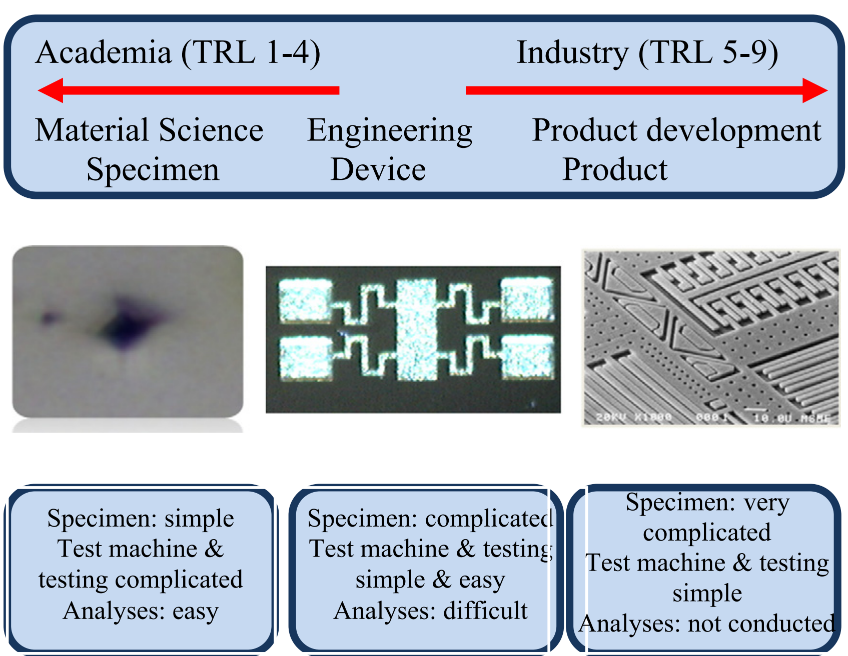

Fig. 1. Three level of reliability evaluation methods [1].