Chapter 8
Nanoindentation Measurements of Cu Films with Different Thicknesses Deposited on a Single Crystalline Si Substrate

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Abstract Thin films of various types are key components of modern microelectronics. By use of the dynamic nanoindentation method, Cu/Si structures with different thicknesses of the Cu film (t = 85, 470 and 1,000 nm) were investigated. It is shown that the film thickness and the wide range of maximum loads applied are some of the main factors influencing the deformation peculiarities and mechanical properties (Young's modulus E and hardness H) of the film/substrate structures.

Keywords Thin films • Nanoindentation • Hardness • Young modulus

8.1 Introduction

The increasing development of nanotechnology tends to reduce the component sizes of micro/nanoelectromechanical system devices (MEMS/NEMS). Thin films of various types are key components of modern microelectronics. Usually, the electronic properties are very important for the thin films. However, the mechanical features are also of interest for long time operation of MEMS/NEMS. During the process of growth and exploitation of films strong internal tensions can occur which after relaxation may cause deformation and destruction. Thin films imply the existence of a substrate from another material with different properties. The mechanical properties of thin films also depend on the features of substrate [1].

The study of the mechanical properties of thin films is done by the nanoindentation method that allows the identification of elastic and plastic deformations. For the determination of Young's modulus E and hardness H of thin films the method proposed by Oliver and Pharr is most often used [2]. This method was intensely applied for bulk materials. On the other hand, for investigations of the

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mechanical properties of thin films it is necessary to take the influence of other factors into account which depend on the substrate features. The determination of the contact surface between indenter and films using the method of Oliver-Pharr is imperfect due to "pile-up" extrusion or "sink-in" deepening of the material around the indentation imprints. With increasing indenter penetration in the material the mechanical properties are determined not only by the films but also by the substrate.

In the case of hard/soft coated systems the elastic and plastic deformation of the substrate also plays a relevant role. For soft/hard coated systems, it is necessary to exclude the influence of the substrate by keeping the penetration depth of the indenter to about 10–20 % of the film thickness. Due to the restricted capacity of the equipment this requirement cannot always be kept due to the very small indentation depths necessary. Thus, it is impossible to exclude the influence of substrate in the final measurements stage [3].

In this paper we study the mechanical properties of Cu thin films deposited on Si substrates. Copper, having a higher conductivity and better electromigration properties is today replacing aluminum in integrated circuits. It is also beneficial to use a material with a low dielectric constant (low-k) to fill the space between Cu interconnect lines in order to reduce the amount of cross talk between the lines and to place them closer to each other. Basically, the entire systems of devices has been changed with the introduction of Cu metallization.

For the mechanical durability mentioned above, the following features of the investigated complex structures are important: elastic modulus, yield stress, fracture toughness, etc. All these characteristics can be obtained applying the nanoindentation method.

8.2 Experimental Techniques

As a substrate, single crystals of n-type Si(100) with a resistivity $\rho = 4,5$ Ωcm doped with phosphorus have been used. The Cu films were obtained by magnetron sputtering at room temperature using a RF sputter apparatus. The thickness of Cu films was equal to $t = 85$ (1), 470 (2), and 1000 (3) nm (see Table 8.1).

The topology of the surface of the thin films was estimated using atomic force microscopy and optical microscopy. Areas of 20×20 μm dimensions were scanned; the roughness R_a values are shown in Table 8.1.

Table 8.1 The thickness t and roughness R_a of the Cu films

Sample		Thickness t, nm	Roughness R_a, nm
Cu	1	85	7
	2	470	9
	3	1000	12.2
Si	4	–	1.95

Nanoindentation was carried out with a Nanotester-PMT-3-NI-02 using the dynamical range of penetration.

A diamond Berkovich indenter with a tip radius of 150 nm was used in these tests. The indentation followed a trapezoidal loading profile with 20 s loading time, 5 s holding time under the load and 20 s unloading time. The loads of the Berkovich indenter varied in the limits $P_{max} = 2$–900 mN. The H and E values were evaluated as the mean of five indentations applied with the same load. Figure 8.1 shows the dependences of H and E as a function of the applied force.

8.3   Results and Discussions

The hardness of sample 1 for the maximum loads $P_{max} < 300$ mN is close to that of the Si sample 4. The increase of H with decreasing load is caused by the appearance of indentation size effects. The influence of the Si substrate on the hardness value of Cu/Si structures is confirmed by the load-displacement curves analysis, where the 'pop-in', 'pop-out' and 'elbow' effects can be noticed (Figs. 8.2 and 8.3).

The influence of P_{max} on the modification of the load-displacement curves is as follows (see Figs. 8.2 and 8.3). In the loading parts of the curves slight oscillations of the indenter displacement are observed for $P_{max} = 10$ mN and 20 mN, even the "pop-in" effect is taking place (indicated by arrows). For the unloading parts the situation is reverse, for $P_{max} = 10$ mN, the shape of curves differs from conventional ones.

Two different effects appear for decreasing loads: the "elbow" effect at 20–40 mN and "pop-out" for $P_{max} = 50$–300 mN. The "pop-in" effect for small loads appears due to pronounced plastic flow of the Cu film when the indenter penetrates in material. The effects of 'pop-out' and 'elbow' in the curves of Cu/Si systems for $P_{max} > 300$ mN occur due to phase transformations in the bulk Si [4].

Comparing the curves in Fig. 8.4a, d one observes the existence of the "pop-out" effect in the unloading curves of the bulk Si (indicated by arrows). The effect of the Si substrate becomes apparent for indentations with maximum loads smaller than 10 mN ($P_{max} = 7$ mN), when the indentation depth is approximately equal to the with films thickness ($t = 85$ nm), which is in accordance with Ref. [3]. This effect was confirmed also by our investigations for the Cu films of 470 and 1,000 nm. The "pop-out" effect cannot be observed when $h_{max}/t = 0.5$, here h_{max} is maximal indentation depth (nm) and t is the film thickness (nm) (see Fig. 8.4b, c).

We observed that the hardness of Cu/Si structure 1 approaches a saturation value for loads $P_{max} > 300$ mN. In this interval hardness value of the Si substrate 4 is smaller than the hardness of Cu/Si 1. This phenomenon takes place due to a protection effect by the Cu film against fragile destruction, i.e. due to the fact that the Cu film changes the geometry of the Berkovich pyramid by rounding the indenter tip a little. As a result, the indenter with its rounded tip more softly acts on the sample.
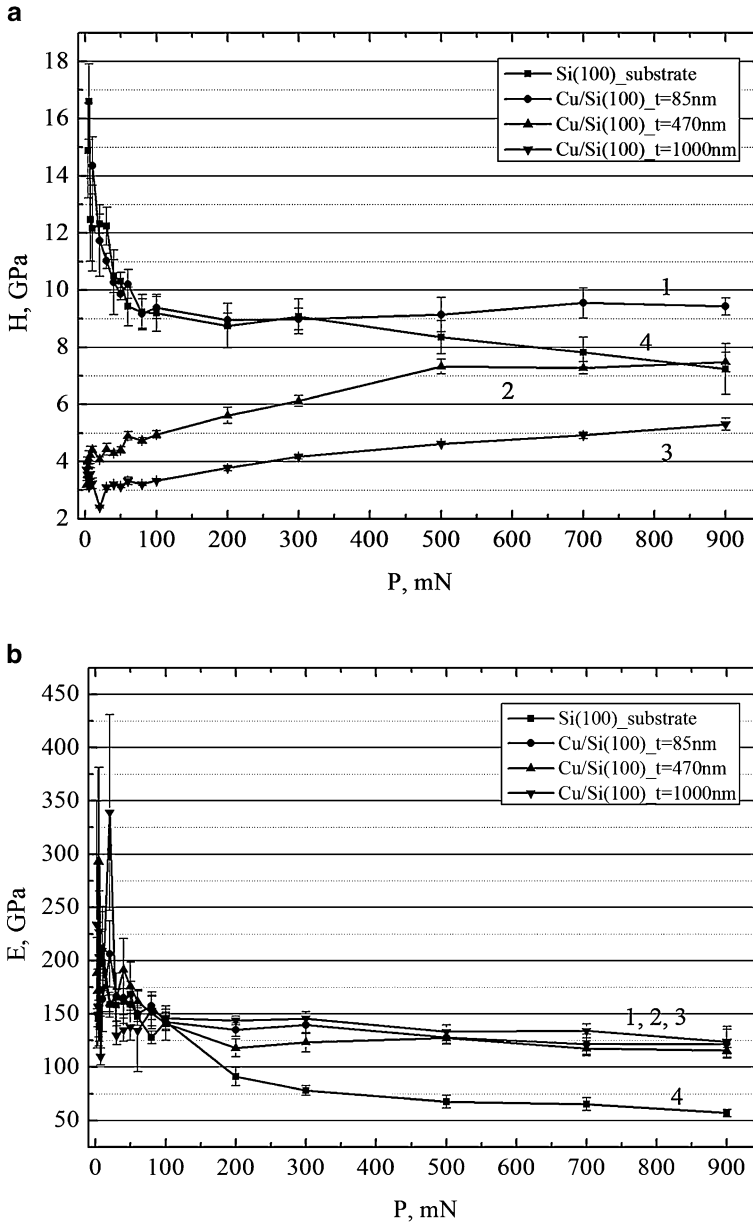
a

b

Fig. 8.1 Dependences *H(P)* (**a**) and *E(P)* (**b**) for the Cu/Si structures (*1*), (*2*), (*3*) and for the Si substrate (*4*)
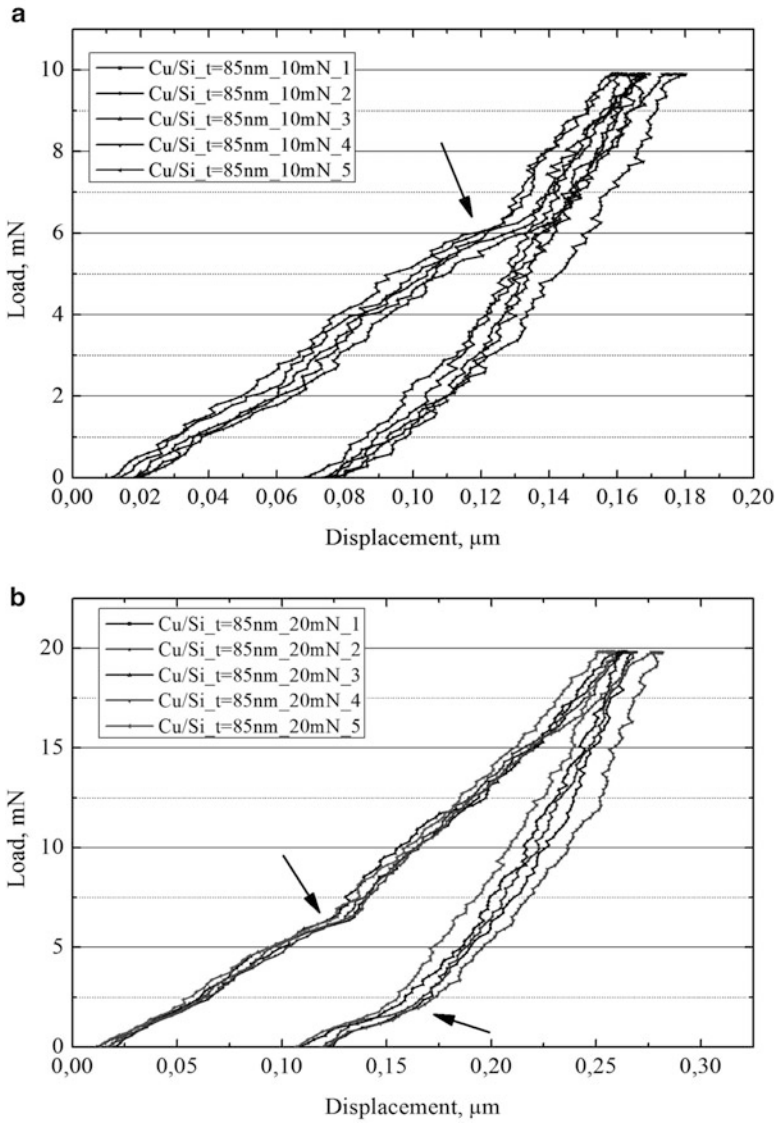
Fig. 8.2 Evolution of the for a Cu/Si structure (t = 85 nm) on the maximum applied load: (**a**) 10, (**b**) 20, (**c**), 50 mN for 5 indentations at each P$_{max}$
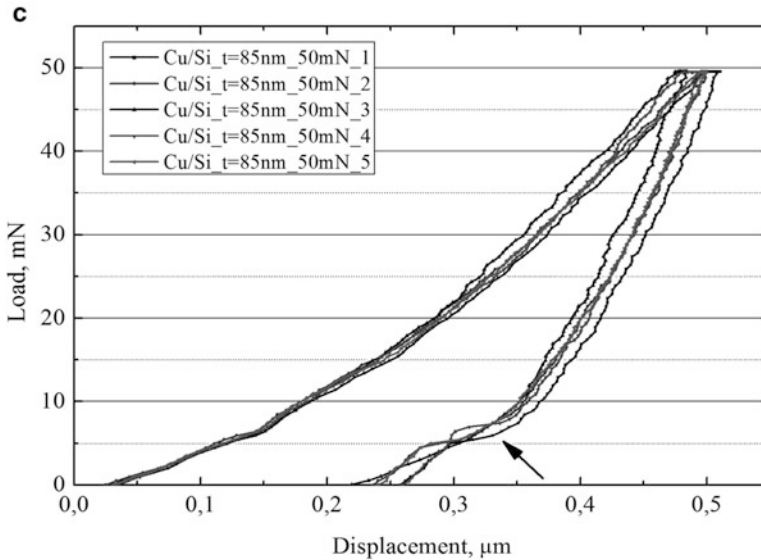
Fig. 8.2 (continued)

To confirm this idea, the Cu film was dissolved by chemical etching and the indentation images of Cu/Si, bulk Si and the residual indentation shapes after chemical etching were compared with each other. The results obtained are presented in Fig. 8.5.

When comparing the indentation images in Fig. 8.5b, c one can see that the cracks and destruction on the bulk Si surface are larger than the cracks on the Si substrate after chemical etching of the Cu film. This leads to an increase of the indentation dimensions; as a result a lower value of H is obtained for bulk Si.

As was mentioned above, the hardnesses of the Cu films 2 and 3 are on the same order of magnitude as described in [3]. The hardness of the Cu/Si structures is two times less than the hardness of bulk Si for small values of P_{max}. One can see from Fig 8.2c, d that the "pop-out" effect does not appear in the curves when the indenter penetrates to a depth of 50 % of the film thickness.

Figure 8.1a shows that in the case of the Cu/Si sample 2 for increasing P_{max} the hardness of the structure also increases reaching the hardness value of bulk Si. On the other hand, the hardness of the Cu/Si system 3 doesn't reach the hardness of bulk Si. The plastic indentations have been observed on the Si substrate after dissolution of the Cu film from the Cu/Si structures 2 ($P_{max} > 100mN$) and 3 ($P_{max} > 200mN$) by chemical treatment. It was concluded that the elastic action of the Si substrate has a dominant influence for $P_{max} < 100mN$. On the other hand, the influence of Si substrate is increasing for $P_{max} > 100mN$, and plastic deformation also takes place during the indentation. This conclusion is confirmed by the E (P) dependence (Fig. 8.1b). So, the Young's moduli E for both the system Cu/Si and bulk Si ($P_{max} < 100mN$) show a similar behavior. However, for $P_{max} > 100mN$ the elastic modulus of bulk Si has a 50 % lower value than the system Cu/Si. The reason of such a behavior can be attributed to the destruction of crystal in the indentation neighbourhood.
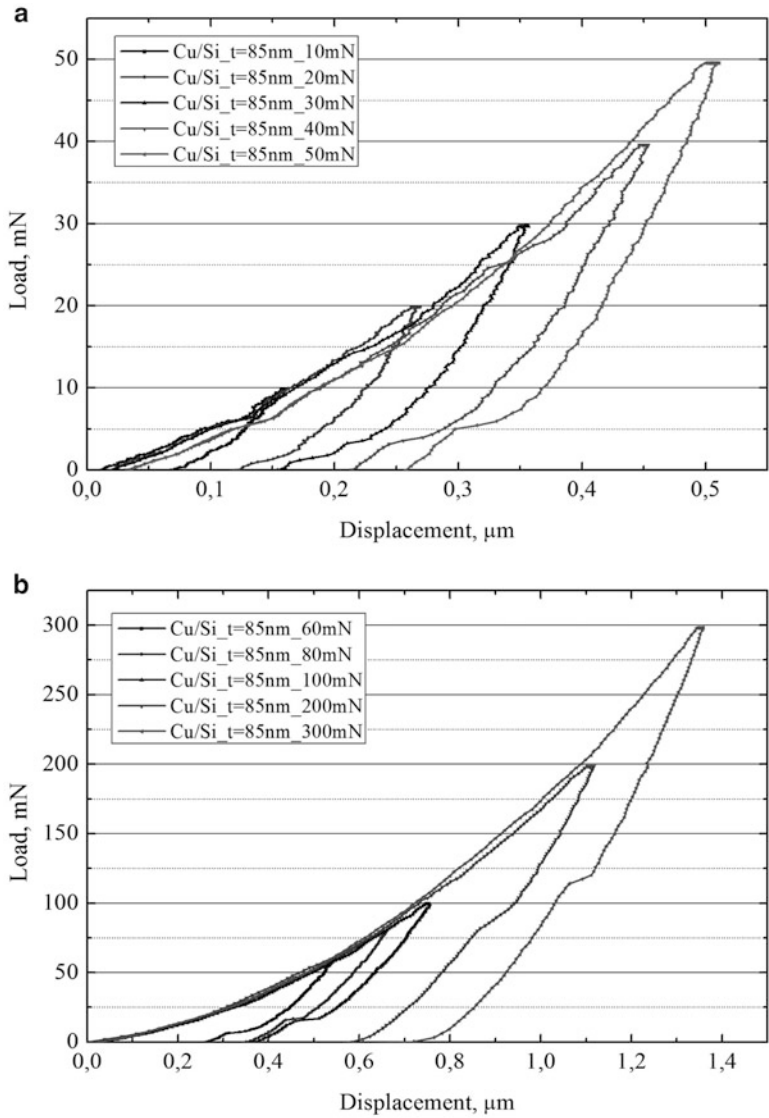
Fig. 8.3 Evolution of the load-displacement curves for Cu/Si structure (t = 85 nm) as function of the maximum applied load: (**a**) 10, 20,30, 40 50, (**b**) 60 80 100, 200, 300 mN

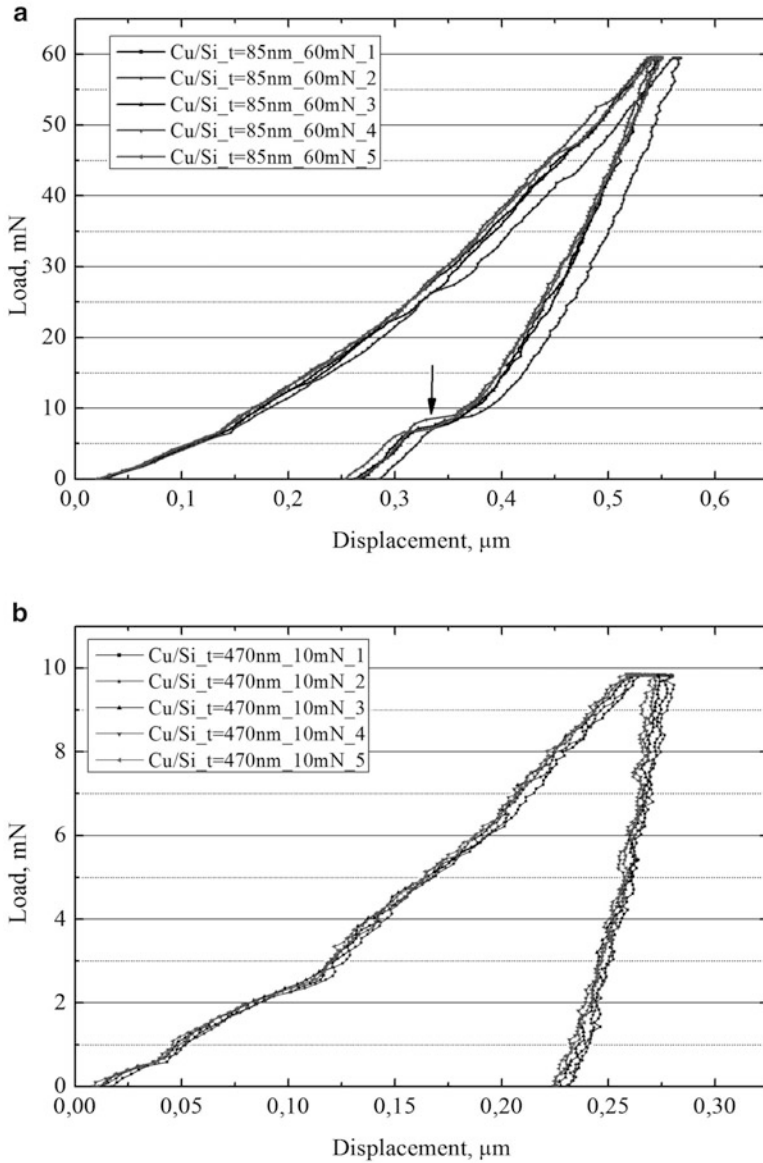**Fig. 8.4** Load-displacement curves for Cu/Si structures: (**a**) t = 85 nm, (**b**) t = 470 nm, (**c**) t = 1,000 nm and (**d**) of the Si substrate
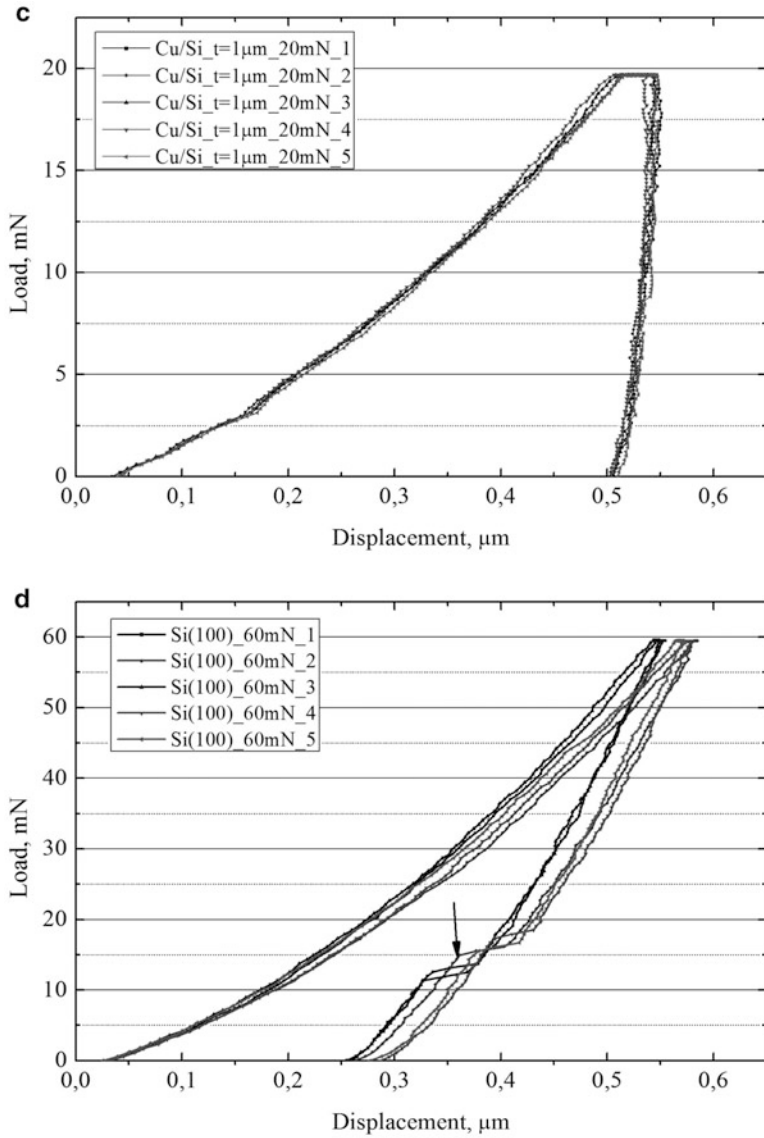
Fig. 8.4 (continued)

8.4 Conclusion

The mechanical properties of Cu thin films grown on Si(100) substrates using the magnetron sputtering method have been studied in the work. By use of the dynamic nanoindentation method, Cu/Si structures with different thicknesses of the Cu film
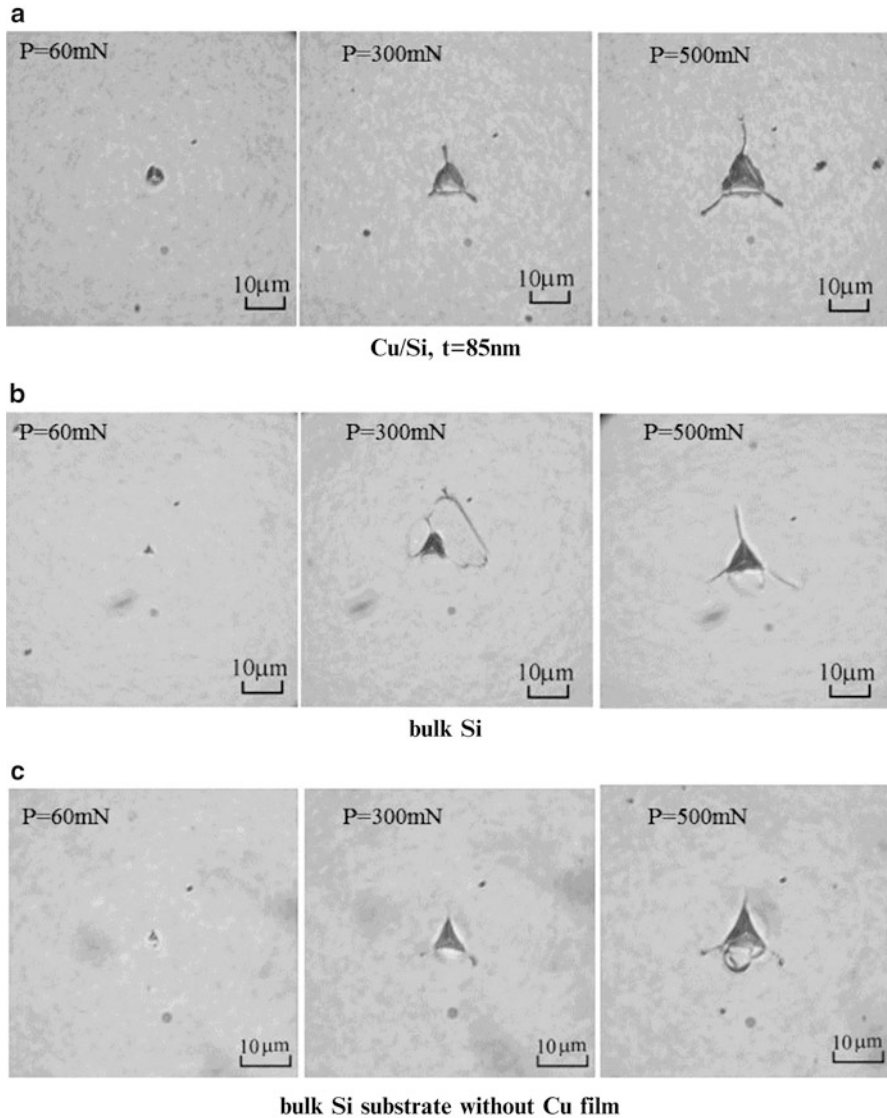
a

Cu/Si, t=85nm

b

bulk Si

c

bulk Si substrate without Cu film

Fig. 8.5 Shape of indentations made on the Cu/Si structure (**a**), bulk Si (**b**) and the residual indentation images on the Si substrate after chemical etching of Cu film (**c**)

(t = 85, 470 and 1,000 nm) were investigated. It was shown that the Cu film thickness and the range of maximum loads applied are some of the main factors influencing the deformation peculiarities and the mechanical properties (Young modulus and hardness) of the film/substrate structures.

References

1. Read DT, Volinsky AA (2007) Micro- and Opto-electronic materials and structures: physics, mechanics, design, reliability, packaging, vol 1, A135
2. Oliver WC, Pharr GM (1992) J Mater Res 7:1564
3. Shugurov AR, Panin AV, Oskomov KV (2008) PSS 50:1050
4. Shikimaka O et al (2010) 5th International conference on materials science and condensed matter physics. Book of abstracts, Chisinau, 13–17 Sept 2010, p 127