Nanoindentation Behaviour and Microstructural Evolution of Au/Cr/Si Thin Films

Woei-Shyan Lee^{1,2,*}, Te-Yu Liu¹ and Tao-Hsing Chen²

¹Department of Mechanical Engineering, National Cheng Kung University, Tainan 701, Taiwan, R.O.China

²Center for Micro/Nano Science and Technology, National Cheng Kung University, Tainan 701, Taiwan, R.O.China

The nano-mechanical properties of an as-deposited composite Au/Cr/Si film comprising a Au layer with a thickness of 800 nm and a Cr adhesive layer with a thickness of 10 nm deposited on a Si (100) substrate are investigated by performing nanoindentation tests to a maximum depth of 1500 nm. The microstructural evolutions of as-deposited indented specimens and specimens annealed at temperatures of 523 K, 623 K or 723 K for 2 min are then examined using scanning electron microscopy (SEM) and transmission electron microscopy (TEM) techniques. The loading curve for the as-deposited Au/Cr/Si thin film is found to be continuous and smooth. However, the unloading curve has a prominent pop-out feature. The hardness and Young's modulus of the Au/Cr/Si thin film indented to a maximum depth of 1500 nm are determined to be 2.7 GPa and 110 GPa, respectively. In the as-deposited sample, the microstructure of the indentation zone is characterised by a mixed structure comprising amorphous phase and nanocrystalline phase. Furthermore, well-defined Au, Cr and Si layers are observed in the interfacial region of the thin film. However, at the highest annealing temperature of 723 K, the microstructure of the indentation zone is recovered to a perfect diamond cubic single crystalline state. Finally, silicidation of the Cr layer takes place in all the annealed samples, resulting in the formation of isolated nano-islands of Cr. [doi:10.2320/matertrans.MRA2008385]

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1. Introduction

The nano-mechanical properties and microstructural evolution of thin films under different temperature conditions are of fundamental importance in microelectronic and optoelectronic applications.^{1,2)} Reducing the characteristic device size and improving the scale of device integration requires the fabrication of films with reduced thickness. However, due to differences in the properties of the various materials used to fabricate micro-electrical devices, the mechanical behaviour of the thin films used in such devices tends to be very complicated. Fortunately, nanoindentation provides a powerful technique for measuring the mechanical properties of thin films.³⁾ Furthermore, the use of high-resolution microscopes⁴⁾ to observe the indentation deformation morphology and microstructural evolution of thin films under different indentation deformations and temperature conditions provides detailed insights into their nano-mechanical behaviour.

Silicon bonding is a crucial technique in integrated circuit (IC) fabrication.⁵⁾ Si-Au compound is considered to be one of the most promising materials for achieving eutectic bonds in IC components because of its low eutectic temperature (636 K).⁶⁾ During IC fabrication, a Ti or Cr layer is generally deposited between the Si substrate and the Au layer to improve the adhesive properties of the joint.⁷⁾ The mechanical properties of the resultant Au/Ti/Si or Au/Cr/Si thin films not only affect the mechanical performance of the device, but also determine its electrical and optical characteristics. The properties of Au/Cr/Si thin films are fundamentally dependent on their microstructure, and even very small microstructural changes are sufficient to prompt a significant change in the film's mechanical properties.

The plastic deformation and microstructural changes induced in heated nanoindented thin Au/Cr/Si films vary

with the heating conditions. Accordingly, optimising the performance of IC devices and micro electro-mechanical systems (MEMS) packages requires a detailed understanding of the nano-mechanical properties and microstructures of Au/Cr/Si thin films in both the as-deposited condition and the annealed state. Therefore, this study commences by investigating the mechanical properties of an as-deposited Au/Cr/Si thin film using a nanoindentation technique with a maximum indentation depth of 1500 nm. The thin film has a composite structure comprising a Au film with a thickness of 800 nm and a Cr layer with a thickness of 10 nm which serves both as an adhesive layer and a barrier to prevent diffusion between the Au film and the Si substrate. The indentation morphologies and microstructural characteristics of the thin film are then investigated in both the as-deposited state and following annealing at temperatures of 523 K, 623 K or 723 K, respectively, for 2 min.

2. Experimental Procedure

The Au/Cr/Si thin films used in the current investigation were prepared by National Nano Device Laboratories, Taiwan. The fabrication process commenced by evaporating a thin adhesive layer of Cr (10 nm) onto a p-type Si single crystal wafer with a (100) orientation. A Au film with a thickness of approximately 800 nm was then deposited on the Cr layer using an evaporation deposition process. During the deposition procedure, the substrate was maintained at a temperature of 423 K to improve the homogeneity of the thin film. The deposition process was carried out within a chamber maintained at a pressure of between 10^{-5} and 10^{-7} Torr using a hybrid system comprising a mechanical pump and an oil diffusion pump. The thickness of the gold thin film was monitored and controlled during the deposition process using a quartz-crystal microbalance and was subsequently confirmed by X-ray reflectometry following the fabrication process.

*Corresponding author, E-mail: wslee@mail.ncku.edu.tw

Table 1 Thermal annealing schedule for indented Au/Cr/Si thin film specimens.

Annealed temperature (K)	Heating time (sec)	Holding time (min)	Cooling time (min)
523	5	2	15
623	5	2	18
723	5	2	20

The nanoindentation tests were performed at room temperature in air using an MTS Nano Indenter XP system fitted with a Berkovich diamond pyramid tip with a radius of 40 nm. The as-deposited specimens were indented to a depth of 1500 nm using the indenter system set in a depth-control mode. The loading-unloading cycle involved the following steps: (1) loading to the point of maximum load, corresponding to the designated maximum depth of 1500 nm; (2) holding in this position for 10 s; and (3) smoothly unloading over a period of 30 s. The maximum range of the calculation depth was greater than 500 nm, with a depth resolution of 0.01 nm. The hardness and Young's modulus of the Au/Cr/Si thin film were then calculated from the load-displacement curve using the Oliver and Pharr method.^{8,9)} The errors accumulated in the nanoindentation calculations were compensated using the semi-ellipse method proposed by Kese *et al.*¹⁰⁾ In addition, the mean load was obtained from the initial oscillatory load using the super impose method.

Following the nanoindentation tests, the specimens were annealed at temperatures of 523 K, 623 K or 723 K for 2 min in a rapid thermal annealing (RTA) processing system. During the annealing process, purified nitrogen gas (99.999%) was passed through the furnace at a flow rate of 3 L/min. The annealing schedules for each of the specified temperatures are summarised in Table 1. The indented surface morphologies of the as-deposited and annealed specimens were observed using a scanning electron microscope (SEM) with an operating voltage of 0.5~30 keV and a resolution of 1.5 nm. Following SEM inspection, thin foil specimens for TEM inspection were prepared from the as-deposited and annealed samples using an FEI Nova 200 focused ion beam (FIB) milling system with a Ga⁺ ion beam and an operating voltage of 30 keV. The cross-sectional microstructures of the various specimens were observed using a Philips Tecnai F30 Field Emission Gun Transmission Microscope operated at 300 keV. The chemical compounds observed at various positions in the as-deposited and annealed samples were identified using an EDAX Energy Dispersive X-Ray spectrometer fitted to the TEM system.

3. Results and Discussion

3.1 Loading-unloading curve

Figure 1(a) presents the loading-unloading curve of the as-deposited Au/Cr/Si thin film. It can be seen that the loading curve is smooth and continuous, which suggests that no debonding or cracking occurs during the loading procedure. However, the unloading curve has a distinct point of discontinuity; generally referred to as a pop-out feature. The maximum indentation load is found to be approximately 122 mN, which is significantly higher than the critical load

of 30 mN associated with the pop-out event in hard Si substrates.¹¹⁾ Furthermore, the unloading slope is very steep, which indicates that the indentation of the thin film involves plastic deformation followed by a very weak elastic return. Note that to verify the allowable drift rate set at the initial stage of the indentation test, the unloading process was terminated at 90% peak load, and thus it is observed that the unloading curve terminates after the pop-out event rather than at zero force. In general, the pop-out features observed in nanoindentation tests have been variously attributed to an undensification of Si,¹²⁾ residual deformation of the thin film,¹³⁾ phase transformation,¹⁴⁾ and the indentation rate.¹⁵⁾ The pop-out feature observed in the present Au/Cr/Si thin-film system is thought to be the result of the indentation rate. Comparing the load-displacement curves generated during nanoindentation at different loading rates, it is found that at the loading rate of 1 mN/s, a well-defined pop-out appeared on the unloading part of the curve, as shown in Fig. 1(a). In contrast, when the loading rate is 0.2 mN/s, a slight elbow (change in slope) can be seen on the unloading part of the curve, as in Fig. 1(b).

3.2 Young's modulus curve of Au/Cr/Si thin film

Figure 1(c) illustrates the variation of the Young's modulus value with the nanoindentation depth. It is observed that the Young's modulus decreases significantly as the tip first penetrates the thin Au/Cr/Si film. However, the modulus value then increases approximately linearly with an increasing indentation depth, and achieves a final value of approximately 110 GPa at the maximum indentation depth of 1500 nm. For indentation depths of less than 10 nm, the contact area between the indenter and the thin film is very small and the Young's modulus has a relatively high value of 40 GPa. The high Young's modulus value arises here because it varies inversely with the contact area when the load is small and virtually constant, as is the case under very low indentation depths. However, as the indentation depth increases, the presence of the Au layer causes the Young's modulus to decrease to a value of 10 GPa. As the indentation depth increases further, the tip makes contact with the Si substrate and thus the Young's modulus gradually increases. At the maximum indentation depth of 1500 nm, the Young's modulus rises to a value of approximately 110 GPa. This value is less than that of the Si (100) substrate (172.8 GPa¹⁴⁾), but is relatively higher than that of bulk Au (88 GPa).¹⁶⁾ The overall tendencies of the Young's modulus curve are governed by the substrate effect. The presence of the Au layer on the upper surface of the substrate reduces the Young's modulus value considerably compared to that observed for an uncoated Si(100) substrate. Li *et al.*¹⁷⁾ investigated the variation of the Young's modulus and hardness as a function of the indentation depth at various peak loads for the case of an undoped Si(100) substrate. Their results showed that the slight change in Young's modulus and hardness at smaller indentation depths was due to the roundness of the indenter tip and surface oxidation of the undoped substrate. The Young's modulus value of the undoped Si(100) substrate was found to be 165 GPa, which is considerably higher than the value of 110 GPa found in the current study for the Au-coated Si substrate.

Fig. 1 Load-displacement curve generated during nanoindentation of as-deposited Au/Cr/Si thin film at different loading rates: (a) 1 mN/s and (b) 0.2 mN/s; (c) variation of Young's modulus of Au/Cr/Si thin film with indentation depth; (d) variation of hardness of Au/Cr/Si thin film with indentation depth.

3.3 Hardness curve of Au/Cr/Si thin film

Figure 1(d) illustrates the variation of the Au/Cr/Si thin film hardness with the nanoindentation depth. At very low indentation depths, the film has a high hardness. As in the case of the Young's modulus, the high hardness value reflects the fact that the contact area between the indenter and the thin film is very small during the initial stages of the nanoindentation process. However, as the indentation depth increases, the hardness drops rapidly to a minimum value of approximately 0.65 GPa as a result of the Au film. Thereafter, the hardness increases slightly and then remains at a constant value until an indentation depth of approximately 800 nm, at which point it increases linearly with an increasing indentation depth as a result of the substrate effect. At the final indentation depth of 1500 nm, the hardness has a value of approximately 2.7 GPa, which is higher than the hardness of bulk Au, i.e. 0.64 GPa,¹⁶⁾ but less than that of the

Si (100) substrate (13 GPa)¹⁴⁾ due to the presence of the Au layer. Li *et al.*^{18,19)} examined the hardness of undoped silicon, p^+ -type silicon, polysilicon bulk and n^+ -type polysilicon film using a nanoindentation technique at loads varying from 0.2 to 15 mN. Their results showed that the polysilicon bulk specimen exhibited the highest hardness of around 13 GPa, followed by the undoped Si(100), the n^+ -type polysilicon film, and finally the n^+ -type silicon, with a hardness of around 5 GPa.

In general, when plotting the measured hardness data against the displacement for a soft film/hard substrate system such as that considered in the present study, three distinct hardness regimes are identified, ranging from "film-only" hardness to "substrate-only" hardness, with each regime corresponding to a specific normalised depth (i.e. the contact depth (h_c) divided by the film thickness (t)). For shallow penetration depths (regime I), the response is related to that

Fig. 2 SEM micrographs of indentation marks on specimen surface in: (a) as-deposited specimen; (b) specimen annealed at 523 K for 2 min; (c) specimen annealed at 623 K for 2 min; and (d) specimen annealed at 723 K for 2 min.

of the film hardness only. However, as the depth increases (regime II), the hardness also increases, and is associated with the mixed response of the thin film and the substrate, respectively. When the indentation depth extends as far as regime III, the hardness response is dominated by the substrate hardness. It has been reported that the critical normalised depth representing the boundary between regime II and regime III is around 10.²⁰⁾ In the current study, the thickness of the Au film is 800 nm. Consequently, it can be inferred that the “substrate-only” hardness regime exists at indentation depths greater than 8000 nm, which is far higher than the maximum indentation depth of 1500 nm considered in the present experiments. Thus, the hardness value obtained at 1500 nm reflects a mixed response of the Au film and the Si substrate.

3.4 Indentation surface morphology

Figure 2(a) presents a SEM micrograph of an as-deposited specimen nanoindented to a depth of 1500 nm. A pile-up effect is observed on the Au surface around the indentation. This pile-up region represents an area of permanent plastic deformation formed in the Au film during the indentation process. The pile-up phenomenon is directly related to the presence of the underlying Si substrate. Specifically, during nanoindentation, the plastic flow of the Au film is constrained by the relatively non-deformable substrate and hence the Au flows preferentially toward the surface.²¹⁾ Figure 2(b) shows that annealing the specimen at a temperature of 523 K causes a change in the shape of the indentation site. A high annealing temperature promotes atomic diffusion within the thin film, and thus causes a homogenisation of the pile-up area and a

Fig. 3 (a) Bright field TEM micrograph of as-deposited indented specimen; (b) High resolution TEM micrograph of the indentation affected zone; (c) EDX spectrum of indentation affected zone; (d) bright field TEM micrograph of Au/Cr/Si interface in as-deposited specimen.

partial recovery of the indentation site. Figure 2(b) shows that an annealing temperature of 523 K is sufficient to homogenise the pile-up area around the edge of the indentation site. However, diffusion takes place less readily in the Si substrate than in the Au film and hence the temperature rise induced in the substrate during annealing at temperatures between 523 K and 723 K is insufficient to recover the indentation site fully (see Figs. 2(b) to 2(d)).

3.5 Indented microstructure of as-deposited Au/Cr/Si thin films

Figure 3(a) shows the morphology of the indentation

affected zone of an as-deposited specimen. It can be seen that the microstructure is characterised by a “semi-crystal” state, i.e. an amorphous area (with no common structure or direction) containing a number of coherent crystal phases. The semi-crystal state of the indentation affected zone is confirmed by the high resolution TEM micrograph presented in Fig. 3(b). The crystal reorganisation in the indentation affected zone is the result of the pressure induced beneath the indenter during penetration, which breaks the original (100) oriented crystal structure. Since the present thin-film system comprises a soft Au film on a hard silicon substrate, the relatively non-deformable Si substrate constrains the plastic

flow in the thin film as the indenter descends, and hence the Au flows toward the surface, where it accumulates around the edges of the indentation. This phenomenon is consistent with the findings presented by Tsui and Pharr²¹⁾ regarding the flow effect in soft films on hard substrates indented to greater depths. Due to the flow behaviour of the Au thin film described above, no Au atoms are found in the indentation zone. In other words, the indentation affected area contains no Au-Si alloy phase. This is confirmed by the EDX analysis results presented in Fig. 3(c), which correspond to the white square labelled A in Fig. 3(a). Figure 3(d) presents TEM micrographs of the Au/Cr/Si interface region. The micrographs show a well-defined arrangement of the interfacial layers with no silicidation effect.

The nanoindentation results and TEM observations presented in this study reveal that in the as-deposited specimen, the indentation deformation prompts the formation of a mixed structure of amorphous and nanocrystalline phase within the indentation zone. This mixed structure results in a lower hardness (2.7 GPa) and Young's modulus (110 GPa) than that measured in an undeformed silicon substrate (i.e. 13 GPa and 172.8 GPa, respectively), which has a perfect diamond cubic single crystalline state.

3.6 Indented microstructure following annealing at different temperatures

Figure 4(a) shows the indented microstructure of a Au/Cr/Si specimen annealed at a temperature of 523 K for 2 min. Comparing the TEM diffraction pattern of the indentation affected zone of the annealed specimen with that of the as-deposited specimen (Figs. 3(a) and 3(b)), it can be seen that following the annealing process, the microstructure of the indentation affected zone still contains a mixed structure of amorphous phase and nanocrystalline phase, as shown in Fig. 4(b). The EDX analysis results presented in Fig. 4(c), corresponding to the white square labelled A in Fig. 4(a), show that the indentation affected area contains neither Au nor Au-Si phase.

The elevated temperature during the annealing process has a marked effect on the microstructure of the Au/Cr/Si interface. The bright field TEM micrograph presented in Fig. 4(d) shows that at an annealing temperature of 523 K, the Cr layer is nonexistent in some regions of the interface. This phenomenon is the result of Si atoms diffusing into the original Cr layer (silicidation) under the influence of the higher temperature. The diffusion phenomenon is confirmed by the diffraction pattern shown in the inset of Fig. 4(d), corresponding to the region marked by the white square labelled A. The corresponding EDX analysis results are presented in Fig. 4(e) and show that the composition consists of 48.4% Si, 38.8% Au and 12.8% Cr. Furthermore, in Fig. 4(d) it is apparent that the well-ordered arrangement of the Au, Cr and Si layers observed in the as-deposited specimen (Fig. 3(d)) is absent in the annealed specimen. It is noted that the chemical composition at the interface region between Au film and CrSi layer (corresponding to the square region indicated by label D in Fig. 4(d)) is examined as 45.1% Si, 43.2% Au and 11.7% Cr. Figure 4(f) shows EDX analysis results for interface region marked by square D in Fig. 4(d).

Figure 5(a) presents TEM micrographs of an indented Au/Cr/Si specimen annealed at a temperature of 623 K for 2 min. It is observed that the indentation affected zone contains a mixed structure of amorphous phase and nanocrystalline phase, while the Si substrate retains a crystalline structure. Figure 5(b) shows the high resolution TEM micrograph of the indentation affected zone. Both structures are confirmed by the corresponding selected area diffraction patterns. The EDX analysis results presented in Fig. 5(c), corresponding to the white square labelled A in Fig. 5(a), show that the indentation affected zone contains no Au. Meanwhile, the TEM micrograph in Fig. 5(d) shows that the diffusion activity at the interface increases under the higher annealing temperature of 623 K, and results in the formation of isolated Cr islands. Moreover, some Au atoms diffuse into the Si substrate. The EDX analysis results presented in Fig. 5(e), corresponding to the white square labelled C in Fig. 5(d), show that the interfacial region between Au film and CrSi layer consists of 16.2% Si, 77% Au and 6.8% Cr.

At the highest annealing temperature of 723 K, the microstructure of the indentation affected zone is very different from that observed in the as-deposited specimen or the specimens annealed at 523 K or 623 K, respectively. The TEM micrograph presented in Fig. 6(a) shows that the indentation affected zone has a perfect diamond cubic single-crystalline structure. This structure is also confirmed by the high resolution TEM micrograph presented in Fig. 6(b). Furthermore, the EDX analysis results presented in Fig. 6(c), corresponding to the white square labelled A in Fig. 6(a), show that the indentation affected zone contains only Si. The TEM micrograph presented in Fig. 6(d) shows the presence of isolated nano-islands of Cr in the interfacial region, which suggests a greater diffusion and silicidation effect at the higher annealing temperature of 450°C. Finally, the EDX analysis results presented in Fig. 6(e), corresponding to the white square labelled B in Fig. 6(d), indicate that the interfacial region between Au film and CrSi layer comprises 8.3% Si, 89.0% Au and 2.7% Cr. It should be noted that the variations of the chemical composition are examined at the same interface region between Au film and CrSi layer located at 1200 nm away from the edge of the indentation site. The examined portion is marked by label D in Fig. 4(d), label C in Fig. 5(d), and label B in Fig. 6(d), respectively. Table 2 lists the variations of the chemical composition at the examined Au/CrSi interface region as a function of annealing temperature. It is apparent that the Au content increases from 43.2% to 89%, while the Cr and Si contents decrease from 11.7% to 2.7% and 45.1% to 8.3%, respectively, as the annealing temperature is increased from 523 K to 723 K. This result indicates that the degree of diffusion activity at the interface region is enhanced while the annealing temperature is increased.

Table 2 Composition (mass%) of the interface region between Au film and CrSi layer of specimen annealed at 523 K, 623 K and 723 K, respectively.

Annealed temperature	Au	Cr	Si
523 K	43.2	11.7	45.1
623 K	77	6.8	16.2
723 K	89	2.7	8.3

Fig. 4 (a) Bright field TEM micrograph of indented specimen annealed at 523 K for 2 min; (b) High resolution TEM micrograph of the indentation affected zone; (c) EDX analysis results for indentation affected zone in Fig. 4(a); (d) bright field TEM micrograph of Au/Cr/Si interfacial region in Fig. 4(a); EDX analysis results for (e) region marked by square A in Fig. 4(d); (f) interface region marked by square D in Fig. 4(d).

Fig. 5 (a) Bright field TEM micrograph of indented specimen annealed at 623 K for 2 min; (b) High resolution TEM micrograph of the indentation affected zone; (c) EDX analysis results for indentation affected zone in Fig. 5(a); (d) isolated nano-islands of Cr in interfacial region in Fig. 5(a); (e) EDX analysis results for interfacial region marked by square c in Fig. 5(d).

Fig. 6 (a) Bright field TEM micrograph of indented specimen annealed at 723 K for 2 min; (b) High resolution TEM micrograph of the indentation affected zone; (c) EDX analysis results for indentation affected zone in Fig. 6(a); (d) isolated nano-islands of Cr in interfacial region in Fig. 6(a); (e) EDX analysis results for interfacial region marked by square B in Fig. 6(d).

4. Conclusions

This study commenced by investigating the nano-mechanical properties of an as-deposited Au/Cr/Si thin film indented to a maximum depth of 1500 nm. The indented microstructures of as-deposited and annealed thin-film specimens were then systematically examined. The results have shown that the load-displacement curve of the as-deposited film has a distinct pop-out feature in the unloading curve. The hardness and Young's modulus values of the film at an indentation depth of 1500 nm are determined to be 2.7 GPa and 110 GPa, respectively. The results have indicated that the microstructural characteristics of the indentation zone are significantly dependent on the annealing temperature. In the case of the as-deposited specimens, the indentation deformation induces a mixed structure of amorphous and nano-crystalline phase within the indentation zone. However, the elevated temperature associated with the annealing process performed at 723 K prompts a full recovery of the microstructure in the indentation zone to a perfect diamond cubic single crystalline state. The results have also shown that the degree of diffusion activity is enhanced as the annealing temperature is increased. This phenomenon leads to a silicidation effect of the Cr layer.

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