Control of residual stress in thin films by substrate vibration and their mechanical property

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Abstract　The prevention of residual stress of thin film syntheses is very important in processing engineering surfaces. In this paper, details of residual stress control by the vibration of the substrate using PZT at voltage of 0 - 100 V are presented. The thin films deposited are crystalline TiN, Ti, Cu, Al films and amorphous C, Si films. The residual stresses are measured using Stoney's method. The results of our experiment show that for Ti, TiN and Cu thin films, compressive stresses are changed to tensile stresses. XRD and Auger spectroscopy results verify that the composition and crystalline structure of the treated and untreated crystalline thin films to be the same. But it is shown that the grain size in the film tend to decrease with increasing vibration amplitude. From the change of the microstructure of the film, a model for the control of residual stress is proposed. The change of adhesion strength is measured by a pin-on-disk tribo-tester with variation of the residual stress. The adhesion strength shows the maximum value at near zero residual stress. Therefore, control of residual stress by substrate vibration is shown to be an effective method for improving the wear life.

1. Introduction

The development of synthesis techniques for thin film deposition has gained considerable attention in recent years. In the field of surface modification techniques, thin films for mechanical uses such as DLC, TiN and CrN films have emerged as successful hard coating films.　However, due to the residual stress, the films delaminate from the substrate and remains to be a problem. Until now, in order to decrease the residual stress, the change of the deposition parameters [1] and the synthesis of buffer layer [2] have been carried out. But these processes have changed the crystalline structure, composition and their mechanical properties remarkably. These methods have not only worsened the properties of the films, but also need much effort to find suitable deposition parameter. Therefore, the development of a new control method for decreasing the residual stress is vital with significant application in various hard coating materials.

In this paper, an excellent and simple method for controlling the residual stress is proposed. Here, substrate vibration that is independent of the deposition condition is used. In this process, only substrates are vibrated by PZT during the deposition. The changes of The development of synthesis techniques for thin film deposition has gained considerable attention in recent years. In the field of surface modification techniques, thin films for mechanical uses such as DLC, TiN and CrN films have emerged as successful hard coating films.　However, due to the residual stress, the films delaminate from the substrate and remains to be a problem. Until now, in order to decrease the residual stress, the change of the deposition parameters [1] and the synthesis of buffer layer [2] have been carried out. But these processes have changed the crystalline structure, composition and their mechanical properties remarkably. These methods have not only worsened the properties of the films, but also need much effort to find suitable deposition parameter. Therefore, the development of a new control method for decreasing the residual stress is vital with significant application in various hard coating materials.

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the residual stress is proposed. Here, substrate vibration that is independent of the deposition condition is used. In this process, only substrates are vibrated by PZT during the deposition. The changes of residual stress are tested on TiN, Ti, Cu, Al, Si and C films. Further more, the relationship between residual stress and the mechanical properties such as wear life hardness and elastic coefficient are clearly shown.

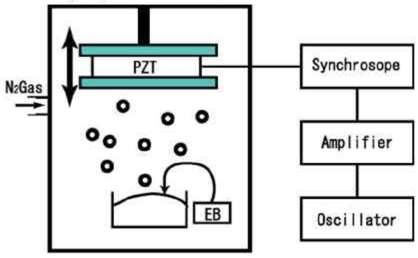


Fig. 1 Schematic of deposition system

Table 1 The thickness and crystalline of the films

Film	Thickness (nm)	Deposition rate (nm/min)	Crystalline
TiN	100,200,400	60	Crystal
Ti	300	60	Crystal
Cu	1000	30	Crystal
Al	1000	100	Crystal
C	100	3	Amorphous
Si	100	40	Amorphous

2. Experimental

A schematic diagram of the experimental set up is shown in Fig. 1. The sample holder has piezoelectric (PZT) vibrator that can vibrate vertically with amplitude of 0-20 nm and frequency of 0-1MHz. A bias voltage varies the amplitude at the rate of 0.2 nm/V. The heat generated was negligible with a temperature increase of less than 30 ℃. The residual stress that occurred during deposition is not related to the thermal stress and is attributed to the intrinsic stress of the films.

Electron beam evaporator was used to deposit various films of TiN, Ti, Cu, Al, C, and Si. TiN films were synthesized in N_2 gas flow during Ti deposition. Background presser is less than 5.0×10^{-4} Pa. The films were deposited on Si (100) wafers that washed by acetone. Table Ⅰ shows each film's thickness and crystallization. The effect of the thickness and crystallization were researched.

XRD and Auger electron beam spectroscopy results verify that the composition and crystalline structure.

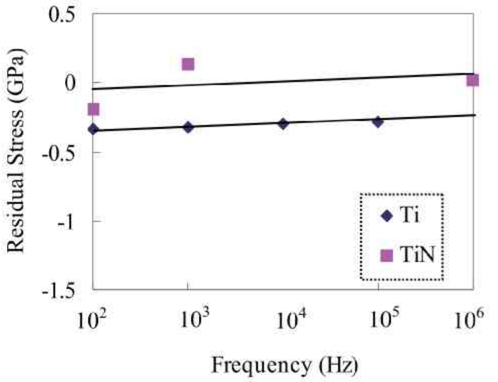


Fig.2 Variation of residual stress with frequency for Ti and TiN films.

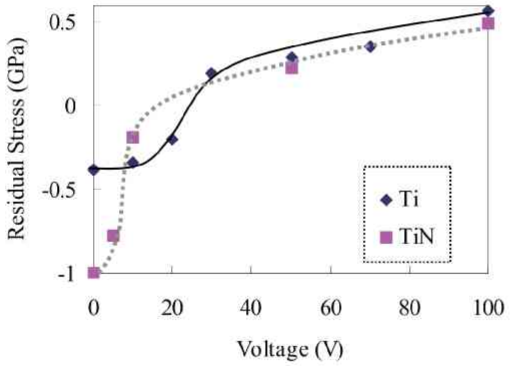


Fig.3 Variation of residual stress with bias voltage for Ti and TiN films.

The residual stress of the films was measured using Stoney's method. The hardness and the elastic constant were measured by a nano-indentation technique with a trigonal diamond indenter (Berkovich-type indenter). The maximum indentation loads were chosen in the ranges of 0.8-3.0 mN.

The friction coefficient and frictional characteristics were investigated by a ball-on-disk type tribo-tester with 550 mN against a steel ball with a diameter of 5 mm in an atmospheric pressure. SEM and optical microscopy were used to observe the wear tracks. The sectional areas of wear tracks were measured by the stylus profile system.

3. Results and discussion

3.1 Control of residual stress by substrate vibration

3.1.1 Relationship between residual stress and vibration parameters

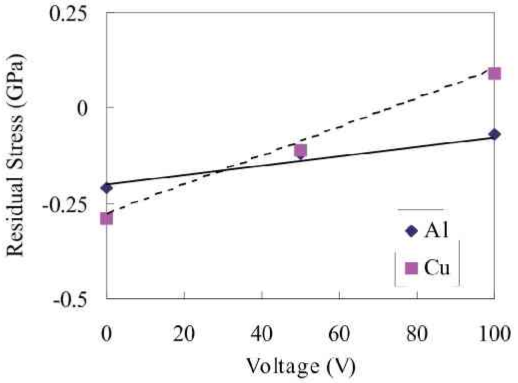


Fig.4 Variation of residual stress with bias voltage for Al and Cu films.

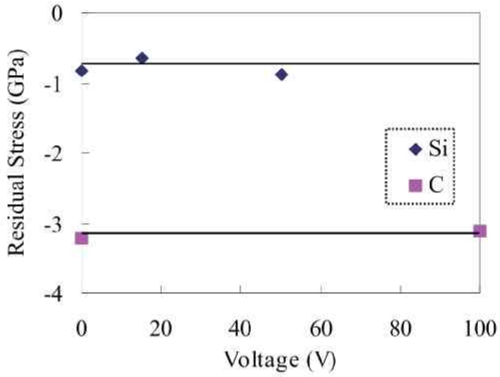


Fig.5 Variation of residual stress with bias voltage for C and Si films.

The vibration parameters are frequency and bias voltage. These parameters are considered to give effective changes in the residual stress. When Ti and TiN films were deposited without substrate vibration, the films show compressive stress. On the contrary, the compressive residual stress decreased to zero internal stress at 30V and reached to the highest tensile stress through the transition range from compressive to tensile stress irrespective of film material when the bias voltage changed.

It is clear seen that the dependence of the bias voltage on the residual stress are larger than of frequency.

The relationship between frequency and residual stress for Ti and TiN films is shown in Fig. 2. Here, the bias voltage is kept constant at 10 volts. Variation of frequency with residual stress in both Ti and TiN films were constant. In Ti and TiN films the relationship between bias voltage and residual stress were showed Fig. 3. The frequency was constant (100Hz). When the voltage was increased (10 and 20V) the residual compressive stress was decrease. But when the voltage was increased (30V) a transition region was revealed where by the stress became tensile. And moreover, when the voltage was increased (50-100V) the residual tensile stress was increased. In TiN films the residual stress changed in a similar way to that of Ti films. So the bias voltage was observed to be the effective vibration parameter for controlling the residual stress.

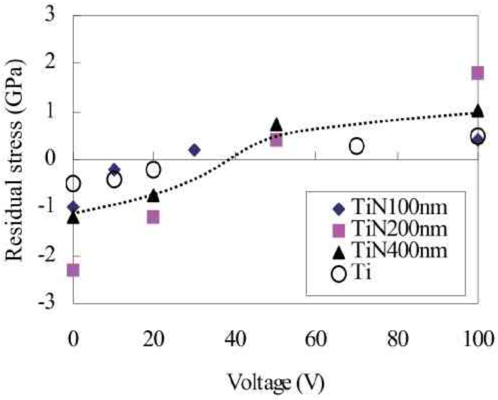


Fig. 6 Variation of residual stress and bias voltage in 100, 200, 400nm thickness of TiN films

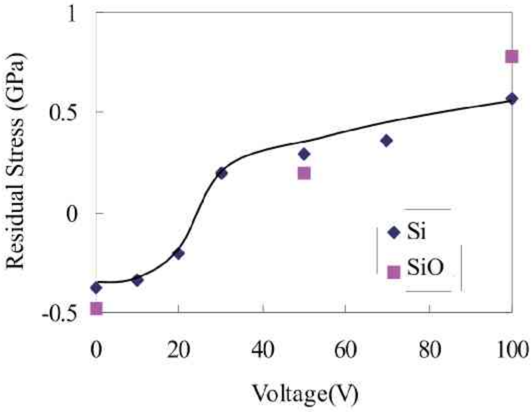


Fig.7 Variation of residual stress with bias voltage for Si and SiO_2 substrate.

3.1.2 Effect of kind of film

The above discussion showed that the residual stresses of Ti and TiN films with crystalline structure could be controlled by substrate vibration. In order to confirm the validity of the same method to Al and Cu films with crystalline structure and C and Si with amorphous structure, experiments were carried out. Fig. 4 and 5 show variation of the residual stress with bias voltage. It is clearly seen that the compressive residual stresses of Al and Cu films were decreased with the bias voltage. However, the residual stresses in C and Si films with amorphous strictures were almost constant at any range of the bias voltage.

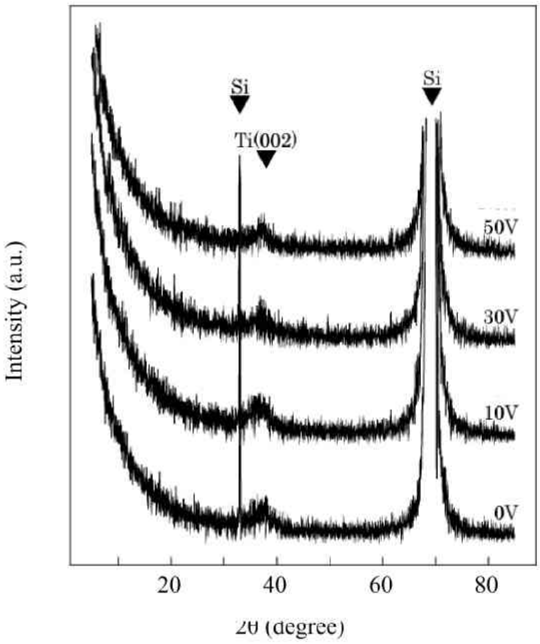


Fig.8 XRD patterns of Ti films on Si(100)with bias voltage.

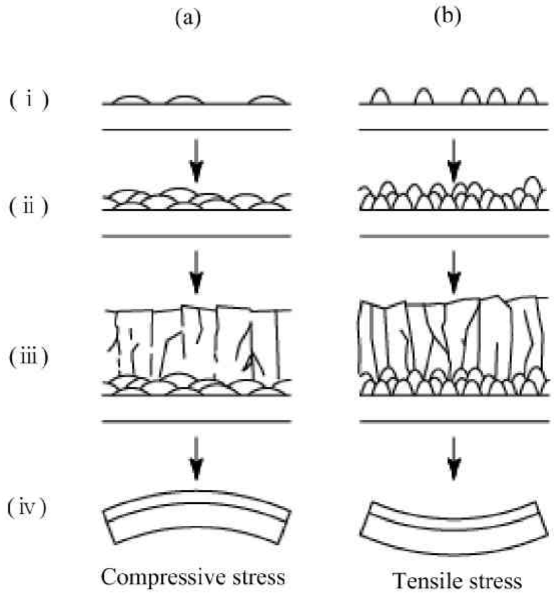


Fig.9 Schematic model for the film growth of crystal.
(a) without substrate vibration (b) substrate vibration

Therefore, substrate vibrations could change the residual stress in crystal films only.

3.1.3 The effect of thickness

Fig. 6 shows variation of residual stress with voltage in different TiN film thickness (100, 200 and 400 nm). The residual stress of each film thickness changed from compressive to tensile stress through zero stress. Therefore, substrate vibration could change the residual stress of any film thickness.

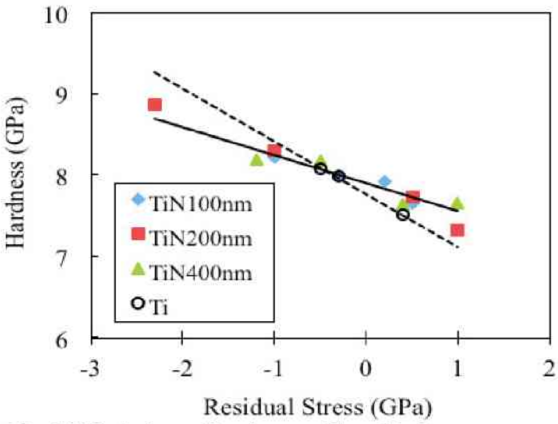


Fig. 10 Variations of hardness with residual stress

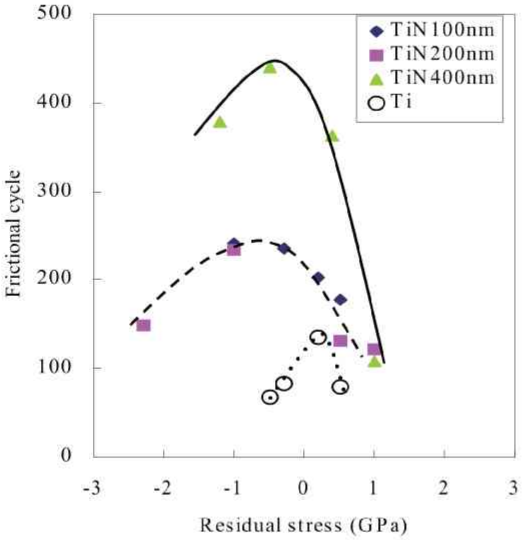
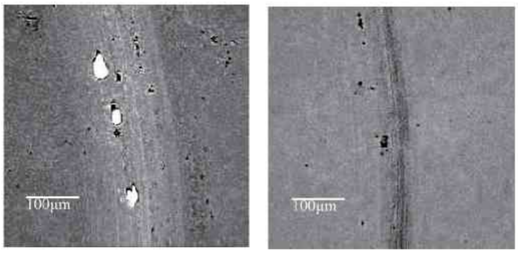


Fig. 11 Results of wear lifes



(a)
-2.4 GPa

(b)
0.4 GPa

Fig. 12 SEM image of wear track after 100cycle in TiN 200nm

3.1.4 Change of residual stress crystalline structure of substrate materials

Fig. 7 shows variation of the voltage and residual stress of Ti films on Si(100) (crystalline) and SiO_2 (amorphous) substrate. The residual stress of Ti films on SiO_2 substrate, changed from compressive to tensile through 0 stress when the bias voltage was increased. Therefore, The crystalline structure of substrate material was independent of the change of the residual stress.

3.1.5 Consideration of mechanism for change of the residual stress by substrate vibration

Variation of residual stress by substrate vibration is independent of the crystallization of the substrate and film thickness. The residual stresses were changed from compressive to tensile stress when the bias voltage is increased. Variation of the composition and crystal structure with bias voltage were constant in all crystal films. Fig. 8 shows the result of XRD for Ti films. The peek does not shift, but broadens when the bias voltage is increased.

A model for the cross section of the film growth is shown in Fig. 9. The model for the case in which substrate vibration is used and that of without vibration is shown in Fig. 9 (a) and 9 (b), respectively. When the substrate is not vibrated during the film growth, the crystal size tends to be fairly large leading to compressive stress on the film layer. On the other hand, when vibration is used during the film growth, the crystal size is much smaller and thus leads to the occurrence of tensile stress on the film layer.

3.2 Relationship between mechanical properties and residual stress in Ti and TiN films.

3.2.1 Variation of hardness with residual stress

in Ti and TiN films with various films thickness. In both Ti and TiN films, the hardness was decreased when the residual stress was changed from compress and tensile stress. The gradients in Ti and TiN films were 0.6 GPa/GPa and 1.0 GPa/GPa, respectively.

3.2.2 Frictional coefficient

The frictional coefficient was determined by ball-on-disk tribo-tester. The average frictional coefficient was calculated from tests taken between 50 and 100 cycles. The frictional coefficients of both Ti and TiN films were not changed with the residual stresses. This demonstrates that the frictional coefficients are not affected by the residual stress.

3.2.3 Wear life

The wear tracks were observed upon increasing the frictional coefficient to 0.6, which corresponds to frictional coefficient between Si (100) and steel ball. Fig 11 shows variation of residual stress and number of cycle. When the magnitudes of residual stresses in both compressive and tensile stress decrease, the wear life of the films increased in both Ti and TiN films. Fig. 12 shows SEM image of the wear track after 100cycle, TiN 200nm films with residual stress of (a) –2.4GPa and (b) 0.4 GPa. High residual stress films (a) have peeling areas but low residual stress (b) films have no peeling area. This clarifies that control of residual of residual

stress by substrate vibration improve the wear life of the films.

4. Conclusion

In the present study, thin films of various materials were prepared on substrate that was vibrated during the deposition process. The effects of substrate vibration on the residual stresses and the mechanical properties were investigated. The following conclusions were drawn:

1) The residual stresses were changed upon the substrate vibration that is controlled by the bias voltage
2) Changing residual stress is independent on film thickness and crystal stricture of the substrate
3) The hardness values decreased when the residual stress was changed from compressive to tensile for both Ti and TiN films.
4) The wear life was long when the residual stress was decreased for both Ti and TiN films.

5. References

[1] C.A.Carrasco, V. Vergara S, R.Benavente G., N. Mingolo, J.C. Rios, Materials Characterization **48** (2002) 81-88

[2] S.Zamir, B.Meyler, E.Zolotoyabko, J. Salzman, Jounal of Crystal Growth **218** (2000) 181-190