Effects of Plasma Immersion Ion Implanted and deposited layer on Adhesion Strength of DLC film

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ABSTRACT

Effects of ion implantation on the adhesion strength of DLC film as a function of ion doses and implanted energies were investigated. Ti ions were implanted on the Si-wafer substrates followed by DLC coating using ion beam deposition method. Adhesion strength of DLC films were determined by scratch adhesion tester. Morphologies and compositional variations at the different ion energies and doses were observer by Laser Microscope and Auger Electron Spectroscopy, respectively. From results of scratch test, the adhesion strength of films was improved as increasing ion implanted energy, however there was no significant evidence with ion dose.

Keywords : Adhesion Strength, Diamond-like Carbon(DLC), Plasma Immersion Ion Implantation and Deposition(PIIID), Scratch Test

1. Introduction

Diamond-like carbon (DLC) films are of considerable research interests because of their widespread applications as protective coatings in areas such as optical windows, magnetic storage disks, car parts, biomedical coatings and as micro-electromechanical devices [1-5].

Although DLC films have been considered as a strong candidate for various applications due to its excellent mechanical properties, their poor adhesion on tool steels limits the applications. In order to overcome this drawback, some ideas were introduced such as the sputter cleaning of the substrate surface [6] and the insertion of interlayer like Si [7], Ti [8,9] between film and substrates. Recently, many results were reported that the surface treatment of ion implantation played an important role in improving the adhesion strength of DLC film.

In this work, we investigated effects of Ti ion implantation on the adhesion strength of DLC films as a function of implanted energies and ion doses.

2. Experimental Details

Metal-Plasma Immersion Ion Implantation (Me-PIII) and DLC deposition has been performed using a Hybrid coatings System (Fig.1), which was equipped with a filtered vacuum arc source and a pulsed high voltage generator for Me-PIII and an ion-gun for DLC films. A pulsed high voltage generator is capable of delivering pulses of up to 50kV, of 10~100μs in pulse length at a repetition rate of up to 1kHz. The substrate was ultrasonically cleaned in acetone and dried with high purity N_2 gas. The chamber was evacuated up to less than 9.9×10^{-4} Pa and than preheated up to 120℃. To remove the surface native oxide and organic matters, the substrates was cleaned by argon ion bombarding with -600V of bias for 10 minutes. Prior to DLC deposition, Ti ions were irradiated onto substrate with different ion doses and implanted energies, 6.36×10^{16}, 1.27×10^{17}, 1.91×10^{17},2.54×10^{17} Ions/cm² at -20kV for bias and -10, -15, -20kV for bias for 15minutes, respectively.

DLC films at approximately 1μm thickness were deposited on ion irradiated surface using end-hall type ion-gun with C_2H_2+Ar mixed gases. Substrate bias for DLC deposition was -80V in this experiment. For evaluating effects of Me-PIII on the adhesion of DLC film, we compared

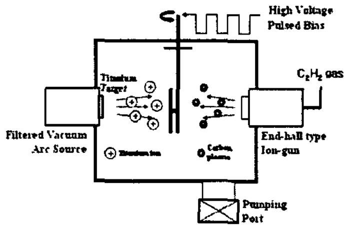

Fig.1 Schematic of Hybrid Coating System

was determined by a nano-indentation, maximum indented depth was fixed with 0.1㎛. The stress in the deposited films was calculated from the radii of curvature of the silicon strips of 100㎛ in thickness before and after deposition using well known Stoney's equation(1);

$$\sigma = \frac{E_s}{6(1-\mu_s)} \frac{t_s^2}{t_f} \left(\frac{1}{R} - \frac{1}{R_0} \right) \qquad (1)$$

The composition of the films and the ion implanted surface of the substrate were measured by Auger Electron spectrometry and the structure of the films was analyzed by Raman spectra. A CSEM-REVETEST scratch tester with diamond tip with a radius 200㎛ was used to asses the adhesion strength. The load range was 0-30N and the diamond tip approaching(loading) speed was

Table 1. Processing parameters for each experiment.

100N/min and the sample moving speed was 10㎜ /min.

3. Results and Discussion

3.1 Physical and mechanical properties.

Thickness of DLC films controlled by changing deposition time on the Ti ion implanted surface. Fig.2 shows the variation of roughness of DLC film surface measured by AFM. As increasing the ion dose and implanted energy, the roughness became larger. It is considered that titanium ions were implanted into the substrate surface at the first stage of PIII process, then, make a Ti film on the surface.

Fig.3 shows Raman spectra of DLC films on Si substrates that were implanted with various ion energies and doses. There is no significant difference between normal DLC film and metal ion implanted substrate prior to DLC deposition. It indicated the structure of DLC films on ion implanted substrate was similar with that of normal DLC film.

Fig.4 and Fig.5 show the mechanical properties such as residual stress and micro-hardness of Ti ion implanted layer and DLC film as a function of ion energies and ion doses, respectively. The stress of specimen was significantly lower than that of DLC film without Me-PIII treatment.

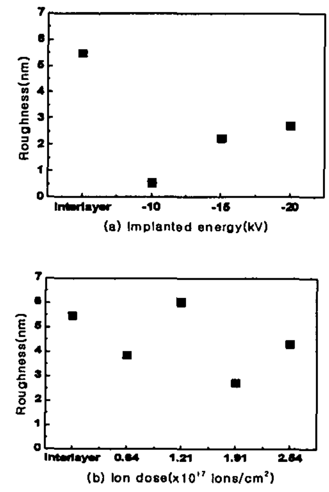

Fig 2. Variation of roughness of DLC films as a function of (a) ion implanted energies and (b) ion doses.

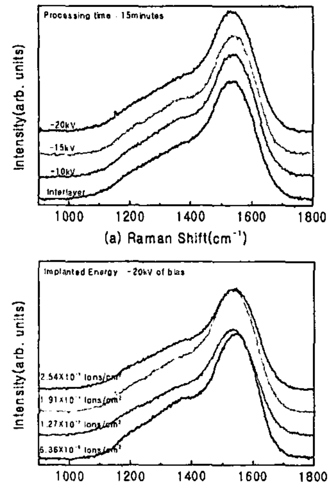

Fig 3. Raman spectra of DLC films on Ti ion implated surface as a function of (a) ion implanted energies and (b) ion doses.

Mirco-hardness, however, gradually increased with ion energy. Residual stress and micro-hardness much decreased as increasing the ion dose also.

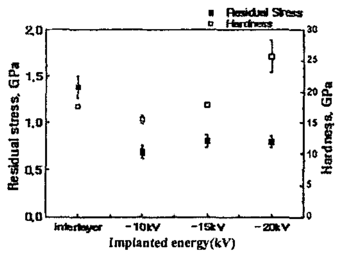

Fig 4. Variation of residual stress and hardness of samples as a function of ion implanted energies.

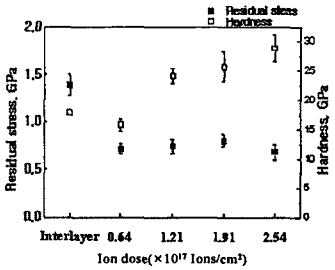

Fig 5. Variation of residual stress and hardness of samples as a function of ion doses.

3.2 Adhesion strength.

The scratch adhesion test is widely used to estimate the adhesion of films deposited on soft of hard substrates. In this method, minimum load at which the failure of the film occurs is defined as the critical load, Lc. The critical load is generally used for the estimation of the film adhesion. However, the critical load is strongly affected by several parameters related to testing conditions and to film/substrate system, such as, scratching speed, loading rate, indenter radius, indenter wear, substrate hardness, film thickness, friction coefficient and residual stress in film [10]. Even if many researchers have reported to estimate the interfacial adhesion strength from the critical load [11]-[13]. We evaluated the adhesion in a qualitative manner from the fracture morphology in the scratch track and acoustic emission signal. Adhesion of the coating was determined by five scratch tests for each specimen.

Fig.6 shows the scratch track of DLC films coated on Ti interlayer and implanted surface as a function of ion implanted energies. Without an intermediate layer and Me-PIII processing, the film was spontaneously delaminated from the substrate when the being removed from the deposition chamber. However, introducing Ti interlayer between the film and the substrate and Ti ion irradiating prior to DLC deposition shows a beneficial effect on the adhesion of the coatings.

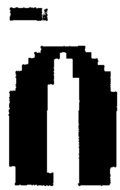

Fig 6 LM image of microstructure scratch track of samples as a function of ion implanted energies.

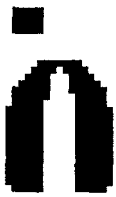

Fig 7 LM image of microstructure scratch track of samples as a function of ion doses.

Especially, the critical load increased with ion energies of Ti. Fig.7 shows the scratch track as a function of ion doses. There is no improvement of adhesion strength of DLC films even high ion dose.

After scratch test, we observed the composition of fracture area of our samples. Fig.8 and Fig.9 show the SEM image and the results of AES analysis of without and with Me-PIII, respectively. In Fig.9, we observed that there was mixing layer(Ti+Si) different from Fig.8.

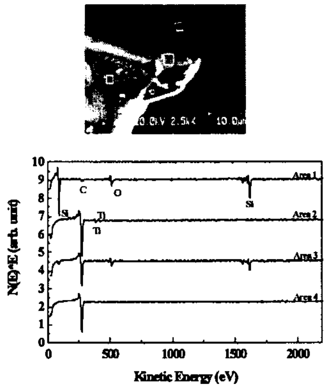

Fig 8 SEM image and AES spectra of fracture area of scratch track of sample without Me-PIII..

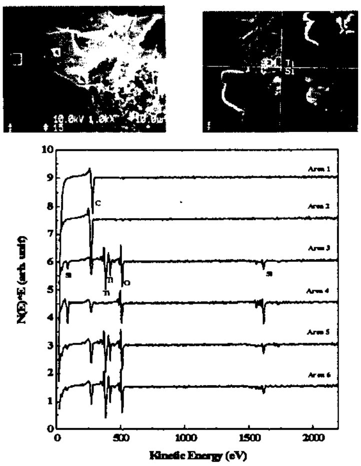

Fig 9 SEM image and AES spectra of fracture area of scratch track of sample implanted energy with-20kV.

4. Conclusions

The experimental results and observations from this study have lead to the following conclusions:

1)The residual stress are decreased but micro-hardness are increased as a function of ion implanted energies and ion doses.

2)The adhesion strength between DLC film and substrate is improved as a function of ion implanted energies. However, there is no significant improvement as a function of ion doses.

This work shows that pre-treatment of metal-plasma ion implantation processing prior to DLC deposition can be used to produce the adherent DLC film.

Acknowledgements

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