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Development of C_{60}/TiN composite film with excellent mechanical properties

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Abstract
In this study, we tried to establish the synthesis technology of the composite TiN thin films containing dispersed C_{60} molecules in order to apply practical uses in the industry fields. We developed an our original homemade vacuum apparatus with RF magnetron sputtering source for TiN and heating vapour deposition source for C_{60} considering to prevent from the influence of plasma. The microstructures analyses clarified the dispersed existence of C_{60} molecules contained in TiN based films. Nano-indentation studies demonstrated cleary that the hardness of the composite film of 2.0 wt.%C_{60}/TiN showed the maximum hardness of 18 GPa and this value was increased up to 40 % larger than 13 GPa of TiN film. Furthermore, both frictional coefficient and wear as tribological properties of the composite film also improved up to 40 %.

Keywords: Dispersed C_{60} molecules /TiN composite film, Unique thin film synthesis system, Improvement of hardness and tribologyical properties

1. Introduction

We developed dispersed C_{60} molecules/Al nano-composite thin films using by a conventional vacuum evaporation method. The microstructural characterizations of the composite films obtained clarified the uniform dispersion of C_{60} molecules in Al based film. Nano-indentation hardness of 1.0 wt.%C_{60}/Al showed surprisingly increase up to 3 times larger than that of Al film. This result has been considered that dispersion of C_{60} molecules with great high hardness in the conventional films contributes to drastic improvement in mechanical properties[1]. In this study, we challenged to improve greatly mechanical properties of TiN thin film already widely used industrially. We tried to establish the synthesis technology of dispersed C_{60} molecules/TiN composite films for the purpose of development of the coating technologies more further.

2. Experimental processes

First, we developed our thin film synthesis vacuum chamber combined with both RF magnetron sputtering source for Ti and heating evaporator for C_{60} as shown in Figure 1. Especially, we devised the arrangement of the heat evaporator of C_{60} molecules enough due to attempt the maintenance of the molecular configuration from the collision of the plasma. It was possible to control the deposition rates of two kinds of evaporation sources separately by preliminary experiments adjusted to arbitrary compositions. TiN films with constant thickness of 100 nm were deposited on Si(100) substrates by water cooling for 30 minutes. The concentrations of C_{60} molecule powder were changed in the range of 0, 1.0, 2.0, 3.0, 15 and 50 wt.%. The other synthesis conditions of the composite films are described in Figure 1. The microstructure, hardness and tribological property of each composite film obtained was analysed using by optical microscope, SEM, X-ray diffraction method(XRD), FT-IR analysis, nano-indentation system and pin-on-disk type tribotester in an ambient environment, respectively.

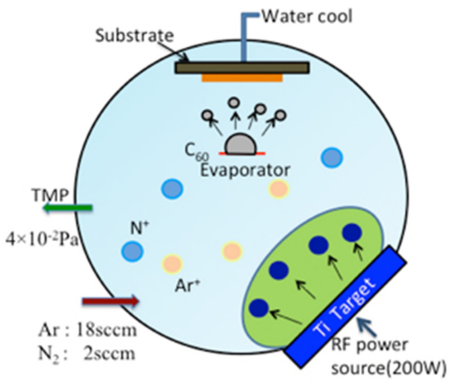

Figure 1. Our original vacuum chamber for fabricating composite films

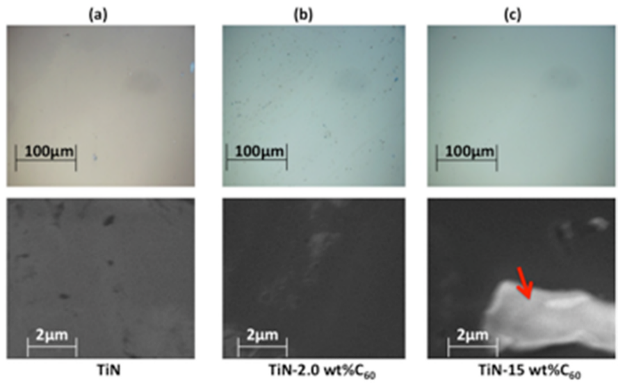

Figure 2. Typical surface images by optical microscope and SEM

3. Results and discussion

3.1. Microstructures

The upper row and the lower row of Figure 2 show the typical surface images using optical microscope and SEM, respectively.

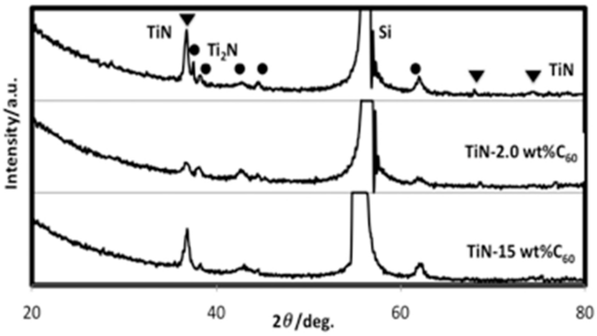

Figure 3. Comparison typical XRD profiles of C_{60}/TiN composite films

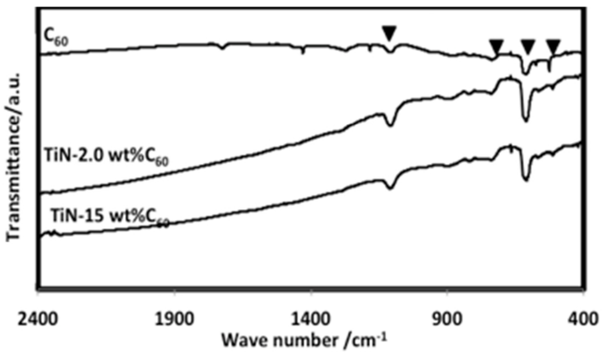

Figure 4. Comparison typical FT-IR profiles of C_{60}/TiN composite films

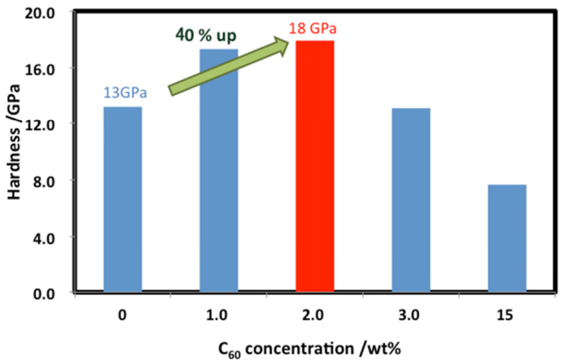

Figure 5. Change of nano-indentation hardness according to C_{60} wt%

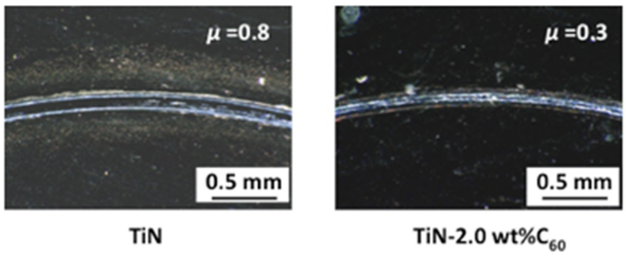

Figure 6. Typical effect of 2.0 wt%C_{60} with highest hardness on frictional coefficient and wear characteristic

The optical microscope images show uniform surfaces regardless of the composition ratios. On the contraly, SEM images show that the existences of foreign substances like precipitation are confirmed in the thin film of 15 wt.%C_{60}. This phenomenon could be observed for the composite films over 3.0 wt.%C_{60}. These foreign substances are guessed aggregations of C_{60} molecules or Al-C compounds. In order to clarify the realities of the foreign substance, typical XRD and FT-IR analysis were carried out for all composition films as shown in Figure 3 and 4, respectively. Both results reveal clearly that all composite films consist of only Ti-N system crystalline structures and C_{60} molecules. So it was clarified that foreign substances observed over 3.0 wt%C_{60} must be aggregations of C_{60} molecules. From view of microstructures, microstructure of the composite films of 2.0 wt.%C_{60} or less can be identified as the composite films with uniform dispersion of C_{60} molecules.

From the above-mentioned results, we made the preferred outcome of synthesis of C_{60}/TiN composite thin films.

3.2. Mehanical properties

Nano-indentation experiments for all films were carried out in the range of maximum indentation loads between 0.5 mN and 2.0 mN. AFM observations (not shown) of all impressions on the films showed no cracks and mean roughness below 10 nm. So there are no effects of the cracks and surface roughness on nano-indentation hardness obtained. The variation of nano-indentation hardness with C_{60} concentration from typical analysis of force-displacement results for is shown in Figure 5. The hardness increases from a value of 13 GPa of TiN film up to a maximum value of 18 GPa for 2.0 wt%C_{60} corresponding to an increase of about 40%, and then it is in the decreasing tendency down to a value of 7 GPa of 15 wt%C_{60}. It should be noted that superior increase of hardness must be attributed to the effect of dispersion of C_{60} molecules in TiN films and decrease of hardnesse after the maximum value condition can be considered due to softer aggregation parts of C_{60} molecules discussed in microstructure discussions above.

The typical comparison frictional properties between TiN film and 2.0 wt.%C_{60} composite film with the highest hardness were examined using a stainless steel ball of 5 mm in diameter were slid at a normal load of 0.5 N and a sliding speed of 2 mm/s. Figure 6 shows the friction coefficients(μ) and the wear tracks images after 300 cycles. It is clearly shown that the frictional coefficient decreases from 0.8 for TiN film to 0.3 for 2.0 wt.%C_{60} composite film and the width of wear track changes narrower. The cross-sectional wear track area was decreased from 11.4 μm^2 to 7.8 μm^2. Both tribogical properties of 2.0 wt.%C_{60} composite film also much improved up to about 40%. It can be considered that these great tribological properties of this composite film are also caused from the synthesis of uniform dispersed C_{60} molecules/TiN composite films with much improved high hardness.

4.Conclusions

The method of dispersed C_{60} molecules/TiN composite films with excellent mechanical properties were developed using by our homemade simultaneous deposistion apparatus with both a RF sputtering source and a heat evaporator. We clarified that the optimum concentration of C_{60} molecule was 2.0 wt%. The mechanical properties of hardness and the tribologyical properties greatly increased up to about 40 % in comparison with a conventional TiN film. We believe that C_{60} molecules must be applied as for innovative development of new composite materials and should be applied to many industrial fields.

References

[1] Itoh R, Takagi M, Iwata H, Matsumuro A 2011 *Japan.Proc. Jpn. Soc. Prec. Eng. Fall Symp. JP. Prec.* 517