Journal of Ceramic Processing Research. Vol. 17, No. 1, pp. 50~53 (2016)

J O U R N A L O F
Ceramic Processing Research

Characteristics of Pt thin films on WC substrate fabricated by ion beam-assisted DC magnetron sputtering for a lens glass mold

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Pt thin films with a Cr or Ti interlayer were deposited onto a tungsten carbide (WC) substrate by ion beam-assisted direct current (DC) magnetron sputtering. The percentage composition of Cr and Ti underneath the Pt films varied and the complete thin film characteristics were used to categorize specimens. Microstructure and surface analysis of the specimens were conducted by scanning electron microscopy (SEM), X-ray diffractometry and atomic force microscopy. Mechanical properties such as hardness and adhesion strength of Pt thin films were also examined. Interlayers of pure Ti or Cr were formed with thicknesses of 40 nm and 50 nm, respectively. The growth rates of both Cr and Ti thin films were similar when exposed to the same deposition conditions. Analysis of the specimen SEM images revealed that all Pt thin films investigated, irrespective of target composition, had anisotropic grains with a dense columnar structure. Hardness and adhesion strength values of the Cr/Pt thin films were higher than those of the Ti/Pt thin films.

Key words: Pt, Ion beam assisted DC magnetron sputtering, Molding core, Tungsten carbide.

Introduction

Recently developed camera modules for mobile devices, digital camera lenses, and other optical communication modules use optical components fabricated by ultra-precision machining and molding technology.

In particular, aspheric glass lenses helped resolve issues of distortion and spherical aberration, resulting in increased demand for these lenses. Plastic aspheric lens and spherical glass lenses, however, are still difficult to with several minimizing optical systems because of their inherent material properties and geometric aberration.

Typically, these plastic lenses are fabricated from a light, brittle molding core, which is compressed in a high-temperature environment. The use of this fabrication method has caused, ultra-precision grinding of core molds and thin film coating technology to become very important and urgent issues [1-3]. Pt-coated hard metal cores with aspheric profile surfaces are drawing special interest because Pt coating of the glass lens mold improves the life-time of a molding core, given its attractive properties of high elasticity and hardness, wear resistance, and chemical stability. Most importantly, these thin layers enable easy detachment of glass from the mold. Thus, many researchers from various fields are investigating this technology as a platform with potential to be widely applied in industry [4-6].

In this study, we investigated the impact of buffer layers on the thin film deposition of Pt in terms ion beam-assisted DC magnetron sputtering.

Experiment

The material used in this study was commercially available tungsten carbide (WC) FB01 provided by DIJET (Japan) and made from tungsten (94%), carbon (5%), and cobalt (1%); its average grain size was approximately 1.0 μm. Specimens used for the thin film coating had a diameter and thickness of 10 mm and 3 mm, respectively, and were cut by a diamond cutting machine. The high hardness of the WC necessitated, wet polishing of the samples with diamond plates of mesh #800, #3000. These specimens were polished using diamond pastes with sequentially decreasing grain size from 1.0 μm to 0.25 μm to remove tool markings. Residual organics and impurities remaining on the surface of the WC specimens after the initial removal process were eliminated using ultrasonic clearing involving sequential 15-minute incubations in alcohol and acetone. The samples were then, rinsed in distilled water, dried and kept in a vacuum chamber to prevent further contamination.

Figure 1 shows the polymer vapor deposition (PVD) coating equipment composed of an ion gun for cleaning the prepared specimens and for magnetron sputtering. This equipment deposits a thin film using the DC magnetron sputtering deposition method. During the coating process, as shown in Table 1, the

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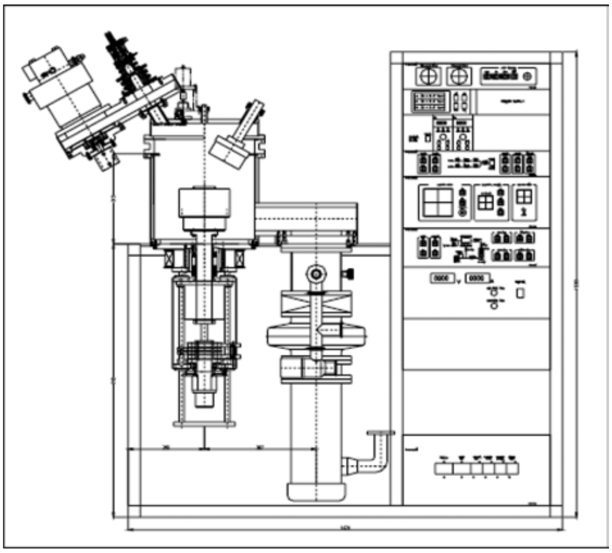
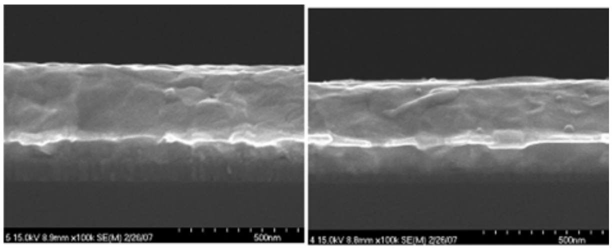


Fig. 1. Schematic of an unbalanced DC Magnetron Sputter with Ion Gun.

Table 1. Parameters of Ion Bombardment and Sputter Deposition.

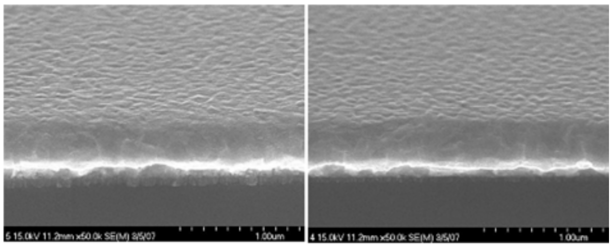
Deposition Parameter	Value
Deposition method	Unbalanced DC Magnetron Sputter
Target	Cr, Ti, Pt (Ø: 4", t: 6 mm)
Substrate	Si (Ø100 mm), wafer, WC (Ø10 mm, t: 3 mm)
Base pressure	5.0 × 10-6 Torr
Working pressure	1.0 × 10-3 Torr
Gas rate(Ar)	9.0 sccm
Target power	200 W
Deposition temperature	300 °C
Target-to-Substrate distance	380 mm
Substrate bias voltage	−80, −100, −150 V rotation
Jig rotation	5 rpm

distance between the target and the test specimens was 380 mm. Targets 4-inches in diameter and composed of Ti (99.9% purity), Pt (99.9% purity), and Cr (99.9% purity) were used. The deposition chamber was filled with ultra-high purity argon gas. To release residual stresses and eliminate cobalt (Co) the specimens, were annealed in the chamber for 30 minutes (5 × 10-3 mTorr, 600°C). Following annealing, samples were again plasma-cleaned to eradicate surface impurities and oxidation layers capable of degrading the quality of the film coating. The properties of the obtained thin film coatings were investigated through X-ray diffractometry (XRD, RIGAKU, DMAX/1200). To quantitatively analyze the compositional quality of the thin films, electron spectroscopy for chemical analyzer (ESCA, VG Multilab 2000) was used and compositions at varing depths were evaluated. A field emission scanning electron microscope (FE-SEM, S-4700) and atomic force microscope (AFM, Nanoscope-III) were used to measure the surface roughness, and too observe the cross-section and measure the thickness of the coating. To evaluate the hardness and the adhesion behavior of the thin films under a critical load condition, a nano-identer (Nano-identer XP, MTS systems) and a scratch tester (Revetest, CSM instrument), respectively, were used.



(a) Cr(50nm)+Cr/Pt(117nm)+Pt(300nm)
(b) Ti(40nm)+Ti/Pt(111nm)+Pt(250nm)

Fig. 2. SEM images of cross-sections of Pt thin films on Si substrate: (a) Cr/Pt and (b) Ti/Pt.



(a) Cr/Pt (b) Ti/Pt

Fig. 3. SEM tilt images of coatings obtained from different target materials: (a) Cr/Pt and (b) Ti/Pt.

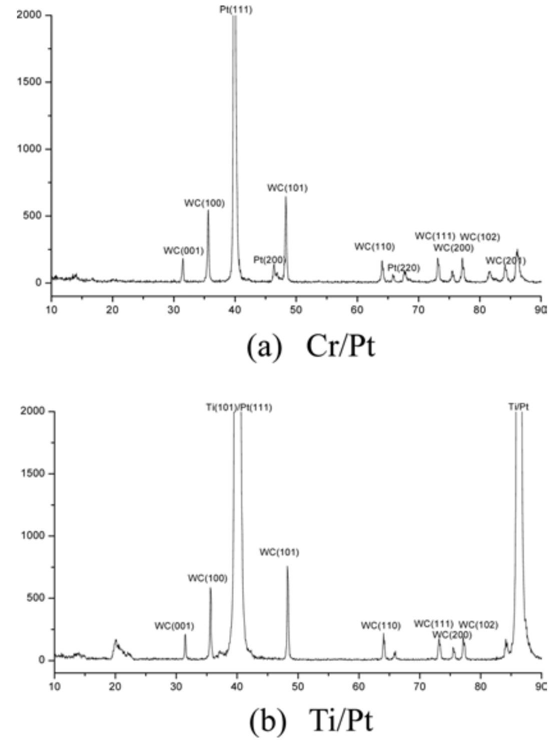


Fig. 4. X-ray diffraction patterns of (a) Cr/Pt and (b) Ti/Pt thin films coated on a WC substrate by ion beam-assisted DC magnetron sputtering.

Experimental Results

Micro and Nano structures of the coated thin films.

Figure 2 is a SEM image of a cross-sectional view of the coating, and displays its thickness and crystal structure. After Ar ion bombardment, deposition of the buffer layer followed by the top layer occurred. These layers resembled the Zone 3 area, where volume expansion is a major mechanism of mass transfer, such that the active movement of atomic momentum increased and equiaxed grain formation occurred [7]. To study the surface characteristics of the sample, we utilized tilt analysis and found that sputtering resulted in Pt films with a low surface roughness and a large equiaxed grain structure as seen in Figure 3. In Figure 4, the X-ray diffraction patterns, analyzed with a JCPDS card, show the preferred orientations of the thin films. Pt naturally has three preferred deposition directions: (111), (200), and (220). In Figure 4(a), Pt has a preferred growth of (111), while the preferred direction in Figure 4(b) is (111) due to the additional effect from Ti (101).

In addition, the diffraction angles of all constituent materials have relatively wide widths of their respective diffraction peaks due to the existence of defects, fine strain, and fine crystallized structures [8]. Figure 5 shows the SEM images of Cr/Pt and Ti/Pt thin films deposited on Si substrate, along with their ESCA depth profiles. Unlike vacuum deposition, sputter deposition typically results in thin films with compositions that resemble the characteristic composition of the target. For this reason, Figures 5(a), and (b) reveal that the top layer was deposited with a composition similar to the single target, while the buffer layers, composed of their Ti or Cr, exhibited compositions resembling their respective targets. Additionally, these images also clearly show the existence of a mixed layer (Ti, Cr/Pt), formed from interactions between the top and buffer layers.

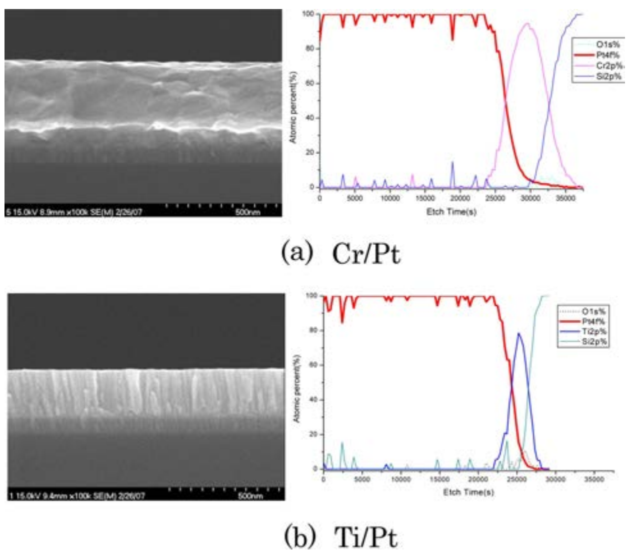


Fig. 5. ESCA depth profiles of (a) Cr/Pt and (b) Ti/Pt thin films coated on a Si substrate by ion beam-assisted DC magnetron sputtering.

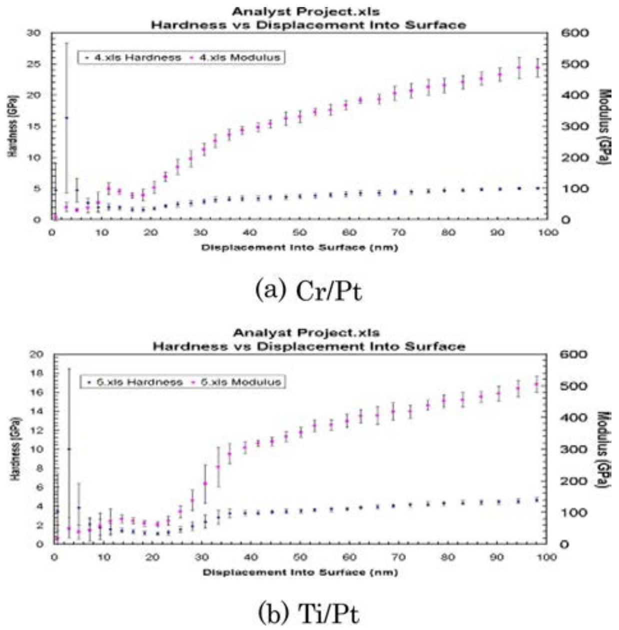


Fig. 6. Surface hardness and modulus of (a) Cr/Pt and (b) Ti/Pt thin films on a WC substrate as function of depth.

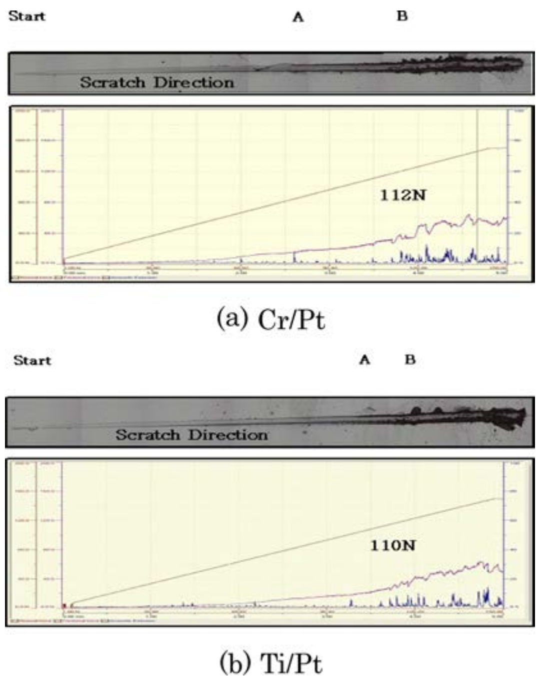


Fig. 7. Critical load (Lc) and scratch track of Pt thin films on WC substrate measured by scratch test.

Mechanical properties of coated thin films.

Hardness

Figure 6 displays the variations in the hardness and Young's modulus of Cr/Pt and Ti/Pt coatings while moving the probe from the surface to a depth of 100 nm. It also indicates the average hardness value of approximately 5 GPa for both coatings.

Adhesion of the thin films

Adhesion of thin films is affected by chemical composition, material structure, substrate reactivity, substrate surface roughness, and residual stress within the film. Additional external factors modulating film adhesion include load, temperature, humidity, and corrosive environment. Among various methods of measuring the adhesion of thin films, the scratch test method has recently been applied to thin highly adhesive layers to determine adhesion quality of very hard and wear-resistant coatings [9].

Figure 7 is the image captured through a microscope under critical load (Lc), as measured by scratch test equipment. The Lc value of the scratch track and for the thin film spalling was determined by comparing the acoustic emission peak. As shown in Figure 7, the critical loads for Cr/Pt and Ti/Pt thin films were 112 N, and 110 N, respectively. This result indicates that the adhesion strengths of both Pt thin films on glass-molded WC are comparable irrespective of the material of the buffer layers Cr or Ti.

Conclusions

1. SEM images of the cross-section view of the coated thin films obtained from a Pt target demonstrate that these films are characterized by a large equiaxed grain structure.
2. Nano-indenter measurements reveal that the hardness of both Cr/Pt and Ti/Pt thin films was approximately 5 GPa, which is relatively low for thin film coating.
3. Scratch test indicates that very minor differences exist in the of adhesion strengths of Cr/Pt (Lc:112N) and Ti/Pt(Lc:110N), which indicates that the adhesion strength of the materials are comparable.

Acknowledgments

This research was financially supported by the Korea Institute of Industrial Technology (KITECH).

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