Journal of Ceramic Processing Research. Vol. 13, Special. 2, pp. s328~s331 (2012)

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

Characteristics of Rhenium-Iridium coating thin film on tungsten carbide by multi-target sputter

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With the recent development of super-precision optical instruments, camera modules for devices, such as portable terminals and digital camera lenses, are increasingly being used. Since an optical lens is usually produced by high-temperature compression molding methods using tungsten carbide (WC) alloy molding cores, it is necessary to develop and study technology for super-precision processing of molding cores and coatings for the core surface. In this study, Rhenium-Iridium (Re-Ir) thin films were deposited onto a WC molding core using a sputtering system. The Re-Ir thin films were prepared by a multi-target sputtering technique, using iridium, rhenium, and chromium as the sources. Argon and nitrogen were introduced through an inlet into the chamber to be the plasma and reactive gases. The Re-Ir thin films were prepared with targets having a composition ratio of 30 : 70, and the Re-Ir thin films were formed with a 240 nm thickness. Re-Ir thin films on WC molding core were analyzed by scanning electron microscope (SEM), atomic force microscope (AFM), and Ra (the arithmetical average surface roughness). Also, adhesion strength and coefficient friction of Re-Ir thin films were examined. The Re-Ir coating technique has received intensive attention in the coating processes field because of promising features, such as hardness, high elasticity, abrasion resistance and mechanical stability that result from the process. Re-Ir coating technique has also been applied widely in industrial and biomedical applications. In this study, WC molding core was manufactured, using high-performance precision machining and the effects of the Re-Ir coating on the surface roughness.

Key words: Rhenium-Iridium, Thin Film, Tungsten carbide, Ra, PV.

Introduction

The recent expansion of the mobile market has increased the market for optical instruments, such as mobile device modules, digital cameras, and optical communication module lenses [1]. However, it is unrealistic to expect high-quality optical performance from the existing plastic or spherical lenses used in such mobile devices. Thus, improved optical performance is achieved through the production of non-spherical lenses that present little error caused by various aberrations generated by Lanes Assay in the glass material [2]. In general, non-spherical glass lenses are produced by the glass molding press (GMP) method, which compresses glass material in high-temperature, high-pressure conditions, and uses a core to form the desired optical structure [3]. Because the core in molded glass lenses must have a high hardness and durability of abrasion in high-temperature and high-pressure conditions, cemented carbide is generally used. However, major disadvantages presented by this method include the chemical changes generated in the contact part, in relation to the characteristics and molding conditions of the glass material, as well as the decreased lifespan caused by the shape abnormality reduced by the high temperature and pressure process [4]. To make up for such weaknesses, thin film of outstanding quality must be formed in the surface of the cemented carbide. Also, diverse studies are being conducted to achieve improvement [5, 6]. Re-Ir is the main method used in surface improvement technologies for thin film because it presents high hardness, chemical stability, and outstanding durability of abrasion that can be applied extensively in various industrial fields [7]. Thin film with high hardness, durability of abrasion, and chemical stability especially is required for the core used in lens molding to reduce shape abnormality and improve lifespan: Re-Ir coating is essential for this process [8]. In this study, WC molding core was manufactured using high-performance precision machining and the effects of Re-Ir coating on the surface roughness.

Experimental

This study cut 15ϕ diameter WC (FB01, DIJET Inc., Japan) into a thickness of 1 cm, considering the size of the deposition jig. Large error is generated according to surface characteristics, such as peak to valley (PV) and Ra of the WC used in glass lens molding. Thus, the WC surface was ground with a grinding machine (KRP-2200F, Kurda Co., Japan). As WC cannot be

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Table 1. Grinding conditions

Material	Tungsten carbide Diameter : 15 mm
Diamond paste (Mash)	#2000
Turbine speed	35,000 rpm
Work speed	350 rpm
Feed rate	0.25 mm/min

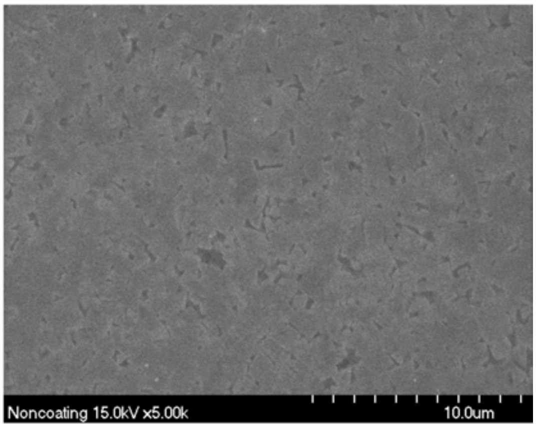


Fig. 1. SEM image of WC surface after completing grinding work.

ground by the general grinder because of its high hardness, a diamond plate was used to perform wet grinding. Grinding conditions are presented in Table 1.

Organic matter and impurities remaining on the surface of the ground WC specimen were eliminated, and the hardness and degree of molding were measured to compare changes in surface characteristics before and after Re-Ir deposition.

Re-Ir thin film presents superior hardness, elasticity, durability of abrasion, and chemical stability. This study used a DC magnetic sputtering technique to deposit Re-Ir thin film on WC. A DC magnetic sputter-installed cold-cathode saddle-field ion gun within a vacuum chamber and coil Ta line was used for a heater. The temperature of outflow cells was monitored by the thermocouple attached on the upper and lower part of the outflow cell for temperature control. For film growth, the distance from the specimen to the target was set at 350 mm, and Cr and Re-Ir were used for the target. The composition ratio of Re and Ir was set at 30 : 70. The degree of vacuum within the chamber was approximately 10^{-6} torr, while the temperature of the substrate was set as 450 °C.

For the formation of the Re-Ir thin film layer, Cr was first deposited at 50 nm, and Cr and Re-Ir were co-deposited at 5 nm, once again. This deposition process is commonly used as a method for increasing the adhesive strength between the hard metal and metalized film. Further, the adhesive strength between substances can be increased by co-deposition of Cr, Re, and Ir substances. The thickness of the Re-Ir thin film was set as 240 nm, and approximately 1 - 2 hours were sufficient for the cooling time after the deposition.

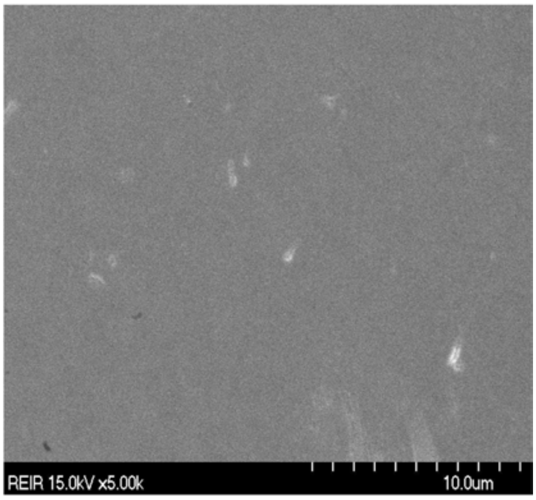


Fig. 2. SEM image of Re-Ir thin film.

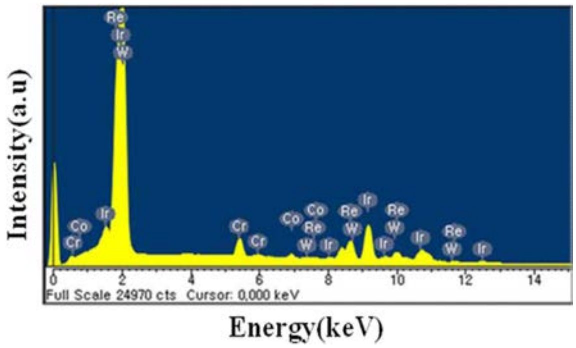


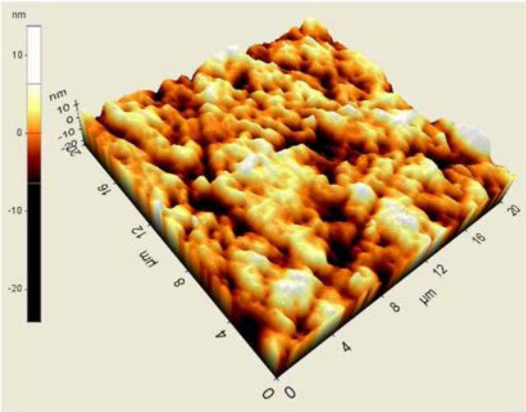
Fig. 3. EDXS analysis conducted on Re-Ir thin film.

Results and discussion

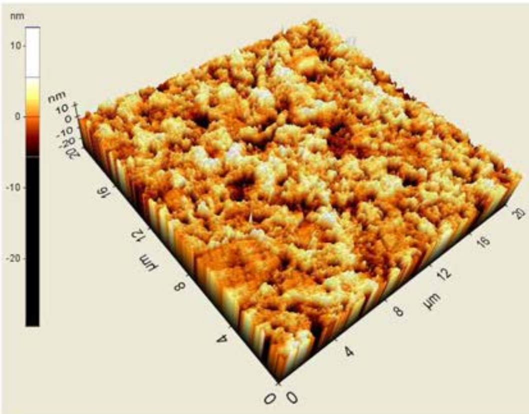
WC-cemented carbide is generally produced by burning metal carbide powder. WC (FB01, Dijet, Japan) was ground using a diamond plate and the surface of the ground WC specimen is presented in Fig. 1.

Tiny particles (within 1.0 um) were observed, even after grinding work, by checking the surface of the ground WC specimen. Re-Ir thin film was coated on ground the WC specimen. SEM images of a Re-Ir-coated WC specimen are presented in Fig. 2. Clear differences were presented in SAM images taken after Re-Ir coating. WC particles were not observed in the Re-Ir coating; it instead presented an even thin film formation.

Energy dispersive X-ray spectrometer (EDXS) analysis was conducted to check the substance composition of deposited Re-Ir thin film. Fig. 3 presents the results that Re-Ir was verified to be composed of 10.68% Cr, 2.80% Co, 4.29% W, 23.91% of Re, and 58.33% Ir. The Co and W extracted from the Re-Ir film deposition were seen in the WC specimen. Cr extracted from the Re-Ir film was presented by the Cr layer used as the intermediate layer to enhance the adhesive strength between the WC and Re-Ir thin film during deposition.

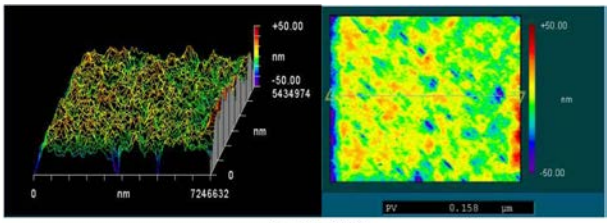


(a) AFM image of WC

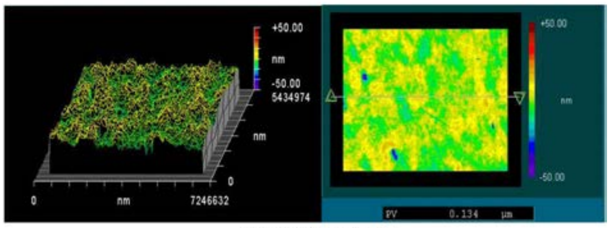


(b) AFM image of Re-Ir thin film

Fig. 4. comparison of AFM images between before and after Re-Ir coating.

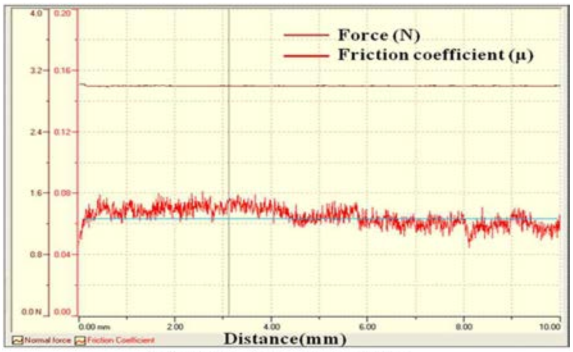


(a) PV of WC

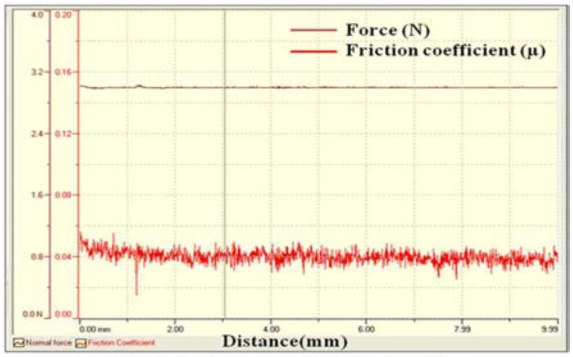


(b) PV of Re-Ir thin film

Fig. 5. Comparison of PV between before and after Re-Ir coating.



(a) Friction factor of grinded WC



(b) Friction factor according to Re-Ir coating

Fig. 6. Friction factors according to Re-Ir coating.

High-quality film was formed for Re-Ir thin film.

Ra and PV were analyzed to observe changes in shape abnormality caused by Re-Ir coating. The Ra before and after Re-Ir coating is shown in Fig. 4 (a) and (b). The average roughness of the surface of the Re-Ir thin film was reduced from 3.223 nm to 2.871 nm after coating. This signifies that Re-Ir coating triggered structural changes in the WC surface that reduced Ra and formed a smoother surface.

A 3D-surface measuring instrument was used to analyze PV before and after Re-Ir coating. The principle of optical coherence was used for the fine-surface form measured by the 3D-surface measuring instrument (NewView5000, Zygo Inc., USA) to measure the partial form without contact. The X axis of 7.25 mm and Y axis of 5.43 mm were measured in equivalent conditions, using 2.5 diameters. Fig. 5 presents the PV before and after Re-Ir coating. In the case of the Re-Ir thin film, PV was increased from 0.158 um to 0.134 um after Re-Ir coating.

Various external factors generally affect the adhesive strength of the film, such as the composition ratio and structure of the thin film and substrate, response between the film and substrate, Ra of the substrate, temperature, humidity, and corrosive environment. Indentation hardness and rebound hardness tests are used to measure the adhesion and hardness of thin film. Although these methods are effective for coating with low adhesion, it is difficult to measure coating with high adhesion. The scratch test method has been used recently for coatings with high hardness and durability of abrasion, this study also uses the scratch test to check the adhesion between the ground thin film and the WC, and the hardness of thin film.

Fig. 6 presents friction factors according to the WC specimen and Re-Ir thin film. A scratch tester (Revetest,

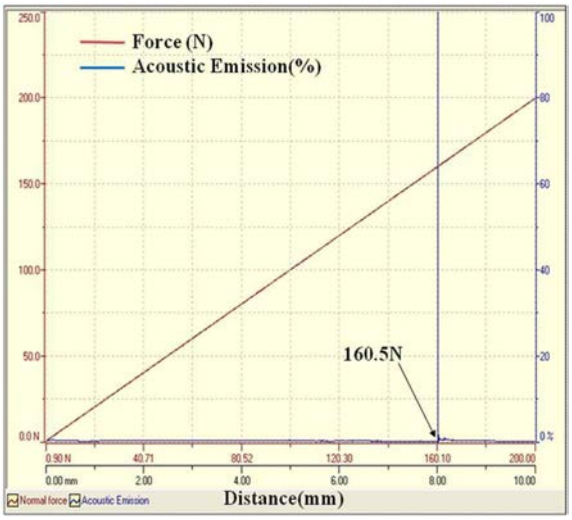


Fig. 7. Scratch test results of Re-Ir thin film.

CSM instrument, Swiss) was used for measurement, and a diamond tip with a diameter of 200 um was used as the indicator. The X axis presents the scan length, while the Y axis presents the friction-factor value and the strength exerted on the tip during measurement. The applied load used in the measurement of the friction factor was selected as 3 N, to be performed in identical conditions. The friction factor decreased to 0.039 μ after Re-Ir coating. Thus, it is judged that the reducing the friction factor would correct the surface-damage phenomenon that occurs during the Re-Ir coating and would result in a better surface than that with the ground WC material. A reduction in shape abnormality and an increase in the core lifespan are expected in lenses molded lenses after coating the Re-Ir in the WC material.

The scratch tester (Revetest, CSM instrument, Swiss) used in this study is composed of a stylus for exerting strength on the specimen in a vertical direction and a driving gear on the specimen support to move the specimen. The stylus can continuously increase the load, and a comparison is made between the coating layer desquamation within the formed scratch and the acoustic emission peak to measure the critical load. The scratch tester used in this study can measure 200 N as the maximum load and presents a 3 mN resolution. Similar to the friction factor measurement, the X axis presents the scan length, while the Y axis presents the friction factor value, strength exerted on tip during measurement, and acoustic emission value. Mechanical characteristics of Re-Ir thin film were analyzed through the scratch tester. The critical load value of Re-Ir thin film was shown to be 160.5 N, according to results of the scratch tester: this is slightly higher than the values reported in other studies, and thus verify the formation of outstanding ground film [1]. Scratch test results of Re-Ir film are presented in Fig. 7.

Conclusions

This study examined the characteristics and mechanical properties of Re-Ir thin film surfaces coated with Re-Ir to enhance the durability of abrasion and to reduce the shape abnormality after using ground WC in the molding of glass lenses with a high-temperature compression molding method. Cr target was used to enhance the suitability between the Re-Ir and the WC to first form the intermediate layer with a 50 nm thickness, and the alloy target with a Re-Ir composition ratio of 30 : 70 was used to coat the Re-Ir thin film of 240 nm. Re-Ir thin film was coated on ground WC, and the surface was observed through SEM. In the results, a noticeable decrease in particles was observed in the ground WC specimen. In particular, an uneven surface with unobserved particles was verified in the Re-Ir thin film. EDXS was used to perform a substance analysis. Re-Ir thin film was composed of 23.91% Re and 58.33% Ir, thus verifying that the film was mainly composed of main components. Further, Ra and PV were evaluated through AFM and a 3D-surface measuring instrument. For Re-Ir, the average roughness of the surface before coating was measured as 2.871 nm, and PV was measured as 0.134 um, but the average roughness was improved to 5.148 nm, and the PV was elevated to 0.112 um after Re-Ir coating. It is suspected that high-quality lenses could be produced using Re-Ir coating during lens molding. The friction factor of the WC that was only ground once before Re-Ir coating was measured as 0.062 μ.

The friction factor was decreased to approximately 0.039 μ after Re-Ir coating. Further, the critical load value of the composed thin film was measured through the scratch tester. The critical load value of the Re-Ir thin film was verified as 160.5 N. It is expected that the Re-Ir coating of WC material will not only reduce the shape abnormality the between surface and the lens used as the molding surface, but will also improve the durability of abrasion.

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