STRUCTURE AND MECHANICAL PROPERTIES OF R. F. REACTIVE MAGNETRON SPUTTERED TUNGSTEN CARBIDE FILMS

By

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TO MY PARENTS

CERTIFICATE

I am satisfied that the Thesis presented by P.K. Srivastava is worthy of consideration for the award of the Degree of Doctor of Philosophy and is a record of the original bonafide research work carried out by him under my guidance and supervision and that the results contained in it have not been submitted in part or full to any other university or institute for award of any degree/diploma.

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ABSTRACT

In the present work tungsten carbide films have been prepared by r.f. reactive magnetron sputtering on stainless steel substrates held in temperature range 200-500°C. Two different geometrical modes, normal mode and high rate mode, have been used to deposit various films. The effect of various parameters, like sputtering pressure, gas flow, substrate temperature, self bias developed on the cathode and the power applied have been optimized to obtain a hard, adherent film. Uniformly distributed fine grains (~200 to 400 A°) of tungsten carbide films with columnar structure having regularly spaced columns (~300 A°) have been obtained in these films. The films deposited in normal mode at lower substrate temperature (T_s = 200°C) have a mixture of phases (f.c.c. B1 WC, hexagonal W_2C and A-15 W_3C) while the films deposited at higher substrate temperature (~300 to 500°C) have a single phase (f.c.c. B1 WC). On the other hand films deposited in high rate mode show a mixture of phases (hexagonal WC, A-15 W_3C, graphite and diamond carbon) at all temperatures (300 to 500°C). The amount of diamond phase in the films deposited at higher temperature (~500°C) is found to be more than in the films deposited at lower temperature, whereas the amount of graphite phase is less in films deposited at higher temperature as compared to that in low temperature deposited film. AES study of these films suggests that these films are nonstoichiometric and have same composition throughout the thickness. AES sputter depth

profile of thick films shows a very wide interfacial region due to intermixing of various species during sputtering process.

Microhardness, adhesion, friction and wear properties of these films have been reported. It is observed that the films prepared in the normal mode of deposition have very high microhardness value ~ 3200 Kgf/mm^2, whereas the films prepared in high rate mode have microhardness ~ 2365 Kgf/mm^2. The bulk hardness value of tungsten carbide is ~ 1800 Kgf/mm^2. Under indentation load the films crack and a sequence of crack propagation with increasing load has been observed. The nature of crack propagation suggests that the films deposited in high rate mode at T_s = 500°C are more brittle than the films deposited in normal mode at T_s = 500°C. Adhesion of the films has been measured by crack indentation method and a value ~ 3.5×10^7 $grams/cm^2$ has been observed in tungsten carbide films (~5.6 μm thick) deposited in normal mode.

Friction and wear of these films have been measured by pin and disc machine. It is found that the films deposited at 500°C in normal and in high rate mode have small coefficient of friction ~ 0.13 and 0.09, respectively. The wear rates of tungsten carbide films deposited at T_s = 500°C in normal and high rate mode deposited on stainless steel are observed to reduce by a factor 20 as compared to bare substrates.

Thin layers (~1000 A°) of titanium, tantalum, tungsten, silicon and molybdenum have been deposited on stainless steel substrate by r.f. diode sputtering. A mixture of phases (TiC (cubic) and TiO (cubic)) has been observed in titanium films, whereas a single face b.c.c. structure of tantalum, tungsten and molybdenum films have been observed. Silicon films are found to be amorphous. Thin layers (~1000 A°) of tungsten carbide films have been deposited in normal mode on coated stainless steel substrate. AES depth profiling of these interlayer films show a wide intermixing (~1500 A°) at the interface due to knock in effects. Complete mixing in the case of tantalum, tungsten, molybdenum interlayers has been observed. ESCA study suggests that the interlayer films are transformed in their respective carbides and no alloy formation between tungsten carbide/interlayer element/stainless steel has occurred.

Microhardness, adhesion, friction coefficient and wear resistance of the tungsten carbide films deposited on interlayer coated stainless steel substrate have also been reported. An improvement in wear life ~ 5 is observed in tungsten carbide-interlayer coated stainless steel substrates.

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