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Structures and properties of Y-ZrO₂ thin films deposited by reactive r.f. sputtering

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Abstract- The Y-ZrO₂ thin films were deposited by r.f. magnetron sputtering method. The microstructure, phase composition and mechanical properties of the thin films were investigated. The studies show that the films had tiny grains, tetragonal phase, and the best properties when they were deposited at the condition of the low substrate temperature and zero oxygen partial pressure.

1. Introduction

Zirconia thin films have advanced mechanical, thermal, optical and electrical properties that made them interesting for theoretical studies and practical application (1-7). Bulk ZrO₂ has three kinds of phases: low temperature monoclinic (M), high temperature tetragonal (T) and cubic (C). Alloying certain stabilizers, such as Y₂O₃, MgO, CaO, and CeO, the T or C phase can exist at room temperature. Recently, the martensitic transformation (T→M) in the bulk materials had been found to be able to improve the fracture toughness and wear resistance (8). Furthermore, a strengthening overcoat of ZrO₂ thin films had been made (4,5). However, they had not got the T phase in the films. A Y-TZP target had been used in our study, and Y-ZrO₂ films containing T phase had been made.

In this paper, the microstructure, phase, and mechanical properties of the Y-ZrO₂ thin films were investigated. The studies show that the thin films have tiny grains about 88 Å, tetragonal phase (60-900 °C), the higher microhardness (2100 kgf/mm²), and the lower wear rate (473 nm²/cycle).

2. Experiment

Y-ZrO₂ thin films were deposited by the reactive r.f. magnetron sputtering. The target was a tetragonal ZrO₂ polycrystal doped with 2.2 mol% Y₂O₃ (Y-TZP). The spacing of the substrate-target was 4 cm. The base pressure was below 8×10^{-4} Pa. A mixture of the argon-oxygen gas at the required partial pressure ratio was introduced. The oxygen partial pressure (%O₂) and the substrate temperature (Ts) were controlled during deposition. The substrates were the Si wafers or the fused silica.

The microstructure and phase of the films were examined by transmission electron microscopy (TEM) and x-ray diffraction (XRD). A quantitative evaluation of the amounts of monoclinic and tetragonal phases was made by the method (9).

The knoop hardness of the films which were deposited on the Si wafer was measured by using a nMT-3 indenter instrument. The wear rate of the films was tested under nonlubricated conditions, using a pin-on-disk tester which applied a 15 g normal force against the rotating disk through a 10.0 mm radius hemispherical pin. The linear speed of the tested area was 10 m/s.

3. Results and Discussion

3.1 Microstructure and phase

The study shows that the films deposited at low Ts and %O₂ have mainly the T phase (72% T) with a small amount of the M phase. As the increments of the Ts or %O₂, the amount of M phase increases, from 28% to 56%, 59%, and 63%. Fig.1 gives their XRD patterns.

Here the T phase identified by not only the XRD data but also a ED pattern. The (200) and (220) peaks in the ED pattern were splitted into doublet. This is the character of the T phase rather than the C phase.

TEM photographs show that the films possess tiny grains. The grain size of the films was calculated from the line broadening of the XRD pattern (10) and it was found be 88Å.

3.2 Film properties

The hardness of the films decreased with increasing $\%O_2$, as shows in the Fig.2. The films deposited at 0.0 ($\%O_2$) and 60C (Ts) exhibit the maximum hardness of 2100 kgf/mm². Because the thickness of the films just was 5000-6500Å, so the values of the hardness included the effect of the Si(100) substrates, but the effect of the substrates was invariant. Substrate heating leads to the relaxation of the film structure and the reduction of compressive stress in the films and results in the hardness drop.

The wear rate of the films increased with increasing $\%O_2$, Fig.3. The films have the highest resistance against wear failure, or the lowest wear rate 473 nm²/cycle when the hardness reaches maximum value.

Fig.1. XRD patterns of Y-ZrO₂ thin films deposited at Ts=60°C with $\%O_2$ =0.0% (a), 3.3% (b), 13.0% (c), and Ts=170°C with $\%O_2$ =13.0% (d)

Fig.2. Variation of knoop hardness of Y-ZrO₂ thin films as a function of the %O₂

Fig.3. Variation of wear rate of the Y-ZrO₂ thin films as a function of the %O₂

4. Conclusions

The Y-ZrO₂ films deposited by r.f. sputtering from Y-TZP target possess mainly tetragonal phase with a small amount of monoclinic phase. However the monoclinic phase increased as increasing %O₂ and Ts.

When the %O₂=0.0% and Ts=60 °C the films have tiny grains about 88 Å the maximum hardness 2100 kgf/mm², and the lowest wear rate 473 nm²/cycle.

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