

Structure evolution and mechanical properties enhancement of Al/AlN multilayer

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Abstract

A set of Al/AlN multilayers with various modulation periods were prepared using DC magnetron sputtering method. Low angle X-ray diffraction (LAXRD) was used to analyze the layered structure of multilayers. The phase structure of the films was investigated with grazing angle X-ray diffraction (GAXRD). LAXRD results indicate that well-defined multilayer modulation structures are formed for the relatively larger modulation periods. However, the loss of multilayered structure is detected in the multilayer with low modulation period. A very wide amorphous peak is observed in multilayer with modulation period of 4 nm. The multilayers show obvious crystallization at larger modulation periods, however, the diffraction peaks are much wider than the Al single layer because of the interruption of the continuous columnar grain growth by alternating deposition processes. Nanoindentation experiments were performed to study the mechanical properties as a function of multilayer modulation period. It is found that the hardness of the multilayers is greater than the hardness calculated from rule of mixtures. With the modulation periods adjusted, the multilayers are even harder than its hard component (AlN). A maximum hardness of 24.9 GPa, about 1.9 times larger than its hard component (AlN) and 3.7 times larger than the hardness calculated from the rule of mixtures, is found at the multilayer with modulation period of 16 nm. The wear test results show that the multilayers possess lower and stable friction coefficient, and superior wear properties.

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1. Introduction

Classical hard metal nitrides coatings, such as TiN and CrN, have received considerable attention as wear-resistant coatings because of their high mechanical hardness, wear resistance, and chemical inertness [1,2]. However, the requirements in many applications are so high that classical monolayered hard coatings cannot satisfy them anymore. Recently, a great deal of attention has been paid to compositionally modulated multilayer films, especially transition metal nitride ceramic/ceramic multilayers, with each layer on the nanometer scale because of their possibility to achieve superhardness [3–5]. The properties of nanoscale metal/metal-nitride multilayers can be quite different from that of micron level multilayers even if they have the same components proportions [6–9]. Multilayer designed of

metal/metal-nitride components is a flexible approach because it makes it possible to simultaneously tailor the microstructure of the surface layer to optimize bending strength and wear resistance, and the microstructure of the inner layers optimizes toughness and flaw tolerance [10,11]. Otherwise, such metal/metal-nitride multilayers can be made in a simple manner, using a single sputtering target, by controlling the flow of the reactive gas used to form the interlayers [6–8,12].

Aluminum is a well-known material for its characteristics: excellent ductility and low density. Aluminum nitride (AlN) may be used in the composite structures containing aluminum for either structural or electronic applications due to its attractive thermal, electronic, and mechanical properties [13,14]. AlN ceramics is also known to have a sufficiently high temperature compatibility with refractory metals. Finally, AlN is an ecologically safe material. The structures and mechanical properties of AlN as a ceramics layer of the multilayer Al/AlN composites have not been well studied [7–9]. In this paper, we prepared nanoscale alternative multilayers

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of a hard component AlN with a soft component Al using DC magnetron sputtering technique. We presented our research results about the structure evolution, and the mechanical properties of Al/AlN multilayer with different modulation periods. The aim of this work was to investigate the influence of modulation periods on the structural and mechanical properties of Al/AlN nanoscale multilayers.

2. Experimental procedure

The Al/AlN multilayers were fabricated with DC magnetron sputtering deposition technique with a columnar target on Si wafers. Details of the deposition system are described elsewhere [15,16]. This technique can be used in large-scaled industrial deposition. Prior to deposition, the Si substrates were first ultrasonically cleaned in acetone and then dried with blowing air. The substrates were mounted on the sample holder, and transferred into the sputtering chamber. The sputtering chamber was then pumped to the achieved base pressure of $1.0\text{--}2.0 \times 10^{-3}$ Pa. During the deposition processes, the argon flow rate were kept at 40 sccm, the source power at 500 W, and the deposition pressure at 0.45–0.5 Pa for Al layer, while for AlN layer the nitrogen flow rate were kept at 50 sccm, the source power at 750 W, and the deposition pressure at 0.70–0.75 Pa. The Al/AlN multilayers were deposited through alternating both experiment conditions. The change from Al to AlN deposition was achieved by controlling the nitrogen flow without plasma interruption and a no less than 30 s transition time for every reactive gas change process, and at the same time the substrate was kept reversed to the columnar target, so that a sharp graded interface was generated. For all the multilayer specimens, the first layer deposited was Al layer to improve the adhesion, and the last one was AlN layer to improve the film oxidation resistance. A set of Al/AlN multilayers ($t_{\text{Al}}:t_{\text{AlN}} = 2.71:1$) no less than 500 nm of total film thickness, with modulation periods, ranging from 2 to 24 nm, were produced. A single AlN film and a single Al film were also deposited under same process parameter as reference samples.

In this study, low angle X-ray diffraction (LAXRD) was performed on D/Max-2400X diffractometer (Rigaku Co., Japan) at Cu K α wavelength, to obtain the diffraction peak profiles of the multilayer thin films along the vertical direction. Grazing angle X-ray diffraction (GAXRD) with a grazing angle of 1° was applied for phase identification and qualitative texture characterization. The hardness of the films was determined using a nanoindentation system (NanoTest 550, MicroMaterials Ltd.) equipped with a Berkovich diamond tip, with the maximum indentation depth being kept at 50 nm (less than 20% of the total film thickness) to minimize the substrate contribution. The hardness and reduced elastic modulus were obtained by analyzing loading-displacement curves. At least five indents were made on each sample, and adopted the average value. The wear test of the films was investigated using a ball-on-disk micro-tribometer (UMT-2MT, CETR Co., US) with 3 mm GCr15 steel ball (6.1 GPa of hardness). A normal load of 1 N was applied. The sliding speed was 100 rpm with a wear track length of 6 mm. The tests were performed in

ambient atmosphere with temperature from 20 to 28 °C, humidity from 40 to 60%. After the wear tests, the wear tracks were examined by optical micrograph.

3. Results

The architecture of the multilayers can be examined by means of LAXRD analysis and the low-angle patterns are really useful to accurately determine the modulation period. Typical LAXRD patterns obtained from Al/AlN multilayers for modulation periods with 4 and 12 nm, respectively, are shown in Fig. 1, from which the modulation period calculated is closed to the determined modulation periods. For the multilayers with determined modulation period of 2 nm, there are no obvious reflections observed in LAXRD scans, which suggests a loss of multilayered structure at this low modulation period. This result can be interpreted that large interface roughness caused by this deposition method and small layer thickness (the determined thickness of AlN layer was only about 0.65 nm) could impede the forming of continuous AlN layers but AlN islands instead, and finally caused the destruction of multilayer modulation structure. The higher orders of satellite peaks do not appear in multilayer with modulation period of 4 nm, which suggests that the interfaces are not as sharp as the ideal atomic-level smooth interface multilayers because of the large interface roughness caused by the large grain size of Al in multilayers. For multilayers of larger modulation period (such as multilayer with modulation period of 12 nm), the diffraction peaks are very weak and low order peaks are submerged in the strong background. For the modulation period larger than 12 nm, no obvious low angle diffraction peaks are detected because of the strong background. Small difference in X-ray scattering factors between the Al and AlN also caused the weak interface reflections.

GAXRD analyses were used to study the phase structure of Al/AlN multilayers with various modulation periods. Fig. 2 shows the GAXRD patterns corresponding to the Al/AlN multilayers with modulation periods in the range of 4–24 nm, and monolithic Al single layer. For the multilayer with modulation period of 4 nm, the individual layer is too small so that no obvious Al and AlN diffraction peaks but only very wide

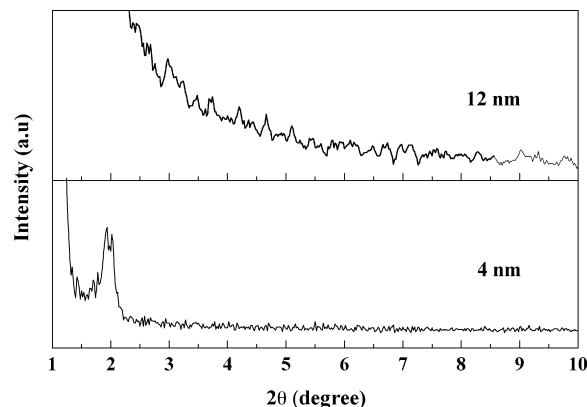


Fig. 1. Low angle diffraction patterns of Al/AlN multilayers with different modulation periods.

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