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Structural, mechanical and piezoelectric properties of polycrystalline AlN films sputtered on titanium bottom electrodes

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ABSTRACT

Polycrystalline AlN coatings were deposited on Ti-electrode films by reactive magnetron sputtering. During the deposition, processing parameters such as the reactive gas pressure and time of deposition have been varied. The purpose was to obtain an optimized AlN/Ti system coating with suitable properties for applications such as piezoelectric sensors, which could monitor the wear rate and the remaining coating life of a specific part. The chemical composition, the structure, and the morphology of the multilayered films were investigated by X-ray photoelectron spectroscopy, X-ray diffraction, scanning electron microscopy and atomic force microscopy techniques, respectively. These measurements showed the formation of highly (1 0 1), (1 0 2) and (1 0 3) oriented AlN films with piezoelectric and mechanical properties suitable for the desired purpose. A densification of the AlN coating was also observed, caused by lower nitrogen pressures, which has led to an improvement of the crystallinity along with an increase of hardness. The coating stability at high temperatures was also examined. Consequently, an improvement of the piezoelectric properties of the AlN films was observed, inferred from the enhancement of *c*-axis (0 0 2) orientation after annealing. Furthermore, the mechanical characteristics (hardness and Young's modulus) were significantly improved after heat treatment. These two parameters decrease rapidly with the increase of the indentation depth, approaching constant values close to those of the substrate after annealing. Thus, thermal annealing promotes not only the rearrangement of Al–N network, but also a surface hardening of the film, caused by a nitriding process of unsaturated Al atoms.

1. Introduction

The wear mechanism of a tribological system is very complex. There are two broad approaches concerning the study of wear: the first is based on describing the effect on tribological performance of various parameters like counterpart, coating (if any), substrate, application conditions and environment, while the second is based on the physical nature of the underlying processes. The solution to wear related problems should start with a detailed examination of the tribological system with all the factors that are involved. Even if these factors are not constant, possibly leading to the premature failure of the system, it is very important for a designer to know the amount a component can be worn before it must be replaced. In the case of coated parts, wear monitoring of the coating could provide the coating status life and also a warning before the coating failure occurs, thus conferring a higher degree of reliability to the tribological system. Embedding a piezoelectric layer between two electrodes into a tribological coating system to monitor the stress variation within the material could be an appealing technique for in situ wear monitoring and, consequently, would potentially help to develop new materials by providing additional information about wear mechanisms.

Aluminum nitride AlN is used in many applications, in microelectronic and optoelectronic devices such as ultraviolet detectors, light emitting diodes [1], thermal interface materials [2] and piezoelectric materials in surface acoustic wave devices [3]. Several techniques like molecular beam epitaxy (MBE), chemical vapor deposition (CVD), pulsed laser deposition (PLD) and reactive sputtering [4–7] can be used to deposit AlN thin films. This type of compound is a promising candidate as a piezoelectric material, which, considering its other remarkable properties (large optical

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Table 1
Deposition parameters of Ti/AlN coating-systems.

Deposition parameters	Ti electrode films	AlN piezoelectric films		
		Series no.1	Series no. 2	Series no. 3
Target material	Ti – 99.99% purity	Al – 99.99% purity	Al – 99.99% purity	Al – 99.99% purity
Sputtering power	1.2 kW	1.5 kW	1.5 kW	1.5 kW
Working pressure	5×10^{-4} mbar	5×10^{-4} mbar	5×10^{-4} mbar	5×10^{-4} mbar
Gas pressure	Ar – 0.25 mbar	N_2 – 0.1 mbar	N_2 – 0.05 mbar	N_2 – 0.1 mbar
Deposition rate	0.8 μm/h	1.2 μm/h	1.2 μm/h	1.2 μm/h
Sputtering time	1 h	2 h	2 h	4 h
Substrate	Steel 100Cr6	Ti-film	Ti-film	Ti-film

band gap, high hardness and good thermal conductivity), should be successfully used in the fabrication of a piezoelectric wear monitoring sensor.

The deposition by sputtering of AlN films with controlled crystal orientation is one of the key issues intensely studied, considering the fact that the piezoelectric properties of such films are strongly dependent on the crystallographic structure. The influence of the deposition parameters such as target power, growth temperature, sputtering pressure and gas composition on the properties of aluminum nitride films has been reported, to some extent, for films deposited by DC [8,9] and RF reactive sputtering [10–14]. According to the literature it seems that the physical mechanism that determines the way in which the Al and N atoms are structurally arranged is related to the kinetic energy of the adatoms during the film growth [15]. At high kinetic energy the AlN films grow with the *c*-axis normal to the surface ((0 0 2) orientation). As the energy is decreased, the *c*-axis of the crystals gradually tilts away from the normal direction, resulting in other preferred orientations such as (1 0 3), (1 0 2), (1 0 1), and, eventually, (1 0 0) or (1 1 0) orientations [15]. Nevertheless, a convincing correlation between the sputtering parameters and the crystal orientation of sputtered AlN films, to the best of our knowledge, has not been reported yet. Furthermore, contrasting results can be found in the literature. For example, if the sputtering pressure is considered, Xu et al. [16] reported that the (0 0 2) texture is improved at low sputtering pressures, while Cheng et al. [17] suggested the use of a moderate pressure for the same purpose. If we consider the nitrogen flow, Cheng et al. [17] report the deposition of AlN films with high (0 0 2) orientation when using high nitrogen flows, while Okano et al. [18] reported that the *c*-axis orientation is improved by decreasing the nitrogen concentration in order to avoid the formation of AlN on the aluminum target (target poisoning). The majority of previous works concluded that the AlN films must exhibit high (0 0 2) orientation in order to obtain the best piezoelectric properties.

In this context, the purpose of the present work is to demonstrate that AlN polycrystalline films produced by DC reactive magnetron sputtering exhibit good piezoelectric properties when a high percentage of coating crystallinity is observed, despite its crystalline orientation. In addition to its piezoelectric properties, the successful fabrication of devices based on AlN thin films requires a better understanding of how the microstructure of the deposited material relates to the mechanical behavior of the film, which determines the practical use of a material. Moreover, if this piezoelectric device based on AlN/Ti films will be integrated into wear resistant coatings, then the as-deposited AlN film has to go through another deposition process to build up the tribological coating on top of it. Thus, the properties of AlN films might not only be influenced by the deposition parameters but also could depend on post deposition heat treatments, such as annealing. In this paper we will present our results concerning the changes in the structural, mechanical and electrical behavior of magnetron sputtered AlN films, subjected to heat treatments.

A series of AlN coatings were deposited using different sputtering conditions on Ti electrode films, in order to obtain an optimized AlN/Ti system coating, suitable for integration into wear resistant systems. Titanium has been selected as bottom electrode because it is a complementary metal–oxide–semiconductor (CMOS) compatible with AlN. Titanium exhibits in the crystalline state a lattice mismatch of 5% to AlN, as opposed to 23% in the case of pure aluminum, thus reducing the mechanical stress near the film interface [19]. The applicability of titanium as bottom electrode is also due to its excellent adhesion properties [21]. The feasibility of using sputtered Ti thin films as bottom electrode was reported elsewhere. Ababneh et al. [20] conclude that the Ti layer surface morphology variation, dependent on different deposition parameters, has a significant influence on the piezoelectric coefficients exhibited by the top AlN coating, synthesized under fixed sputter deposition conditions. Consequently, for this paper, the Ti underlayer was prepared using fixed sputtering parameters, limiting its influence on the AlN coating. This factor allowed us to correlate the change of structure and physical properties, shown by the AlN films, to the deposition parameters and subsequent post deposition heat treatments.

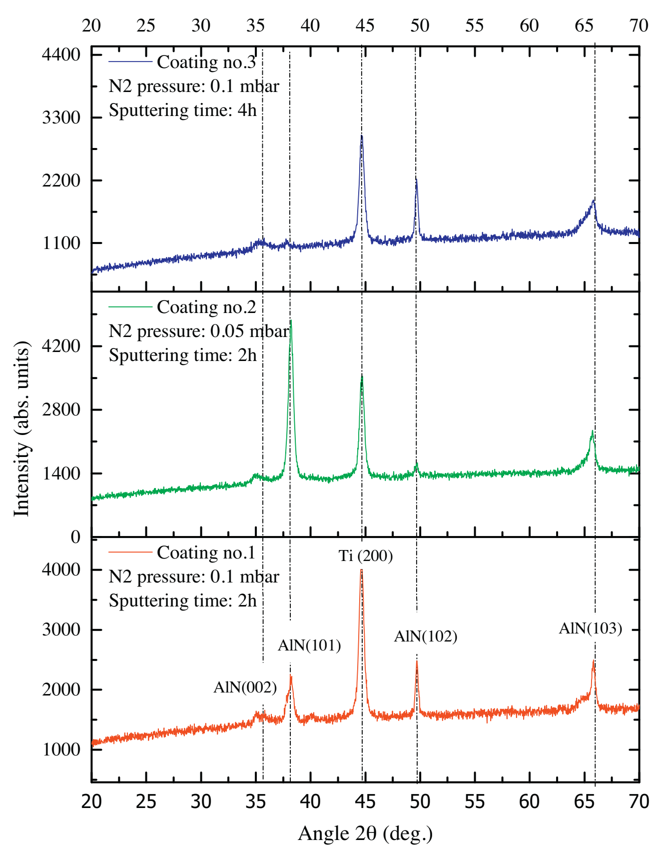

Fig. 1. XRD spectra of the as deposited AlN films.