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Effect of Ti Seed Layer on Microstructure and Surface Morphology of Cu Thin Film Postprint

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Abstract

Cu thin films without a seed layer and Ti/Cu thin films with Ti as a seed layer were prepared by magnetron sputtering. The microtexture of Cu thin films without a seed layer and Ti/Cu thin films with a Ti seed layer was investigated using electron backscatter diffraction (EBSD), and the surface morphology of both types of films was observed using atomic force microscopy (AFM). The results indicate that the addition of a Ti seed layer enhances the {111} fiber texture of Cu thin films and exerts a favorable epitaxial effect on film growth. Additionally, the Ti seed layer reduces the probability of annealing twin formation in the films after annealing treatment, but induces void formation during the annealing process.

Full Text

Effect of Ti Seed Layer on the Texture and Surface Morphology of Cu Thin Films

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Abstract: Cu thin films were deposited by magnetron sputtering on SiO_2 substrate without and with a thin Ti seed layer. The micro-texture and surface morphology of the films were characterized by electron back-scattered diffraction (EBSD) and atomic force microscopy (AFM). The results show that the Cu thin film deposited on the Ti seed layer exhibited a highly oriented {111} fiber texture, demonstrating excellent epitaxial growth. Meanwhile, the presence of the Ti seed layer could decrease the generation probability of annealing twins in the films after heat treatment, but voids appeared during the annealing process.

KEY WORDS: metallic materials, Cu thin film, Ti seed layer, texture, surface morphology

Introduction

In 1988, Peter Grünberg [1] and Albert Fert [2] independently discovered giant magnetoresistance (GMR) effects exceeding 50% in Fe/Cr/Fe and Fe/Cr multilayer systems, respectively. This breakthrough endowed GMR materials with broad application prospects in high-density read heads, magnetic sensors, and magnetic random-access memory, sparking intense research interest in GMR thin films within condensed matter physics and spintronics.

Extensive research has been conducted worldwide on GMR multilayers, with various approaches attempted to improve their structure and performance. These efforts primarily involve controlling thin film deposition parameters [3], adjusting annealing processes [4, 5], and incorporating seed layer materials [6, 7]. Seed layers play a crucial role in controlling grain orientation and modulating interface structure during film growth, thereby improving interface roughness [8-10] and film texture [11, 12] to enhance GMR performance. Bouziane et al. [13] introduced Fe, Cr, and Ta seed layers into GMR Co/Cu multilayers, demonstrating that interface roughness directly correlates with magnetoresistance ratio. Amir et al. [14] added Ag monolayers to Co/Cu multilayers to balance surface free energy on both sides of individual layers, creating sharper and more symmetric interfaces that improved thermal stability. Chihaya et al. [15, 16] found that Ti seed layers induced a transition from {100} to {111} fiber texture in Co/Cu multilayers as the Ti thickness increased, thereby affecting the magnetoresistance. These studies underscore the importance of understanding seed layer effects for optimizing GMR thin film performance. While seed layers are known to modify texture and interface structure, their influence on internal microstructural features such as grain orientation distribution, grain boundary misorientation, and the relationship between texture and surface morphology requires further investigation.

Co/Cu multilayers exhibit high room-temperature GMR ratios and promising applications. These periodic multilayers consist of alternating ferromagnetic Co and non-ferromagnetic Cu layers, making it essential to investigate how the microstructure and grain orientation of each layer type affect the overall system. This study employs EBSD to examine how Ti seed layers influence Cu thin film microstructure, complemented by AFM observations of surface morphology.

1 Experimental Methods

Cu thin films and Ti/Cu bilayers were prepared by magnetron sputtering at room temperature on thermally oxidized SiO_2 (100) substrates. The Cu film thickness was 100 nm, while the Ti seed layer thickness was 20 nm. The sputtering power was 250 W, the base vacuum was better than 1.0×10^{-5} Pa, and the sputtering pressure was 0.5 Pa. After deposition, the films were annealed at 400°C for 1 hour.

The texture of the 20 nm Ti seed layer was determined by pole figure measurements using a Bruker D8 diffractometer. Film microstructure was analyzed using EBSD in an ULTRA™ 55 field-emission scanning electron microscope operated at 20 kV, with a scan step of 0.02 m over a 3 m$\times$3 m area. The acquired data were processed using specialized software to obtain crystallographic information on local orientations and grain boundaries. Surface morphology was characterized using a Veeco Multimode 8 AFM to obtain three-dimensional topographic images and analyze surface roughness.

2 Results and Discussion

2.1 Effect of Ti Seed Layer on Cu Thin Film Grain Orientation

Pole figure measurements of the 20 nm Ti seed layer are shown in [Figure 1: see original paper]. The {0002} and {11-20} pole figures reveal a strong {0001} fiber texture in the Ti layer deposited on the substrate.

Orientation imaging maps of Cu thin films without and with Ti seed layers are presented in [Figure 2: see original paper], illustrating the distribution of grain orientations with crystal planes parallel to the film surface: blue represents {111} planes parallel to the surface, red indicates {100} planes, green denotes {110} planes, and black regions are unindexed. The Cu film without a seed layer shows randomly distributed grain orientations [Figure 2a: see original paper], whereas the film with a Ti seed layer exhibits predominantly {111} planes parallel to the surface [Figure 2b: see original paper].

Inverse pole figures of the Cu films are shown in [Figure 3: see original paper]. The film without a seed layer [Figure 3a: see original paper] contains grains with both {001} and {111} planes parallel to the surface. In contrast, the film with a Ti seed layer [Figure 3b: see original paper] develops a strong {111} fiber texture parallel to the surface.

The Ti seed layer also promotes {111} fiber texture formation in Cu films, as evidenced by the combined pole figure and orientation imaging data. This enhancement arises from two primary factors. First, the Ti seed layer has a hexagonal close-packed (hcp) structure, while Cu is face-centered cubic (fcc). Hcp and fcc structures readily form the Shoji-Nishiyama orientation relationship of (111)fcc||(0001)hcp, where the close-packed planes align parallel to each other. Consequently, Cu films grown on {0001}-textured Ti seed layers tend to develop {111} fiber texture. Second, the surface energy of Ti{0001} is 0.297 J/m^2, significantly lower than that of Cu{111} (1.952 J/m^2), Cu{110} (2.237 J/m^2), and Cu{100} (2.166 J/m^2) [17]. This large surface energy difference between the Ti seed layer and Cu film provides a strong driving force for {111} texture formation [18].

Additionally, lattice mismatch calculations based on the crystal constants of Cu, Ti, and Si reveal that the mismatch between Ti{0001} and Cu{111} is the smallest, as shown in . This minimal mismatch facilitates preferential Cu growth along {111} planes when deposited on Ti seed layers.

2.2 Effect of Ti Seed Layer on Cu Thin Film Grain Boundaries and Misorientation

The Ti seed layer influences not only the microtexture but also grain boundary formation in Cu films. After annealing at 400°C, the Cu film without a seed layer shows new grains within existing grains that are identified as annealing twins, whereas the film with a Ti seed layer exhibits no annealing twins.

Grain misorientation distributions obtained from EBSD analysis are presented in [Figure 4: see original paper]. The film without a seed layer shows a high frequency of 60° misorientations, characteristic of twin boundaries. In contrast, the film with a Ti seed layer displays predominantly low-angle grain boundaries ($<5°$ misorientation). Since low-angle grain boundaries have lower interfacial energy, their migration does not produce annealing twins. Furthermore, the Ti seed layer increases the stacking fault energy within the film, reducing twin formation probability and effectively alleviating lattice mismatch and thermal expansion coefficient differences between Cu and Si. Therefore, annealing twins are less likely to form in Cu films with Ti seed layers.

2.3 Effect of Ti Seed Layer on Cu Thin Film Surface Morphology

AFM observations of the 400°C-annealed Cu films reveal distinct surface morphologies [Figure 5: see original paper]. The film without a seed layer shows surface undulations and prominent island-like structures [Figure 5a: see original paper], while the film with a Ti seed layer exhibits a smoother surface without undulations or islands but with noticeable voids [Figure 5b: see original paper].

Refractory metals and elements with diamond cubic structures such as Si, W, Cr, Fe, and Ta are low-diffusivity materials. According to Abermann' s experiments on polycrystalline film deposition on various substrates [19], films deposited on these materials exhibit low atomic diffusivity even at high temperatures. Consequently, sputtered Cu films on Si substrates essentially "freeze" the initial island nucleation and growth structure, leading to further island-mode growth and the morphology shown in [Figure 5a: see original paper].

Conversely, Kamijo et al. [20] observed that Al films with strong {111} fiber texture developed on ultrathin metal seed layers exhibited very smooth surfaces. Kohama et al. [21] proposed that smaller wetting angles correlate with higher nucleation rates for {111} grains, and good wetting between film and substrate produces continuous films on Ti seed layers. The relationship between surface morphology and texture is evident when comparing the orientation imaging maps [Figure 2: see original paper] with the AFM images [Figure 5: see original paper]. The strong {111} fiber texture in Cu films with Ti seed layers corresponds to the lowest surface free energy. The surface energy γ relates to the wetting angle through the Young-Dupré equation, where lower surface free energy yields smaller wetting angles. Therefore, growth along {111} planes minimizes the wetting angle [22], and the developed texture reduces variations in wetting angles between adjacent grains, decreasing surface roughness. The root-mean-square surface roughness values are 2.6 nm for the Cu film without a seed layer and 2.4 nm for the film with a Ti seed layer. However, voids form during annealing in the Ti-seeded Cu film, creating the morphology shown in [Figure 5b: see original paper].

The insertion of a Ti seed layer between the Si substrate and Cu film modifies grain orientation and three-dimensional surface morphology by reducing the substrate' s surface free energy and providing a driving force for {111} fiber texture formation. The wetting angle is influenced by the surface energies of both substrate and film. Grains growing along {111} planes have the smallest wetting angle, resulting in smoother surfaces for {111}-textured Cu films. Since surface morphology and roughness significantly affect magnetic properties in GMR multilayer systems [23, 24], controlling film texture and surface characteristics by tuning substrate-film surface energy and lattice mismatch through different seed layers represents a promising approach for optimizing GMR performance.

Conclusions

1. The Ti seed layer, with its low surface free energy, provides a strong driving force for {111} fiber texture formation in Cu films, resulting in excellent epitaxial growth.
2. The Ti seed layer promotes the formation of low-angle grain boundaries with lower interfacial energy, whose migration does not generate annealing twins. Additionally, the Ti seed layer increases the stacking fault energy, reducing the probability of annealing twin formation in Cu films after heat treatment.
3. Texture formation influences surface morphology during film growth. Cu films without seed layers exhibit surface undulations and island-like structures, whereas {111}-textured Cu films with Ti seed layers show smoother surfaces without undulations or islands but develop voids during annealing.

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