Production of Beta-Tungsten as a Function of Sputtering Pressure

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Primary CNF Tools Used: MOS hood, AJA sputtering system #2, FleXus film stress measurement system

Abstract:

Metastable phases of tantalum and tungsten that are only present in thin film form are technologically important for the microelectronics industry and show promise for new magnetic memory applications with exhibition of the Giant Spin Hall Effect. Nonetheless, there has been considerable debate about the crystal structural and formation mechanism of these beta phases. Recent advances in the understanding of beta-tantalum provide a road map for how to bring similar clarity to beta-tungsten. A series of five films were made at varying Ag sputter pressures in the CNF AJA #2 sputtering system (3, 5, 6.8, 7, 7.8 mTorr). Substrate curvature was measured before and after each deposition to estimate the average film stress. Theta-2-theta XRD scans were used to examine the resulting crystal structure. Preliminary results show that highly tensile films (made at the higher sputtering pressures) were nearly entirely beta-tungsten, while the other films had significantly more of the alpha phase. While we have conclusively produced beta-tungsten, precise indexing of the theta-2-theta peaks will require higher precision XRD measurements and finder control over film stress generation.

Summary of Research:

This research on tungsten is inspired by recent work our group has done on tantalum [1]. Like tantalum, tungsten can form two phases, either alpha or beta. The alpha phases of both metals have a BCC crystal structure, are present in bulk form, have good electrical conductivity, and are relatively ductile [1]. The beta phases are metastable, found in only some thin films, and are brittle with poor electrical conductivity [2-6]. Both phases of tantalum are technologically important for the microelectronics industry. Additionally, the beta phases of both materials show strong Giant Spin Hall Effect (GSHE), which could be used in advanced magnetic memory devices [5-9]. Yet, even though tantalum has been studied for decades, the formation mechanism and crystal structure for beta-tantalum was only recently well established [1]. Reviewing the literature, it is interesting to note that the historical debates about the formation mechanisms of the metastable beta phases for each material mirror each other closely. Beta-tungsten, like beta-tantalum, for some time was believed to be an oxide rather than a distinct phase [2,6]. At other times, researchers believed that the beta phases of the two metals may share the A15 crystal structure [1,2,6]. In order to sort out the confusion, very carefully controlled deposition experiments need to be performed to understand the role of both sputter pressure and impurity atoms (especially oxygen). Eventually, it was shown that the actual crystal structure of beta-tantalum is a distorted Frank-Kasper sigma structure ($P\bar{4}2_1m$) and requires a template of tungsten oxide to form [1].

Our goal in this research is to explore the formation of beta-tungsten and determine if a similar formation mechanism exists as was discovered for beta-tantalum. The first step in this process is to determine deposition parameters that would reliably produce beta-Tungsten. To do this, the AJA #2 sputtering system was used. Five four-inch wafers were MOS cleaned (but retained the native oxide layer) and sputtered with a 99.995% pure tungsten target at 450 Watts at argon sputtering pressures of 3, 5, 6.8, 7, and 7.8 mTorr. These pressures were chosen based on the CNF supplied process data that showed that films in this pressure range could be expected to vary their stress widely from highly compressive (3 mTorr) to highly tensile (7 mTorr). Each wafer's curvature was measured with the CNF FleXus stress measurement system immediately before and after deposition to determine the average film stress. The films' thickness was then measured via contact profilometry and it's out-of-plane texture was investigated with XRD theta-2-theta scans to look for the presence of BCC alpha-tungsten or a more complex beta-tungsten crystal structure.

Preliminary results show that the 3 mTorr sample did develop compressive stress with little to no evidence of beta-tungsten. The other films produced high tensile stresses, with the films in the 6.8 to 7.8 mTorr regime possibly being composed almost entirely of the beta phase. It is hard to say with certainty in this preliminary data since the <110> and <220> alpha peaks are nearly identical in location to the <210> and <420> beta peaks which creates some ambiguity. However, we believe that these peaks can be indexed by carefully controlling film stress and shifting them through their stress-free positions. Previous work by Ellis, et al., in 2018 [1] showed that a peak can be unambiguously indexed by tracking its position as film stress changes and noting that it moves through the equilibrium position of a peak corresponding to one phase but not the other.

Future work will include both a sputtering pressure series to first be able to unambiguously index all the observed diffraction peaks, and then an oxygen partial pressure series to determine the amount of oxygen necessary for the formation of beta-tungsten.

References:

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