CONTROL OF CRYSTALLINE PHASE OF TANTALUM DEPOSITED ON STEEL BY MAGNETRON SPUTTERING

Anamika Patel, Charanjeet Singh Paur, Leszek Gladczuk, and Marek Sosnowski

Electrical and Computer Engineering Department, New Jersey Institute of Technology
University Heights, Newark, NJ 07102, U.S.

Tantalum coatings can effectively protect steel from corrosive and the erosive wear when the metal is in bcc, α-phase, characterized by toughness and high ductility. In deposition of films, however, hard and brittle tetragonal β-phase is usually formed [1], which is not desired in protective coatings because it leads to failure under stress. The meta-stable β-phase transforms to the equilibrium α-phase at 750 °C [2] but heat-treated steel, subjected to such high temperature, loses its desired properties. For the same reason sputtering is preferred for this application over chemical vapor deposition, which requires still higher temperatures [3]. The purpose of this work was to explore the range of sputtering process parameters that results in α-phase films on steel substrates held at temperatures below 400°C.

Depositions were carried out using a DC magnetron sputtering source with a 50 mm diameter target Ta (99.95% pure). The source was mounted in a high vacuum chamber pumped to the base pressure of $8 \cdot 10^{-8}$ Torr. Mass-flow controllers regulated flow of gases to the chamber and a residual gas analyzer monitored the gas composition during deposition. The distance between the target and a heated substrate holder was 50 mm. Substrates of 4340 steel were prepared by polishing (with 0.5 μm polishing diamond paste) and then cleaned with methanol and acetone in an ultrasonic cleaner. They were also baked at 200°C in high vacuum prior to deposition, together with control substrates of Si and SiO_2/Si. Several substrates (12.5mm x 12.5 mm) could be mounted on one holder and up to 7 holders were loaded on a revolving platter for one process cycle. Sputtering was carried out at pressures 1 - 20 mTorr, and flow rates 2 - 20 sccm. Ar or Kr gasses were used for Ta sputtering. In some experiments N_2 gas was added to the noble gasses to promote nitride formation in a reactive sputtering process. Typical target voltage was -300V and the power delivered to the target 150W. Deposition rate, determined from the measured film thickness and the deposition time, ranged from 1 to 5 nm/s.

The films were analyzed using x-ray diffractometry (XRD), four point probe resistivity measurements, and profilometry for thickness measurements. In addition, elemental analysis was performed on some samples with Rutherford back-scattering spectroscopy and nuclear reaction analysis.

The results show that films with tetragonal β-phase or with α-phase and β-phase mixture are readily formed on all substrates at room temperature in a wide range of process parameters. Pure α-phase films were successfully deposited on steel heated to 250°C and 300°C, when Kr and Ar sputtering gases were used, respectively. Films with pure α-phase were also obtained on steel, which was not heated, when a thin layer of nitrided Ta was formed the substrate prior to Ta deposition. Preliminary analysis indicates that the nitrided layer that promotes the growth of α-phase Ta consists of stoichiometric TaN. The advantage of this method, over a deposition of an interfacial metal layer to promote the growth of α-phase, is that nitriding by reactive sputtering id done with the same source and target as for Ta coating, only by changing the working gas composition.

Generally, good quality films were obtained with both methods at pressures of 5 mTorr and below. They were well adhering to steel and their surface was very smooth and highly reflective, similar to the surface of polished substrates before deposition.

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Figure 1.The XRD spectra of Ta films deposited on steel. Spectra a and b represent α-phase Ta films deposited with nitrided interfacial layer. In contrast to spectrum c characteristic of mixed phases of films deposited on steel at room temperature. The peaks with crystallographic plane designation correspond to the Ta fcc phase.

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