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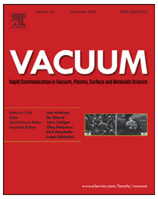

Growth defects in magnetron sputtered PVD films deposited in UHV environment

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ABSTRACT

The growth defects in magnetron sputtered coatings have been well studied in industrial environment. In this work, on the other hand, the emphasis is in observing the same phenomena in a lab-scale UHV environment. TiN and CrN films were deposited at a thickness of 1–2 μm. The statistical evaluation of defect density was based on stylus profilometry scans (2 mm^2 area), and consequent analysis of hillock density in dependence of threshold height. Morphology of defects was observed by SEM, while on selected defects we made FIB cross-sections. In this way we were able to measure the chemical composition of individual seeds – starting points for growth defects. In contrast to expectations, there was only a relatively minor reduction of defect density by using UHV environment.

1. Introduction

Growth defects are imperfections in thin films, deposited by physical vapor deposition (PVD). Their primary undesired consequence is a reduced corrosion resistance of the coatings. Indeed, the majority of papers related to growth defects emphasize the corrosion phenomena, both from the analytical point of view as well as towards methods to reduce the undesired effects of growth defects [1–4]. Unsurprisingly, many papers report on growth defects on coatings, deposited in industrial conditions, including our previous work (see Ref. [5] and references therein).

Several types of growth defect types have been identified [6,7], but the most commonly observed ones, and also the most commonly discussed ones, are the nodular defects [8]. The other common type, a trough, is usually a remnant of a nodular defect in the case the latter was expunged from the film due to strong internal stress. The basic precondition for a nodular defect is a seed. It can be a droplet from the film material, a distinct particle from a foreign material or an imperfection on the substrate surface. We have identified cases of iron, copper and tantalum seeds [5], all traceable to a component in the vacuum chamber, as well as carbon-based ones, probably remnants of organic impurities. Consequent nodular defect growth is relatively uniform, regardless of seed type. Therefore looking for growth defect origin is in a large extent looking for the seed and its composition.

There are generally three groups of seed sources. One is distinct for a particular type of PVD process, while mostly absent in the other ones. A typical case are droplets in cathodic arc evaporation [9]. This is a well-known phenomenon, so far intensively studied, and will not be a topic of this paper, since the deposition method of choice in this work was magnetron sputtering. The other group are the intrinsic sources, pertinent to all the PVD processes. They cannot be avoided, though they might be reduced to some extent. These include dusty plasma phenomena [10], and various types of microarcing. Though arcs can in general be avoided by proper component cleanliness and target purity, local irregularities on the target surface will always exist and cause droplet formation. The seed composition in these intrinsic sources is in most cases equal to the overall film composition, possibly with some stoichiometry variation.

The third group of seed source is related to the vacuum chamber. A deposition chamber, particularly an industrial one, contains many components, such as shields, fixtures, clamps, etc. Many of these components move during the deposition, often in several modes of

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rotation. Thus there is a large effective surface being moved, which acts as a potential source of seeds. One of the most important mechanisms is stress release from remnants of previous depositions, which yields seeds of various composition; indeed, a history of previous films. Parts of the components can be released too, giving mostly iron particles. Another important source are the moving joints, as they are not oil-lubricated. All these chamber-related sources can be considerably reduced with regular cleaning and optimized design. However, in industrial scale many of these steps are not economical or cannot be realistically accomplished.

The main question addressed in this paper is the following: Can we spot those growth defects which are intrinsic to the PVD process itself while avoiding the extrinsic ones, related to the industrial environment? For this purpose we deposited several nitride films (TiN and CrN) in a dedicated lab-scale UHV environment, in contrast to the standard HV industrial-size equipment. These films were compared to our previous results [5,11], which were exclusively obtained in HV.

2. Experimental

The substrates used in our experiments were silicon wafers and two types of steel discs: sintered high-speed steel ASP30 and cold work tool steel D2. They were used for comparison to the industrial batches where these types of steel had been commonly used. The substrate cleaning consisted of successive rinses in ultrasonic baths of trichloroethane, acetone, methanol, and deionized water, and blown dry with dry N_2.

The TiN and CrN films were grown in a load-locked stainless-steel magnetron sputter deposition system KJLC CMS18, applying the TORUS® balanced magnetron sources. The distance between each target center and the substrate holder center is 17 cm. The residual pressure in the main chamber was maintained in the range of 10^{-8} mbar. Pure (99.995%) titanium and pure (99.95%) chromium targets were sputtered in DC regime, in argon atmosphere with admixture of pure (5,5N) nitrogen gas. The disk-shaped targets (3 inch diameter) were sputtered at DC power of 300 W for 210 min. The gases were introduced through the high-precision solenoid valves at various flow rate ratios, while the total pressure was maintained constant at 5×10^{-3} mbar for all depositions. The films were deposited onto electrically-floating substrates, at the temperature of 450 °C. Following deposition, the samples were allowed to cool down to room temperature (this took about an hour) before transferring them to the load-lock chamber which was then back-filled with dry N_2. Detailed explanation of similar depositions of transition metal nitrides in the same system is given in Ref. [12]. The balanced magnetron plasma was kept close to the target surface, with only greatly rarefied plasma in contact with the substrate surface.

In comparison to the industrial deposition system (see details in Ref. [5]) our lab deposition system offers several advantages towards a reduced formation of defects. In addition to lower base pressure, the lab chamber has far fewer moving parts, a much simpler fixturing system and only single rotation (a dynamically tight feedthrough with residual pressure at rotation never exceeding 10^{-7} mbar). There are shutters on the cathodes to be used in the first stage of the deposition (target cleaning), and the samples are mounted in the downward direction.

Surface topography was evaluated by a Taylor-Hobson Talysurf contact profilometer. The evaluation area was 1 mm × 2 mm with a resolution of 1 μm in x and 2 μm in y. Effective vertical resolution was around 5 nm. After measurement acquisition, waviness was excluded by a Gaussian filter (cut-off 0.25 mm) and the number of points above a given threshold height was counted; this was achieved by the Talymap software.

Top-view SEM imaging was performed in a Jeol JSM-7600F field-emission microscope. Focused ion beam (FIB) cross-sections on selected growth defects were made using a FEI Helios NanoLab 600i station. Imaging was performed either by secondary electrons or by ions; where possible, EDS analysis was conducted too.

3. Results and discussion

3.1. Density of growth defects

A suitable quantity is needed to gauge the density of growth defects. The definition of a hillock to be counted as a growth defect is a matter of definition. In our previous paper [5] we explained that the 0.5 μm threshold height is meaningful. In standard industrial PVD hard coatings (3–5 μm thickness) the defect density with such a threshold is in the range 100–500 mm^{-2}.

However, the films deposited in the UHV system were thinner, 1–2 μm, due to a lower deposition rate. Therefore the 0.5 μm threshold is too high as it gives a density of no more than 10 mm^{-2}. Comparing two samples with, say three and four growth defects is statistically not justified. A different gauge is necessary, while preserving the data acquired on industrial samples using the 0.5 μm threshold.

For this purpose we evaluated the defect density of each sample at several threshold values. In addition to the standard 0.5 μm threshold, we also used the threshold of 0.6 μm, 0.7 μm, etc, altogether covering 30 different thresholds from 0.1 μm to 3 μm. Fig. 1a shows such results: dependence of defect density on chosen threshold height. The results are presented in a logarithmic scale, where the curve is roughly linear so there is an exponential dependence of the defect density on the threshold height.

A line can be fitted in the log-chart (Fig. 1b); a line is typically expressed by two parameters: the constant (value at zero) and the slope. The former may be interpreted as extrapolated density at zero threshold height, though in practice at close to zero the roughness becomes the prevailing component, not the individual defects. Nevertheless, the extrapolated value at zero can be a useful gauge of the density of small defects; we will use this designation in the text later on. The slope on the other hand is not an intuitive parameter; in contrast we prefer to use the interpolated density at 0.5 μm threshold height. It corresponds to the 0.5 μm evaluation used in previous papers, with an advantage of not being an integer; we will call it the density of large defects.

The results shown on Fig. 1a correspond to two groups of samples of CrN films, deposited in either an industrial, or a lab deposition system. The latter is shown on three substrates: silicon, and two types of steel. Both deposition systems apply magnetron sputtering, however, the lab system works in much cleaner conditions than the industrial one. In addition, the film thickness in the industrial system is several times larger than the lab ones.

By comparing the three curves from Fig. 1 one can deduce the following conclusions. The defect density in the industrial deposition is larger for all threshold heights, which is expectable. However, the difference (to be more precise: the ratio) between the two is increasing with threshold height. Thus the density of small defects is comparable, while in larger defects, the industrial coatings dominate. There are essentially no growth defects above 1 μm height in lab conditions. The defect height directly depends on the seed size, so a large seed will give a high defect. However, a very large seed will probably not stick within the growing film, thus defects higher than the film thickness are rare. Quite remarkably on the other hand, the three curves for different substrates basically overlap. This means that the defect population does not depend on the substrate but on the process alone.

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