SVC Topics

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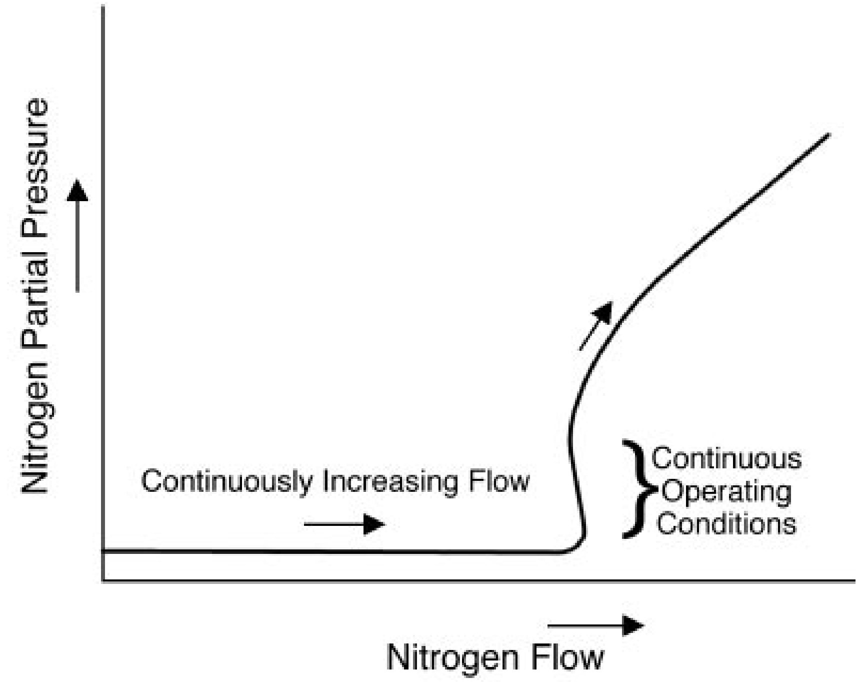
PVD Processes: Reactive Deposition—Gas Control

Reactive sputter deposition processes typically use a mixture of gases. One gas is usually argon, which is good for sputtering, and the other is a reactive gas, such as nitrogen, which reacts with the depositing film material to form a film of a compound material. During the reactive sputter deposition process, the reactive gas is continually being removed by chemical reaction with the depositing film material. This pumping, or "gettering" action, can be quite significant, and depends on the deposition rate and the area over which the material is being deposited.

Consider, for example, the case of deposition of titanium over 10^4 cm^2 at a rate of 10Å/sec. A square centimeter of surface contains about 10^{15} atoms, so 10^4 cm^2 would have 10^{19} atoms on the surface. A deposition rate of 10Å/sec is about three atomic layers/sec. This gives a deposition rate of 3×10^{19} atoms/sec. If each of these atoms reacts with a nitrogen atom to form TiN, this would mean removing about 1.5×10^{19} nitrogen molecules/sec, which is equivalent to about 30 standard cubic centimeters/min (sccm) of nitrogen gas. A Torr-liter is 3.5×10^{19} molecules, so the pumping speed resulting from the chemical reaction is about 0.5 Torr-liter/sec, or, at a typical sputtering pressure of 5×10^{-3} Torr, 100 liters/sec pumping speed.

The figure shows the effect of this pumping on the partial pressure of the nitrogen gas as a function of mass flow rate with increasing flow during reactive sputter deposition of TiN. At low nitrogen flow rates, the nitrogen is initially used up by reaction on the surface of the depositing film, and the partial pressure does not increase with flow rate. At a higher flow rate, the nitrogen enters at a greater rate than the reaction rate, and the partial pressure begins to rise. The increasing partial pressure of nitrogen, however, allows the titanium target surface to react with the nitrogen (be "poisoned"), giving a lower sputtering rate, which allows the partial pressure to be higher at a lower flow rate and results in a "knee" in the curve. At higher flow rates, the nitrogen partial pressure rises rapidly. The shape of the knee will depend on the target material, target design, sputtering parameters, and the reactive gas being used. Even the amount of use the target has seen can influence the shape of the curve because, with use, the sputtering area and geometry changes.



Partial pressure of nitrogen as a function of the nitrogen flow rate in the sputter deposition of TiN. The curve is for increasing gas flow and does not show the hysteresis associated with a decreasing gas flow after the target has been poisoned.

The ideal flow rate for the reactive gas is at the knee—the sputtering rate is still high, but there is enough nitrogen to completely react with the titanium film to form a TiN film. The flow rate at the knee of the curve will depend on the deposition rate and the area over which the film is forming. The variation of this value with fixturing, or the number of parts being processed (*i.e.*, area) is called the "loading factor."

If the sputtering target is poisoned by increasing the nitrogen flow rate above the knee and then decreasing the flow rate, the partial pressure will show a hysteresis caused by the lower sputtering rate of the poisoned target surface, as compared to the clean target surface during the increasing flow curve. This hysteresis will keep the partial pressure higher at a given flow rate than on the increasing flow curve until the target is sputter-cleaned and the sputtering rate increases. Too high a reactive gas pressure during deposition has been shown to decrease the microhardness of reactively sputter-deposited TiN films.

In reactive deposition using a plasma environment surrounding the deposition fixture, the partial presssure of the reactive gas in the plasma is an important processing variable. In a typical reactive sputter deposition processing system, the total gas pressure and the individual gas flow rates are measured and controlled. The total pressure is measured using a capacitance manometer gauge or a viscosity gauge. The flow rates are measured using mass flow meters.

Measuring the mass flows and total pressure may not tell much about the partial pressure of the reactive gas in the system, because the gas is continually being removed by reaction with the depositing film material. They are, however, good measurements for process control in production because they are relatively inexpensive, and process reproducibility can be attained from one run to another if the geometry of the system is not changed. There may have to be some parameter adjustment with target use as a result of changes in target geometry.

More accurate control of the partial pressure of the reactive gas can be done using a mass spectrometer, by monitoring the optical emission from the plasma (plasma emission monitor), or by using atomic absorption techniques to monitor the sputtering rate. These techniques use the output of the detection system to drive a feedback control on the mass flow system. The mass spectrometer samples the gas from the chamber; care must be taken that ions from the plasma do not enter the detection system. Generally, the mass spectrometer must be differentially pumped to obtain the necessary sensitivity.

Optical emission from the plasma is relatively simple, and by using collimators and fiber optics, the emission from a selected region near the target can be obtained. Generally, the emission from the sputtered metallic species is monitored, and the gas flow is controlled as a function of the peak height of a specific emission wavelength. Atomic adsorption spectrometry (AAS) can also be used to monitor the sputtering rate of the metallic species and provide the control signal. In AAS a specific wavelength of light that is absorbed by the vapor species is transmitted through the vapor flux and compared to an unadsorbed reference value. The light source is typically a hollow cathode gas-discharge lamp whose cathode is of the same material as that of the vapor to be measured, and whose output is the optical emission spectrum of the cathode material. The light source emits a spectrum of radiation and the bandpass filter (or a monochrometer) eliminates all the radiation except the wavelength(s) of interest.

Using a hydrocarbon gas (such as acetylene) as the reactive gas to deposit carbide films can pose special problems. At low partial pressures, the hydrocarbon molecules will decompose inthe plasma and provide a source of carbon for the deposition process. This carbon will co-deposit on surfaces whether or not it reacts for form a carbide. Unreacted carbon in a film can result in undesirable film properties. At high partial pressures, the hydrocarbon in the plasma will dissociate and vapor phase nucleation will create particles called "soot." In the plasma, these particles will become charged negatively with respect to the plasma. The particles will not deposit on surfaces in contact with the plasma because they, too, are negative, with respect to the plasma. These suspended particles, however, will deposit on surfaces when the plasma is extinguished. These particles should be swept out of the system as much as possible. Soot particles on the surfaces in the vacuum system will adsorb water vapor and change the pumping change the pumping characteristics of the system. **P&SF**

Reference

W.D. Sproul, P.J. Rudnik, M.E. Graham, C.A. Gogol & R.M. Müller, "Advances in partial pressure control applied to reactive sputtering," *Surf. Coat. Technol.*, **39/40**, 499 (1989).