Particle contamination formation in magnetron sputtering processes

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Defects caused by particulate contamination are an important concern in the fabrication of thin film products. Often, magnetron sputtering processes are used for this purpose. Particle contamination generated during thin film processing can be detected using laser light scattering, a powerful diagnostic technique which provides real-time, *in situ* imaging of particles >0.3 μm on the target, substrate, or in the plasma. Using this technique, we demonstrate that the mechanisms for particle generation, transport, and trapping during magnetron sputter deposition are different from the mechanisms reported in previously studied plasma etch processes, due to the inherent spatial nonuniformity of magnetically enhanced plasmas. During magnetron sputter deposition, one source of particle contamination is linked to portions of the sputtering target surface exposed to weaker plasma density. There, film redeposition induces filament or nodule growth. Sputter removal of these features is inhibited by the dependence of sputter yield on angle of incidence. These features enhance trapping of plasma particles, which then increases filament growth. Eventually the growths effectively ''short-circuit'' the sheath, causing high currents to flow through these features. This, in turn, causes mechanical failure of the growth resulting in fracture and ejection of the target contaminants into the plasma and onto the substrate. Evidence of this effect has been observed in semiconductor fabrication and storage disk manufacturing. Discovery of this mechanism in both technologies suggests it may be universal to many sputter processes. [S0734-2101(97)08504-7]

I. INTRODUCTION

Particle contamination produced during plasma processing is a topic of much current concern and study because of the deleterious effects of contaminant particles on substrate surfaces. Sputtering, a ubiquitous plasma processing technique used in thin film deposition, is also prone to particle contamination. Particle contamination can result in pin holes, delamination and interconnection shorts or opens in metallization processes. On magnetic storage disks, particle contamination can also result in read–write errors, bad sectors, and total disk failure. To minimize this problem, it is important to identify the causes and contributing factors to particulate contamination.

Sputtering has long been known to result in visually observable and microscopic defects on the target surface. Floro *et al.*[1] described analysis of 2–50 μm long whiskers of 0.05–0.5 μm diameter formed following ion bombardment of graphite surfaces. Metallic targets have shown development of sputter cone surface impurities.[2,3] To date, however, these target problems have not been directly linked to wafer or disk contamination problems. Still, these problems are commonly observed during many sputtering processes.

At first, particles present in magnetron sputter deposition processes might be expected to exhibit similar behavior and properties as particles formed in plasma etch or chemical vapor deposition processes.[4,5] As such, these particles have enhanced charge and show differences in transport from nonionized environments.[6,7] Also, the charge on particulates, combined with the negative sheath boundary can result in suspended and ''trapped'' particles.[8] These suspended particles grow larger due to sputter deposition. Momentum transfer from ions drifting towards the sheath region also contributes an ''ion drag'' force that enhances the transport of particles.[9] In addition to electrical forces, particles in plasmas are influenced by the same forces that influence particles in nonionized environments: thermophoresis, neutral drag (Stokes force), turbulence, and gravity.[10] At typical sputtering plasma pressure (<10 mTorr), neutral drag is small and turbulence is negligible. Since the mean free path at typical sputtering pressures is 5–50 mm, thermophoresis is possible only under some circumstances. Gravity is typically a weak force for all but very large particles (i.e., those larger than a few hundred microns).

It is believed that homogeneous (i.e., gas-phase nucleation) as well as heterogeneous (e.g., reactor wall flaking) sources contribute to microcontamination. Only recently have these issues been carefully addressed by scientific study.[11] Because magnetron sputtering is operated at very

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low pressures, homogeneous contributions are likely to be negligible. As a result most contamination problems in magnetron sputtering processes have been attributed to heterogeneous contamination sources, such as wall flaking. In particular, film deposition on the walls of the plasma chamber can flake due to thermal expansion mismatch, film stress, and from poor adhesion of the film to the wall.[12] Also, corrosion and ion bombardment may promote film flaking, as can the presence of impurities or water vapor on the process chamber walls.

Laser light scattering has been successfully used to detect particle contamination problems in a wide range of host tools. This technique, which has been described in detail elsewhere,[8,13,14] is also applicable to the analysis of sputter deposition problems. However, unlike the previously published work, for analysis of particle contamination sources in magnetron sputtering processes, it is especially useful to detect particles and their precursors directly on the magnetron target, or on the substrate. Recently, laser light scattering (LLS) has also been used for *in situ* detection of particles on wafers during processing.[14]

II. EXPERIMENT: PARTICLE DETECTION APPARATUS

The experimental LLS method used is a variation of the apparatus previously used for detection of particles in gas-phase plasmas. Both forward-direction and backscattered LLS were used and depended on the tool geometry and process studied.[15] In the work performed at Seagate, a large mirror was mounted at the bottom of a large, in-line sputtering tool. The laser light was directed down onto the mirror, from the laser mounted on the top of the tool, and the reflected beam was viewed with a video camera also mounted on the top of the tool. The laser illumination plane was set parallel to one of the opposing sets of targets. In the work performed at Materials Research Corporation (MRC), backscattered laser light was employed. Both methods provided sufficient sensitivity to analyze the sources and mechanisms of particle contamination. This was due to the relatively large filaments on the targets observed in both cases.

In magnetron sputtering, the plasma is confined near the target surface with magnetic fields resulting in regions of different plasma density which sputter the target nonuniformly producing a distinct target erosion groove or racetrack. This results in significant changes in plasma sheath thickness over the surface of a sputter target. Over the racetrack and close to it, the sheath thickness is very small because the plasma density is highest. In this region, it is difficult to discern suspended particles from particles present on the target. To detect particles on the target, the beam is slightly angled, so that the laser is rastered parallel to the target surface at grazing incidence. Fixed surface defects, however, can easily be discerned from particles suspended very close to the target because of the random movement the latter shows during video imaging.

The set-up employed at Seagate is shown in Fig. 1(a). Particles can be detected both on the disks or on the targets using a grazing incidence method. Imaging detection of the scattered light is accomplished with a high resolution video camera. In the work performed at MRC, the laser beam was rastered across the edge of the target at grazing incidence. This setup is seen in Fig. 1(b).

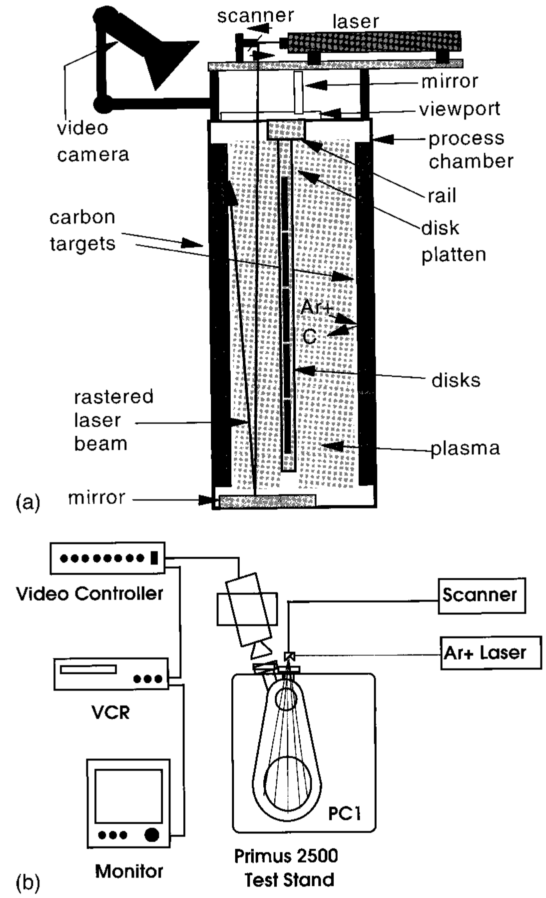

FIG. 1. (a) LLS setup used to detect particles in the in-line disk sputtering tool at Seagate. Note that both forward and reverse laser light scattering may be used by placing a large mirror at the base of the tool. (b) LLS setup in the MRC Primus sputtering system. Note that only backscattered light could be collected in this system.

III. RESULTS

Similarities in microcontamination generation were noted between the rf sputtering plasma used for carbon deposition on magnetic storage disks and the rotating dc magnetron plasma used to reactively sputter TiN from a Ti target onto silicon wafers. In both cases, the dominant source of contamination was attributed to the growth, movement, and eventual *mechanical* ejection of filaments or nodules on the targets. In both cases, the targets initially appeared clean and free of filament growth. However, after several hours of sputter operation evidence for these target structures was observed. Results for the two sputtering technologies are summarized below:

A. Magnetic storage disk manufacturing

Laser light scattering studies of the rectangular carbon target surfaces show the presence of filaments along the target regions exposed to low ion bombardment. After several hours of sputtering, these filaments become heated and eventually shoot off the target, striking and embedding into the disks. LLS results obtained for the targets and disks are discussed below.

1. Carbon targets

Starting with a clean tool, *suspended* particles smaller than 0.5 μm were seen ''swarming'' around small defects or elongated grains on the target after several minutes of sputtering. These particles were near the racetrack region. After 10–30 min, these particles occasionally adhered to the surface grains, causing the formation of slightly elongated filaments. Growth of the filament increased with time. It was evident that these filaments electrostatically attracted particles due to the slightly elevated plasma potential near the trap and the negative particle charge.[16,17] Sputter deposition onto these trapped particles increased their size, decreasing their random movement and eventually causing the particles to touch and adhere to the filaments.

As the target filaments grew longer, many became incandescent, an effect attributed to the passage of high current through these fine features. Some filaments grew as long as 2–3 mm and were <10 μm in diameter. Eventually the filaments fractured and were released into the plasma. Fracture of the filaments appeared to be a violent process, probably due to the impulse provided by vaporization of filament material. Also, the filaments become negatively charged once they leave the surface due to attachment of electrons. Because of this, negatively charged particles are accelerated by the repulsive sheath field surrounding the target. Using the known framing rate of the frame camera and distances inside the tool, the velocity of some of these filaments was determined to be in the range of 100 cm/s. During passage through the high density regions of the plasma, the particles became increasingly heated. Heating of particles, especially large particles in a plasma, has previously been shown.[18] Figure 2 shows a series of sequential, captured video images of a heated, ejected filament shortly after release from the surface of the targets. Note the movement of the light scattering defect seen in Figs. 2(a) and 2(b) (upper right corner), eject from the target and move downwards in Fig. 2(c) and finally disappear completely from view in Fig. 2(d).

Another interesting observation seen was the release of very fine particles from the surface of the carbon target during the first few seconds of power application. Laser light scattering showed that dense clouds of very fine dust (believed to be less than 0.2 μm in diameter) ''bubbled'' from the racetrack surface in the first ten seconds of sputtering. This fine dust is close to the grain size of the graphite pressed powder target and so may be caused by the abrasive method employed for target cleaning, prior to processing. These fine particles may act as nucleation centers for growth of larger particles.

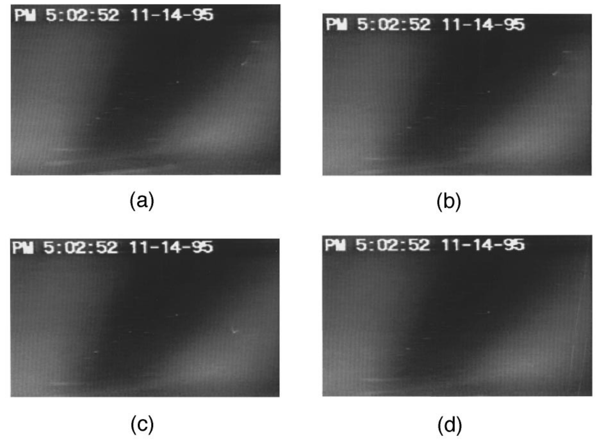

FIG. 2. Four captured video images are shown, separated by 1/30 s, the camera framing rate. Note the fixed incandescent filament in the upper right center portion of (a) and (b). This filament is ejected in (c), moving to the right center edge of the figure and finally disappears from view completely in (d).

2. Particle detection on disks

To further evaluate wall flaking and target effects, a holder containing a number of clean disks was placed between the targets for static deposition. The disk pallet was mounted at a sharp angle, around 80°, to allow for scanning across the disk surface from the laser mounted on the top of the tool. Backscatter was used to detect particles. The disks were scanned prior to, and during sputtering. In some cases, particle deposition on the disks could be observed under high magnification with the video camera, without the aid of the laser. Disk surface scans were compared with the LLS measurements of the targets.

By monitoring different disks aligned opposite to various portions of the target, it was possible to analyze the variations in particle deposition on each disk and to relate these measurements to the nearby target surfaces. Those disks located across from the low sputtering regions of the target showed a *very* high level of contamination, providing a confirmation of the target LLS results. This was especially evident for disks located in front of the centers of the targets, when compared to those disks closest to the racetrack. In time, the particles observed on the disks also appeared to grow larger, probably due to film deposition over the particle. This indicates that particle contamination on the disks may become buried by subsequent film deposition.

B. TiN deposition in integrated circuit metallization

In the manufacture of integrated circuits, contacts to silicon, interconnections between levels of metallization, and reflective conductors require reactively sputtered titanium nitride (TiN) films for barriers and antireflective coatings. These reactive sputter processes are known to contribute to particulate contamination and die yield loss. Laser light scattering was successful in diagnosing mechanisms of particle generation, transport, and trapping.

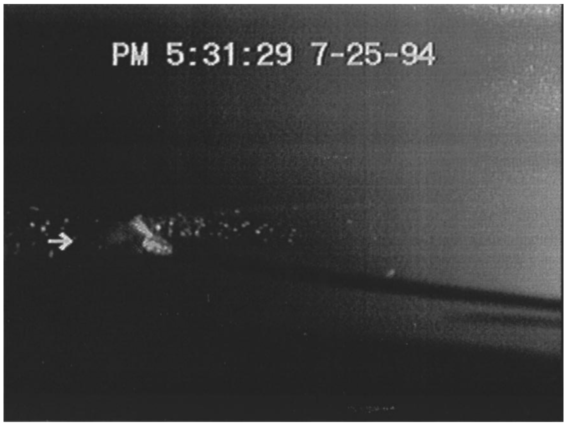

FIG. 3. Video image shown the edge of a Ti planar magnetron target and anode (dark space) shield. Filaments visible at the edge of the target and nodules visible on the surface of the target are marked by arrows a and b, respectively.

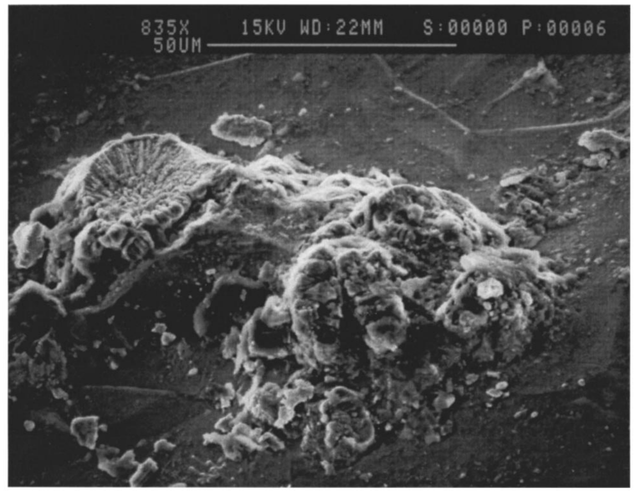

FIG. 4. Scanning electron microscopy micrograph showing the microstructure of broken off TiN filaments on a Ti target surface.

Reactively sputtered TiN was deposited in an Ar/N_2 ambient from a Ti target onto 200 mm silicon wafers. Typical processing pressures were 2–4 mTorr at magnetron powers between 2–10 kW. LLS measurements were made on the surface of a 295 mm diam Ti target operated with dc power and a series of rotating magnets behind the target. Although redeposition onto the target surface occurs, this design promotes nearly full target erosion. However, during reactive sputtering features resembling filaments or flakes may grow to sizes up to hundreds of microns in the regions of the target where the sputter rate is lowest, typically at the edge of the target.

Through the use of smooth chamber shields, flaking and delamination of film deposits due to increased film stress and poor adhesion was induced. Along the edges of the target, numerous target flakes or filaments ranging from 50–500 μm long and 2–25 μm wide were observed. Occasionally, these flakes became trapped at the edge of the sputter target and were oriented nearly normal to the target surface with the free end pointing towards the plasma. As in the case of the stationary magnetron plasma, filaments at the edge of the target were observed to grow at rates of up to 1 mm/h due to local electrostatic traps accumulating additional particles. The filaments deflected in a pendulumlike fashion oscillating with the same periodicity as the magnetron rotation. This motion is probably due to electrostatic forces on the filaments which are at the cathode potential that arise from local changes in the plasma potential as the magnetic field is rotated.

Similar to the work on carbon targets, when a filament was positioned near the interelectrode region between the target and the anode (ground), it became incandescent. Figure 3 shows a captured video frame of the target and dark space shield viewed at near grazing incidence in which two filaments are visible. The motion of the filaments eventually causes breakage and ejection of fragments into the plasma. Figure 4 shows the microstructure of one such broken-off filament. Filament ejection, which contributes to surface contamination, was also observed at plasma shutdown. Some improvement was observed when magnetron power was slowly ramped down prior to plasma shutdown. Filament formation was *not* observed during argon sputtering of Ti, nor were any trapped, submicron particles observed during this chemically unreactive sputtering process. This indicates the importance of homogeneously grown, trapped particles for growth and ejection of particulate contamination during TiN sputtering. In contrast, Ti sputtering is well known for showing much less wafer contamination than the reactive TiN process. The improved cleanliness of the target seen by LLS is in agreement with these well-known fabrication differences between Ti and TiN sputtering.

Also by turning off the reactive N_2 gas and sputtering the Ti target in Ar, the number of edge flakes could be reduced. This demonstrates that the formation and growth of filaments is controllable by processing Ti periodically to clean the target surface.

Finally, sub-0.5 μm TiN particles were observed suspended in the electrode region between the anode ground shield and the edge of the target. These particles appeared to be strongly trapped and exhibited random motion during plasma processing. Significant accumulation of trapped particulates in this region may ultimately cause arcing which also can generate and distribute microcontamination throughout the process chamber.

IV. DISCUSSION

Similar results were observed in both of the cases described above. A mechanism is suggested below and is schematically illustrated in Fig. 5.

(1) For carbon sputtering, the nucleation process is likely initiated by the evolution of very fine C dust generated upon initiation of the plasma from the target. Some dust is generated by the explosive evolution of adsorbed water vapor during the first few seconds on ion bombardment on the target. This step likely results in particles sized below 50 nm that enter the plasma, become

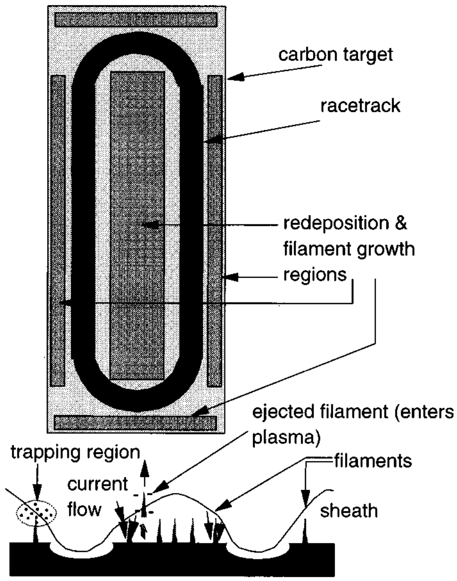

FIG. 5. Schematic illustration showing the presence of filaments on the target surface on the sputtering tool, the presence of trapped, fine particles, and the fracture of these filaments, causing their violent release and heating in the plasma.

charged and are then subject to trapping effects near the surface of the target. For reactive TiN processes, extremely small particles may be homogeneously grown. In both cases, particles can also be generated during chamber maintenance.

(2) Ion drag carries particles toward the sputter target. This force is balanced by electrostatic repulsion in the sheath resulting in particles suspended at the sheath just above the target surface, typically less than 0.5 mm.

(3) Trapping in the plasma enables the growth of suspended particles. This increases the mass and charge of the particles.

(4) Filaments, target roughness or nodules, especially in the weak plasma region between the racetrack and at the edges of the target, form electrostatic ''traps'' that attract and confine these particles, in a manner as previously found for other plasma processes.[19] Target roughness can be influenced by density or composition gradients, inclusions, grain structure and even by some cleaning procedures.

(5) Small particles orbit and surround the filaments and other protrusions from the target. Eventually, the particles bump into the filaments and adhere to them, making the filaments grow larger. Larger filaments result in stronger trapping potentials. The up and down sheath motion caused by a rotating magnetic field can also induce the particles to contact the filaments.

(6) As filaments increase in size, they can become a significant fraction of the sheath thickness. Filaments in contact with the powered electrode become an enhanced path for current flow. Increased current flows through the filaments, causing the filaments to heat up and glow.

(7) Heating of the filaments results in fracture and ejection of the filament. The ejected fragments become electrically isolated from the target and remain negatively charged.

(8) The charged particle is accelerated by the sheath field, with a force of $q\mathbf{E}$ where q is the charge of the filament and $\mathbf{E}$ is the electrical field in the sheath. This further propels the filament into the plasma.

(9) Movement of the filament is influenced by the kinetic energy released during ejection, electrostatic forces in the sheath and in the plasma.

(10) The hot filament impinges onto a wafer disk or chamber walls often at high velocities. In some cases, the combined heat and kinetic energy of the filament is sufficient to melt the disk at the point of impact and permanently imbed the filament into the disk.

V. CONCLUSIONS

The formation and transport of particles during plasma film deposition processes shows both similarities and differences with other plasma processes. Particle nucleation, growth, and trapping are observed during film deposition, similar to that observed during etching processes. However, significant differences are also observed. The highly nonuniform plasma density typical of magnetron sputtering processes are especially prone to simultaneous material removal and redeposition rates in different target regions. Because of this, filament formation can occur in low plasma density regions of the target. When these filaments form near the high density regions of the plasma, the sheath thickness is changing and is thin due to the higher density plasma. Because the filaments are resistively heated by increased current flow, violent mechanical failure can result, carrying the filament into the plasma. Combined with repulsion between the negatively charged filament and the sheath region, this process results in an acceleration of the filaments away from the sputter target. As the filaments cross high density regions of the plasma, electron/ion impingement and neutralization processes result in rapid heating of the particles. As a result, a hot, fast-moving particle can impinge onto a substrate, possibly causing melting and penetration into the disk substrate.

Laser light scattering is a powerful tool for diagnosing particle contamination problems in a wide variety of plasma tools and processes. Its widespread use and application is only starting now. Clearly, its use continues to offer significant insight and benefit into the analysis and control of particulate contamination problems in a wide range of applications.

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