In Situ Analysis of Particle Contamination in Magnetron Sputtering Process During Magnetic Media Manufacturing

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ABSTRACT

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 can cause electrical shorting, pin holes, problems with photolithography, adhesion failure, as well as visual and cosmetic defects. Particle contamination generated during thin film processing can be detected using laser light scattering, a powerful diagnostic technique that provides real-time, in situ imaging of particles > 0.3 µm in diameter. Using this technique, the causes, sources and influences on particles in plasma and non-plasma processes may be independently evaluated and corrected. Several studies employing laser light scattering have demonstrated both homogeneous and heterogeneous causes of particle contamination. In this paper, 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. During magnetron sputter deposition, one source of particle contamination is linked to portions of the sputtering target surface exposed to weaker plasma density. In this region, film redeposition is followed by filament or nodule growth and enhanced trapping that increases filament growth. Eventually, the filaments effectively "short-circuit" the sheath, causing high currents to flow through these features. This, in turn, causes heating failure of the filament fracturing and ejecting the filaments into the plasma and onto the substrate. Evidence of this effect has been observed in storage disk manufacturing. Discovery of this mechanism in this technology suggests that this mechanism may be universal to many sputtering processes.

INTRODUCTION

Present thin film deposition plasma processes include: physical vapor deposition (PVD), (i.e., sputtering), and plasma-enhanced chemical vapor deposition (PECVD). Conventional and reactive magnetron sputtering are commonly used thin film technologies for the deposition of: (1) conductors and refractory metals such as Al, Cu, Ti and TiN for integrated circuit interconnect metallization; protective carbon coatings on magnetic storage disks; oxides such as ITO for flat panel displays; Ta and Au for ink-jet heads as well as TiN and TiC, which are common metallurgical hard coatings. Plasma-enhanced CVD processes include: silicon dioxide for gate contacts and silicon nitride diffusion barriers and etch masks [1].

Particulate contamination during plasma deposition and plasma etch processes cause severe problems during the production of thin films. These problems generally fall into two classes: (1) direct yield loss or scrap during the production cycle caused by point defects [2], or (2) reliability problems contributed by film failure during product use. Although easily detected, the former problem increases the cost of production by reducing the product yield and requiring more frequent and extensive preventive maintenance measures to compensate for yield loss. Testing required to analyze reliability issues also increases the cost of production. The labor, materials and product losses incurred by yield loss that result from particulate contamination can shift the economics of a line to unprofitable [3].

Particles present in magnetron sputter deposition processes are expected to exhibit similar behavior and properties as in plasma etch processes. The kinetics of particulates in these processes are much different from the aerosol behavior normally observed in non-ionized, or conventional environments. As a result of the difference in the relative mobilities of ions and electrons, particles acquire a negative charge in a plasma, the magnitude of which depends on the electron density and temperature [4,5]. The difference in the rates of electron and ion collisions with particulates results in a net increase in negative charge until Coulombic repulsion screens low-energy electrons. The charge on the particulate eventually reaches steady state due to a zero net current due to positive ions and electrons with sufficient energy. The charge on particulates combined with the negative field at the sheath boundary can result in suspended particles that can then be transported significant distances in a plasma. Momentum transfer from ions in the plasma also contribute an "ion drag" force that can aid transport of particles [6]. As a result, a single source of particles in a plasma reactor may contaminate other regions of the plasma and chamber volume, rather than only near the point of generation [7].

Both homogeneous (i.e., gas-phase nucleation) and heterogeneous (e.g., reactor wall flaking) sources contribute to microcontamination. Only recently have these issues been carefully addressed by scientific study (for an overview of work in this area, see Reference [8]). For low-pressure plasma processes (< 10 mtorr), such as magnetron sputtering, it is believed that the contributions to microcontamination due to homogeneous mechanisms are negligible. However, wall flaking or heterogeneous processes still contribute to contamination problems, and are often as problematic as homogeneous sources. Below, we describe a new mechanism for particle generation and transport unique to magnetron sputtering.

It is not surprising that particles can be formed in plasma deposition processes. Silane-containing plasma have long been known to cause a “white-out” of contamination [9], especially if the power, pressure and temperature of the process is not properly controlled. Chemically reactive plasmas, such as plasma-enhanced chemical vapor deposition (PECVD) processes are prone to homogeneous nucleation problems because of the higher processing pressures. Particles may also be formed by homogeneous nucleation processes, thought to be related to the agglomeration of negative ions in the plasma. Film deposition on the walls of the plasma chamber can flake due to thermal expansion mismatch, film stress, or from poor adhesion of the film to the wall [10]. Corrosion and ion bombardment can promote film flaking, as can the presence of impurities or water vapor on the process chamber walls.

Clearly, prior understanding of particle forces indicates that very low pressure operation can be significantly different from higher pressure plasma processes. For example, suspended particles have been observed in a variety of plasma etch and deposition processes. In these cases, the life cycle of the suspended particle ends either when it is flushed out of the plasma, into the pump port, when it grows too massive for electrostatic suspension in the sheath, or when the plasma process is extinguished. Most commonly, when the plasma is turned off, suspended particles fall onto the substrate, chamber walls, or the target [12]. This is typically when surface contamination results.

Laser light scattering has been successfully applied for detection of particle contamination problems in a wide range of host tools. This method, provides a real-time, in situ means of detecting particles larger than 0.2 μm during plasma processing. A total of over 15 plasma tools have been studied using this technique; particles have been detected in each case, including conventional and high-density source design, etch and PECVD tools. Much of this work has been previously published [8,13,14]; however, results in this paper deal specifically with differences in particle detection and formation in magnetron sputtering tools.

EXPERIMENTAL: PARTICLE DETECTION APPARATUS

In film deposition processes, particles of interest are equal to or larger than the wavelength of visible light. These particles interact with light by scattering. Dielectric particles scatter light predominantly in the forward direction. In contrast, conducting particles, scatter mainly in the direction opposite the incident light, as described by MIE scattering. In the directions other than forward or reverse scattering, the angular distribution of the scattered light goes through local maxima that depends on particle geometry as well as material [15].

Most particles in film deposition processes are dielectric in nature, either because the film is dielectric, or because impurities and other process components become entrained in the particles. Accordingly, in most cases, the greatest detection sensitivity can be obtained by collecting light in the forward scattered direction [14]. Also, since the particles are largely localized at the plasma sheath, light should be directed along this plane. A laser is ideal for this purpose, since the beam can be easily rastered to illuminate a plane at the plasma sheath using a single-axis scanning mirror [16].

Often, detection of the scattered light from particles is possible with the eye. However, to avoid potential injury and for better detection sensitivity, a video camera can be used. Video data collection is useful to document particle generation, transport and trapping as well as providing the capability for slow motion and still frame analysis of individual and collective particle movement. This is particularly useful for following particle trajectories during the rapid transport or release of particles and velocity measurements [17]. The laser light scattering (LLS) setup is shown schematically in Figure 1.

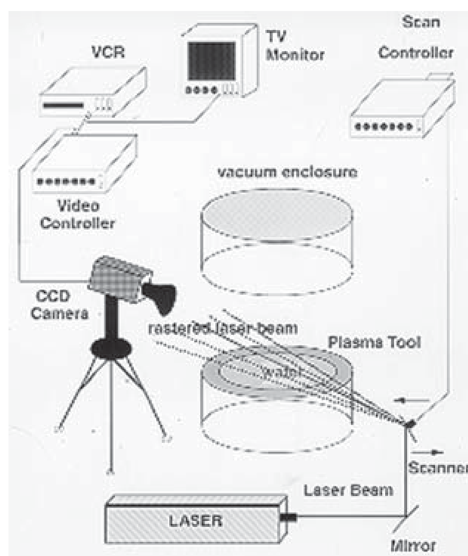

Figure 1: Typical laser light scattering (LLS) setup employed for detection of particles in semiconductor processing

In some cases, forward-direction LLS is not feasible [18]. In the work performed in system used for magnetic media production (Figure 2), a large mirror was mounted at the bottom of a large, in-line sputtering tool. The laser light was reflected 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. In this case, also, the laser illumination plane was set parallel to one of the opposing sets of targets.

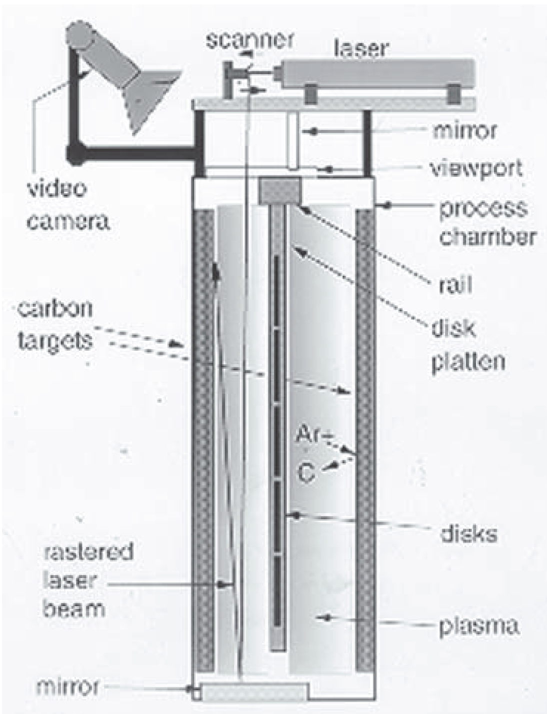

Figure 2: LLS setup used to detect particles in the in-line disk sputtering tool. Note that both forward and reverse laser light scattering may be used by placing a large mirror at the tool.

In magnetron sputtering, the plasma is confined near the target surface with magnetic fields resulting in regions of different plasma density that erodes the target, non-uniformly 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. This can also be an advantage, since particles on the target can influence particles on the substrate, as shown in this work. To detect particles on the target, the beam is slightly angled, such that the reflected beam strikes sections of the target at grazing incidence. As shown in Figure 2, 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.

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* failure 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 growths was clearly observed. Results related to the work carrier out during magnetic production are described below.

Magnetic Storage Disk Manufacturing

Laser light scattering studies of the target surfaces show the presence of these filaments along the regions of the targets exposed to low plasma intensity and that after several hours of sputtering, these filaments become heated and eventually shoot off the target, striking and embedding into the disks. Below, we analyze the detection and behavior of particles during the PVD process in different sections of the tool.

Carbon Targets

A carbon deposition in-line disk manufacturing tool was monitored by LLS as a function of time for 8 h of sputter deposition starting with a clean tool. Within several minutes of starting the plasma with new carbon targets, particles smaller than 0.5 μm were seen “swarming” around small defects or elongated grains on the target, especially in the redeposition region between the racetrack on the target. Within tens of minutes, these particles occasionally adhered to the surface grains, causing growth of these features into slightly elongated filaments. The greatest density of filaments was seen in the center region of the target, between the racetracks and at the outer edges of the target, beyond the racetrack region. With longer process time, even faster filament growth was observed because the rate of sputter removal is much lower in this region than in the high-density regions of the plasma, (i.e., the racetrack).

Some redeposition was also seen on the anode plane and along other grounded surfaces in the tool. In these regions, as on the target surface, LLS showed that filament growth occurred by heterogeneous nucleation: very small particles were detected “swarming” around the carbon filaments protruding from the target face. In time, these particles grew and eventually adhered to the filaments, increasing their size. It was evident that the filaments resulted in localized “particle traps,” which electrostatically attract particles due to the slightly elevated plasma potential near the trap and the negative particle charge [19,20]. Deposition onto these trapped particles increased their size, eventually causing the trapped particles to hit the filaments and adhere, once again increasing the size and trapping field surrounding the filaments. Continued growth of the filaments increased during the viewing period.

As the filaments on the carbon target grew longer, many became incandescent, an effect attributed to passage of high current through the filaments. As the filaments heated further, they fractured from the surface and were released into the plasma. This was seen to be a violent process, probably due to the impulse provided by vaporization of some of the carbon material in the filament while exposed to vacuum, and the fact that the filaments become negatively charged once they leave the surface. Negatively charged particles are also accelerated by the repulsive sheath field surrounding the target. Using the known framing rate of the frame camera and known distances inside the tool, the velocity of some of these filaments was determined to be in the range of 100 cm/s.

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 gm in diameter) "bubbled" from the racetrack surface in the first 10 s of sputtering. This fine dust is close to the grain size of the graphite-pressed powder target. It may also be due to the abrasive method employed for target cleaning, prior to pump-down and plasma operation. It is important to note that these fine particles may act as nucleation centers for growth of larger particles, and that target maintenance operations may actually increase subsequent contamination problems, especially those due to embedded particles in the disks.

Filaments Ejected into the Plasma

Particle ejection into the plasma was clearly observed. This phenomenon increased with prolonged plasma operation. After several hours of carbon sector operation, particle ejection was observed every 2 to 3 s within the relatively small region imaged by the video camera. Since only the region representing approximately 1% of the target surface was imaged by the camera, it is reasonable to assume that over the full surface of the target particle, ejection occurs at a rapid rate.

Ejection of particles was seen both by LLS and by the red-colored incandescence of filaments as they heated up inside the plasma. Slow-motion analysis of the video measurements showed changes in the shape and structure of the filaments once ejected into the plasma. Close to the target, the filaments were tightly coiled. During passage through the high-density regions of the plasma, the particles became heated further and appeared less-coiled with increasing distance from the target. In other words, the filaments acted like "springs" when ejected from the surface. Because of the combined high velocity and high temperature of these filaments, it is reasonable to infer that these projectiles can imbed themselves into a disk, if substrates are in their trajectory. Heating of particles, especially large particles in a plasma, has previously been shown [13].

During passage through the plasma, the filaments did not follow a straight trajectory. Instead, they sometimes veered, due to the influence of ion drag and/or thermal gradients. This was verified further by intentionally injecting carbon dust into the plasma. A shutter used on one viewport to block film deposition between LLS measurements proved to be a convenient source for injecting the carbon dust. This was done by placing dust on the closed shutter when the tool was opened. Then, by rapidly moving the shutter during processing by means of a vacuum feed-through, particles flew off the moving shutter and into the plasma. Like the ejected filaments, these particles also showed evident changes in trajectory while passing through the interelectrode plasma region.

Particle Detection on Disks

To further evaluate wall flaking and target effects, a holder containing a number of clean disks were placed between the targets for static deposition. The disk pallet was mounted at a sharp angle, around 80°, to allow for scanning across the disc surface from the laser mounted on the top of the tool. Backscatter was used to detect particles. Nine discs were placed on the pallet, although only six of them could be measured due to the angle of the pallet. The discs were scanned prior to, and during sputtering. In some cases, particle deposition on the discs 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.

Because this was done outside a clean room environment, many of the discs showed particles larger than 5 μm in size prior to deposition. However, after 9 h of deposition and even more so after 24 h of deposition, the number of particles on the disks increased greatly. Some particles were seen to move on the surface of the disks when the plasma was first started. On occasion, a particle was seen to fall through the opening in the center of the disc. Other than these isolated observations, however, the center open region of the disks had no noticeable effect on particle contamination or particle movement. No traps were observed in this region.

By monitoring different discs aligned opposite to various portions of the target, it was possible to analyze the variations in particle deposition on each disk and 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. This was especially evident for disks located in front of the centers of the targets, when compared to those disks closest to the racetracks. After 24 h of sputtering, one of the six discs within measurement range showed an impinged, melted filament. This disc was opposite the edge of a target, where a very high density of target filaments was also seen. In time, the particles observed on the disks also appeared to grow larger, probably due to film deposition over the particle. Except for during the first few seconds of operation, the surface particles did not move at any time. This indicates that particle contamination on the disks may become "buried" by subsequent film deposition.

DISCUSSION

It is seen that an important cause of contamination in sputtering process results from filament formation on the targets, followed by rapid growth of the filaments due to localized trapping effects, and is followed by heating of the filaments. Eventually, this results in fracture of the filament and violent ejection of the contaminant into the plasma. Once there, the particle rapidly becomes heated further due to attachment of electrons and neutralization by ion bombardment. This results in an ejected particle, one that may move fast and can become sufficiently hot to become embedded into the substrate, or which can be subsequently covered by film deposition.

The mechanism summarized above is illustrated in Figure 3, and is sequentially describes in detail below.

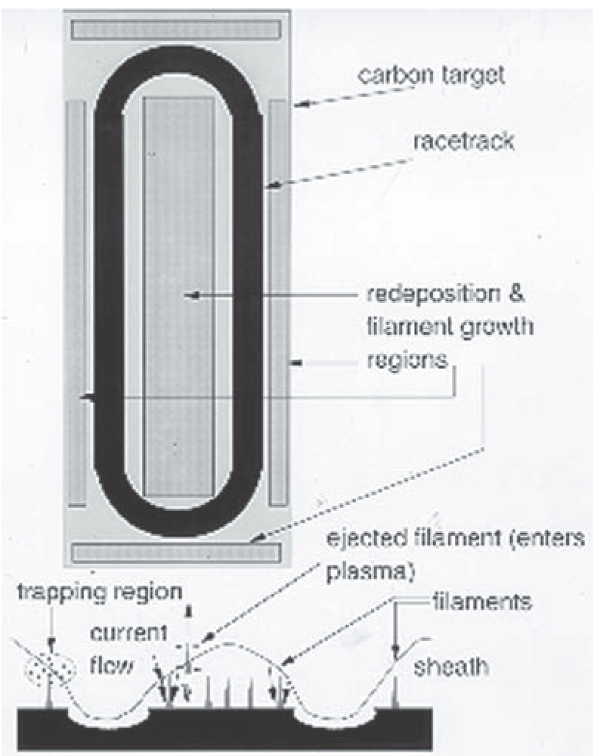

Figure 3: Schematic illustration showing the presence of filaments on the target surface of the sputtering tool, the presence of trapped, fine particles, and the fracture of these filaments, causing their violent release and heating in the plasma.

(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 charged, and are then subject to trapping effects near the surface of the target. In some cases, particles can be generated during chamber maintenance.

(2) Plasma chemical processes grow these and other very small particles to larger sizes, increasing their mass and net charge. Sputtering of metallic electrode sections augments other particle sources.

(3) Larger cross-sections of the particles result in deposition onto these nanoparticles, growing particles of sub-micron size.

(4) Ion drag, the force contributed by ions striking and nearly striking particles on their way to the target, carries the particles close to the target surface. This force is balanced by electrostatic repulsion at the sheath boundary and results in suspended particles very close to the target surface, typically less than 0.5 mm.

(5) Filaments and target roughness, especially in the weak plasma region between the racetrack and at the edges of the target, form electrostatic "traps" or disturbances that attract and confine these particles, in a manner as previously found for other plasma processes [21]. Ironically, target roughness may even be caused by inappropriate tool cleaning steps. The suspended particles move toward the filaments and surround them.

(6) 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 a greater trapping field. This attracts more of the small, suspended particles, resulting in a runaway process with ever-increasing size and trapping potential.

(7) As the filaments increases in size, they become a significant fraction of the sheath thickness. Since the filaments are also attached to the powered electrodes, they become a path for current flow. In essence, the impedance of the filament/sheath combination is less than the sheath impedance in the absence of filaments. Accordingly, increased current flows through the filaments. With increased current flow, the filaments heat up to high temperature and glow, emitting blackbody radiation. This explains the filament incandescence seen with prolonged plasma operation.

(8) As the filaments heat up, their connection to the electrode is eventually broken. This results from volatilization of a section of the filament, likely due to resistive heating. The remainder of the filament is now electrically isolated from the target and will become negatively charged by the rapid attachment of electrons.

(9) The negatively charged particle finds itself in the repulsive potential of the sheath field and is electrostatically repelled by qE where q is the charge of the filament and E is the time-averaged electrical field in the sheath. This rapidly propels the filament (now hot from ion/electron recombination) into the plasma.

(10) It is also possible that the arc that freed the filament from the target also contributes kinetic energy to the momentum of the particle from residual film stress. Evidence for this effect was seen in the changing shape of the ejected filaments with distance from the target.

(11) Movement of the filament is influenced by the kinetic energy released during vaporization, repulsion of the sheath, and the presence of the non-uniform plasma density typical for magnetron sputtering.

(12) The rapidly moving, red-hot filament then impinges into a wafer or disk, if the substrate is in its trajectory. The combination of heat and kinetic energy of the filament is sufficient to melt the filament into the disk. The much higher melting point of Si relative to Al, makes this step unlikely for silicon wafers.

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 is observed during film deposition, similarly to that observed during etching processes. However, significant differences are also observed. The highly non-uniform plasma density typical of magnetron sputtering processes are 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, whereas in high density regions any surface filaments formed would be sputtered away. When these filaments form near the high-density regions of the plasma, the sheath thickness is small due to the higher density of electrons. Because the filaments are resistively heated by the passage of current through them, this can result in explosive vaporization, carrying the filament away from the target. Combined with repulsion between the negatively charged filament and the sheath region, the vaporization process results in fast acceleration and movement of the filaments. Some filaments were observed moving as fast as 100 cm/s. Also, as the filaments cross the high-density regions of the plasma, electron/ion impingement and neutralization processes result in rapid heating of the particles, which have poor heat loss mechanisms due to the low operating pressures of sputtering processes. The result of this is the impingement of hot, fast-moving particles onto substrates, possibly causing melting and penetration by the contaminant particles.

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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