Chapter 14: Thin Film Deposition Processes

Up to this point in the course almost all of the emphasis has been placed on the techniques involved with certain activities related to achieving and characterizing a vacuum environment. Now we will turn our attention to the reasons for working so hard to achieve a vacuum: the processes that are conducted in this environment. The deposition of thin films has made a tremendous impact on the level of technology we utilize in our daily lives. Thin film coatings provide enhanced optical performance on items ranging from camera lenses to sunglasses. Architectural glass is often coated to reduce the heat load in large office buildings, and provide significant cost savings by reducing air conditioning requirements. Microelectronics as we know them today would not be possible without vacuum technology. Microcircuits fabricated in multi-step vacuum processes are used in devices ranging from wrist watches to microwave ovens to automobile ignition and monitoring systems. The computer industry would not exist if it were not for vacuum technology. In 1990 the world market for integrated circuits was \$50 billion; and for the electronic devices which rely on these microcircuits, \$0.9 trillion. Decorative coatings applied to jewelry and plumbing fixtures is another large industry based upon vacuum technology. Many of the components of plumbing fixtures are manufactured by depositing thin films of chromium onto injection molded plastic parts. The useful life of tool bits has also been increased by the application of thin films that are chemical compounds. Tool steel cutting tools used in lathes and mills are often coated with the chemical compound titanium nitride to reduce wear of the cutting edges. The deposition of thin films composed of chemical compounds may be performed in several ways. Co-deposition is a technique in which vapors of two different materials are generated simultaneously. These two vapors condense together, forming an alloy or compound. Other techniques for deposition of compounds include thermal evaporation of the compound (as is performed for salt coatings), sputtering of the compound, and reactive sputtering or evaporation. In the reactive processes, atoms of the evaporant (typically a metal) chemically react with gas species which are intentionally injected into the process chamber. Each of these processes will be described in detail.

Thin Film Deposition in a Vacuum Environment

Early references to the science of thin film deposition include the research conducted by Michael Faraday in 1857. In this series of experiments, Faraday created thin metallic films by exploding metal wires in a vacuum vessel. Historically, the techniques for thin film deposition have evolved in approximately this order: thermally induced evaporation (by electrical resistance heating, induction heating, and electron beam heating), sputtering (diode, triode, magnetron, ion beam), arc processes, and most recently, laser ablation.

In general, there are three steps in any physical vacuum deposition (PVD) process: creation of an evaporant from the source material, transport of the evaporant from the source to the substrate (item to be coated), and condensation of the evaporant onto the substrate to form the thin film deposit. There are two reasons why this process is best conducted under vacuum: 1. the process of evaporation involves significant amounts of heat, if oxygen were present, any reactive metal would form oxides; 2. collisions with

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gas molecules during the transport of evaporant from source to substrate would reduce the net deposition rate significantly, and would also prevent growth of dense films.

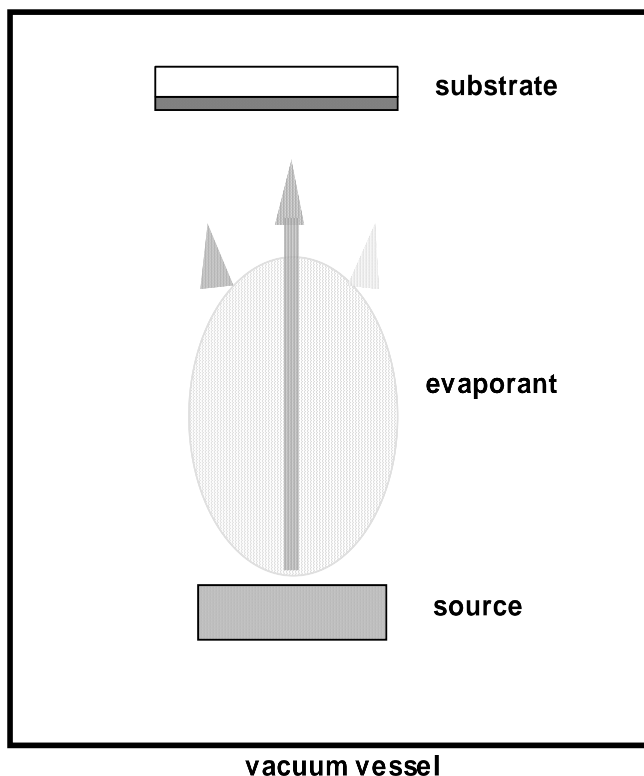

Figure 14.1 The three basic steps in any physical vapor deposition process: evaporation from the source, transport of evaporant, and condensation of the evaporant.

Upon arrival at the substrate, evaporated material condenses on the substrate in a complex sequence of events that determine many of the physical properties of the deposited film. The steps in the growth of thin films are generally referred to as nucleation and growth. In nucleation, the atoms and molecules which are arriving (called ad atoms) at the surface lose thermal energy to the surface, and the surface absorbs that energy. Depending on the amount of thermal energy the ad atoms and the surface have, the ad atoms move about on the surface until they lose the thermal energy required to move about the surface (referred to as Adam mobility). As nuclei continue to form, the film grows into a continuous sheet covering the substrate. Chemical interactions between the ad atoms and the surface determine the strength of the bond between the film and substrate. Gold, for example, does not form a chemical bond with silicon dioxide, and therefore, the adherence of gold films on glass are very weak. Improvement of this adhesion may be made by first depositing a thin (500Å thick) "Binder" layer of chromium or niobium, then depositing the gold over the binder layer. Chromium and niobium do form chemical bonds with the silicon dioxide in glass, and also form metallic bonds with the following gold layer. Once a few monolayers of evaporant have condensed on the substrate, the film continues to grow in thickness as if the entire substrate were made of the material being deposited. During film growth the

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microstructure of the deposited film will be developed. This microstructure may be described in terms of grain size, orientation, porosity, impurity content, and entrained gases. Normally, vacuum deposition processes are selected over other processes (electrochemical deposition, flame spraying, etc.) to achieve the following desirable properties:

1) High chemical purity.
2) Good adhesion between the thin film and substrate.
3) Control over mechanical stress in the film.
4) Deposition of very thin layers, and multiple layers of different materials.
5) Low gas entrapment.

For each of the vacuum deposition process described in this unit, keep in mind the ultimate goal is to provide a means for depositing a thin film having the required physical and chemical properties. The parameters one can control to achieve the specified goals are:

1) Kinetic energy of the ad atoms.
2) Substrate temperature.
3) Deposition rate of the thin film.
4) Augmented energy applied to the film during growth.
5) Gas scattering during transport of the evaporant.

By varying these parameters one can generate thin films of a given material that have different mechanical strength, adhesion, optical reflectivity, electrical resistivity, magnetic properties and density.

Thermally Induced Evaporation

In this process, heat is input into the source material (often called the charge) to create a plume of vapor which travels in straight-line paths to the substrate. Upon arrival at the substrate, the atoms, molecules, and clusters of molecules condense from the vapor phase to form a solid film. The heat of condensation is absorbed by the substrate. On a microscopic scale the localized heating from this process can be enormous. It is common, in the development of metal coating techniques for thin cross-section plastic parts, to melt substrates during the initial deposition runs. With experience, one can select source-to-substrate distances and deposition rates which will allow coating of temperature sensitive substrates without melting.

There are several methods by which heat can be delivered to the charge to cause vaporization: electric resistance heating, induction heating, and electron beam heating. Deposition of thin films by laser ablation and cathodic arc could be grouped in this section with thermal processes, but there are some unique characteristics of these techniques which are beyond the simple model of thermally induced evaporation. For this reason we will cover these two deposition techniques separately.

Resistance Evaporation

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Evaporation of material by electrical resistance evaporation is very likely the easiest of the thermal evaporation techniques. Quite simply, in a vacuum environment the charge (which may be an elemental metal, an alloy, a mixture or a compound) is heated to become a vapor. Low voltage, high current (typically 10 to 40 VDC, 1 to 10 amps) power is brought into the vacuum vessel using electrical power feed throughs. The electrical power is passed through a filament which is in intimate contact with the charge (see figure 13.1). Filaments are often heated to 1000 to 2000° C. A materials requirement for efficient thermal evaporation is that the charge have an appreciable vapor pressure at the operating temperature of the filament.

Table 14.1 Temperatures for efficient evaporation of selected metals.

Material	**Evaporation temperature, °C**	**Comments**
Zinc	325	High vapor pressure at RT
Aluminum	1390	
Copper	1516	
Chromium	1612	
Lead	1680	Toxic
Iron	1829	
Nickel	1848	

All materials evaporate, even at room temperature. The addition of heat simply accelerates the process. At a specified temperature the pressure of the vapor emitted by a material is called the equilibrium vapor pressure.

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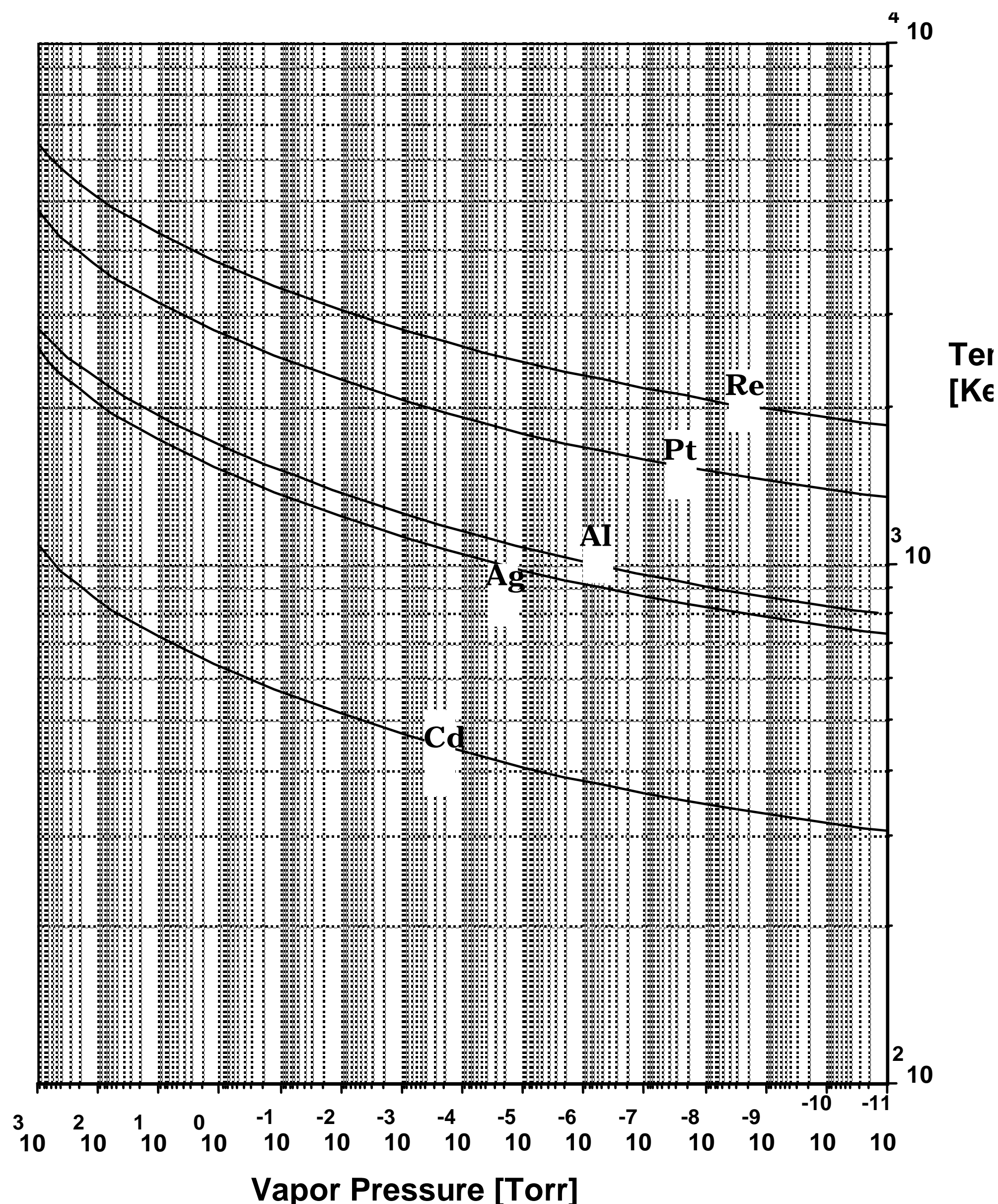

Figure 14.2 Equilibrium vapor pressure as a function of temperature for five metallic elements.

The relationship between the equilibrium vapor pressure of a material and the temperature generally follows the pattern shown in figure 14.2. Cadmium, for example has a significantly higher vapor pressure at all temperatures than any of the other metals shown in this figure. Similarly, Rhenium has the lowest vapor pressures of these five metals at all temperatures. Vapor pressure curves for many of the metals that are used as charge material are provided in Appendix Y. Numerical values for the vapor pressure of a given metal at a specified temperature may be read directly from vapor pressure curves like that shown in figure 14.2. Cadmium, for example, has a vapor pressure of approximately 5 x 10^{-3} Torr at 500 K (227 °C). By comparison, rhenium does not achieve a vapor pressure of 5 x 10^{-3} Torr until it is heated to almost 3000 K (2730 °C)!

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Sample Problem:

14.1 Use figure 14.2 to determine the vapor pressure of aluminum and silver at 1300 °C.

14.2 Equilibrium vapor pressure curves, such as that presented in figure 14.2 are useful for thin film evaporation. What other uses for this information can you imagine for vacuum technology?

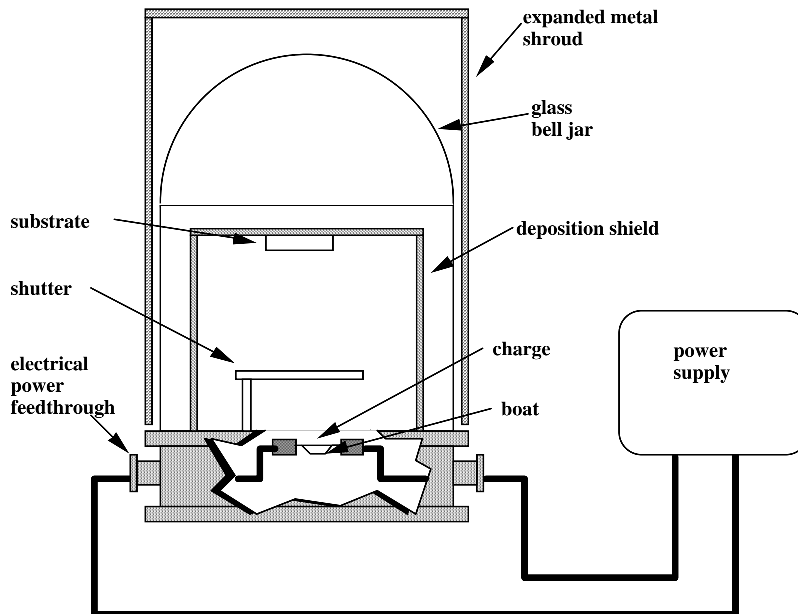

Figure 14.3 Bell jar vacuum system for deposition of thin films by electrical resistance heated thermal evaporation.

Thermal evaporation is typically conducted under high to ultra-high vacuum conditions. Bell jar type vacuum vessels are often selected to perform this process on a laboratory scale, as they offer a great deal of design flexibility as well as the advantage of direct observation of the process at a very reasonable cost.

Filaments are usually made of refractory metals such as Tungsten, Tantalum, or their alloys. Some of the requirements for a good filament material are:

1) High melting point.
2) Low solubility for the charge materials.
3) Filament should be wettable by the charge materials.
4) Filament should withstand thermal shocks well.

There exists a great variety of filament configurations ranging from straight and coiled wires to "boats" and boxes (see figure 14.4). Each type of filament is designed for a unique application. Coil filaments made of refractory metal strands are loaded with the charge by hanging small sections of wire made of the charge material on the coil. Upon heating, the charge melts, and wets the coil. Further heating causes the evaporation of

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the charge from the coil filament. Very rapid heating of a filament with hanging charge material may cause the charge to melt locally and fall off the filament.

Some skill is required to attain thin film deposits from run to run which have consistent thicknesses. One technique that helps is to carefully weigh the charge for each run, keeping the weight the same, and operating the filament so as to completely evaporate the charge each run.

There are some inherent disadvantages of resistance heated thermal evaporation that should be kept in mind when selecting a deposition technique:

1) The source may generate impurities which may co-deposit in the condensing thin film.

2) Accurate control of the deposition rate is difficult.

3) The composition of alloy thin films deposited may differ from that of the charge material (especially if the elements in the alloy have markedly different vapor pressures).

4) The amount of material which may be evaporated per run is limited.

5) The substrate will experience heating due to radiant energy from the source.

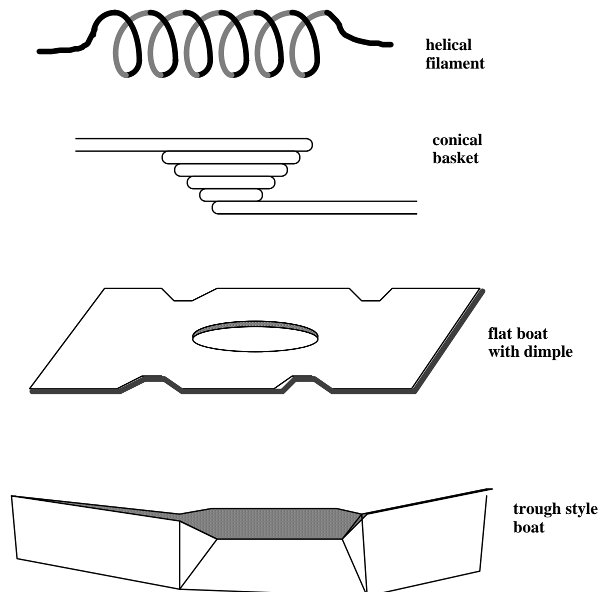

Figure 14.4 Configurations for filaments used in electrical resistance heated thermal evaporation.

Molecular beam epitaxy (MBE) is a modern application of electric resistance heated thermal evaporation. This technique is used to accurately deposit ultra-high purity semiconductor materials with specified crystallographic orientations between the layers of different materials. The vacuum environment for MBE is typically in the extreme ultra-

high vacuum range (10^{-9} to 10^{-11} Torr). Evaporation of materials in an MBE vacuum system is conducted using a special thermal evaporation source called a Knudsen cell.

Sample Problem:
14.3 Define "Epitaxy". How is an epitaxial thin film different than other films that are deposited on a crystalline substrate?. {Hint: see the dictionary}.

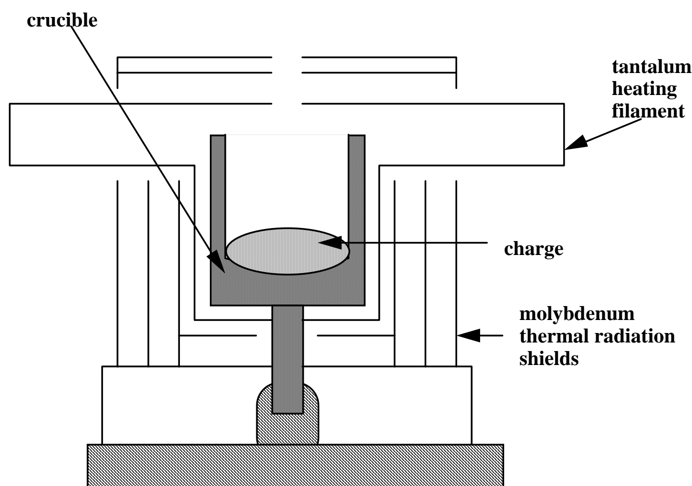

Figure 14.5 Knudsen cell used in the thermal evaporation of material in molecular beam epitaxy vacuum deposition system.

Induction Heated Thermal Evaporation

In this technique an electric current is induced to flow through an electrically conductive charge material by the application of radio-frequency (RF) alternating current. The RF current is generated by a power supply which may range in output from 1 to 50 kilowatts, depending on the size of the charge. The AC current is flowed through the copper coil which surrounds a refractory ceramic crucible.

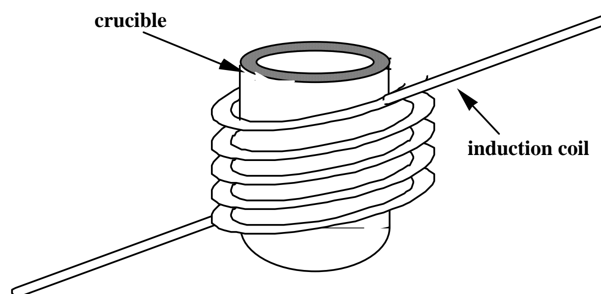

Figure 14.6 A crucible and coil used for induction heating for thermal evaporation.

For all forms of thermal evaporation which employ ceramic crucibles, selection of the appropriate material for the crucible is vitally important. If the incorrect selection is made, the charge may chemically react with the crucible, ruining both and possibly harming other components of the vacuum deposition system.

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Table 14.2 Maximum operating temperatures for refractory oxide crucibles.

		Crucible	material		
Metal to be evaporated	**ThO_2**	**BeO**	**ZrO_2**	**Al_2O_3**	**MgO**
Nickel	stable	stable	stable	stable	stable
Titanium	<1800 °C	<1800 °C	<1800 °C	<1800 °C	<1800 °C
Silicon	<1600 °C	<1600 °C	<1600 °C	<1400 °C	<1400 °C
Zirconium	<1800 °C	<1600 °C	<1800 °C	<1600 °C	no data
Tantalum	<1900 °C	<1600 °C	<1600 °C	<1900 °C	<1900 °C
Molybdenum	<2300 °C	<1900 °C	<2300 °C	<1900 °C	<1900 °C
Tungsten	<2200 °C	<1800 °C	<2000 °C	<1900 °C	<1900 °C

Advantages of induction-heated thermal evaporation as compared to electrical resistance evaporation include:

1) Low contamination of the deposited thin films .
2) Improved control of deposition rate.
3) Larger charges can be loaded per deposition run.

Disadvantages of induction heated thermal evaporation include:

1) The charge must be electrically conductive.
2) RF power supplies and matching networks can be expensive and quite large.
3) Chemical interaction between the charge and crucible can occur.

Sample Problem:

14.4 What property of materials makes thermal evaporation possible? How can one manipulate this property to deposit thin films of materials?

Electron Beam Evaporation

In this thermal evaporation process, a beam of energetic electrons generated from a heated filament supplies the thermal energy to evaporate of the charge. There are several variants of the electron beam evaporation process. All electron beam thermal evaporation systems have an anode (biased positively) and a cathode (either grounded, or biased negatively with respect to the anode). The two divisions we will cover are self-accelerated and work-accelerated electron beam evaporators. In the work-accelerated scheme, electrons emitted from a heated tungsten filament are attracted to the charge

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material by an applied high voltage bias (10- 40 kV DC). A focusing aperture aids in minimizing spurious heating of the vessel interior by off-axis electrons.

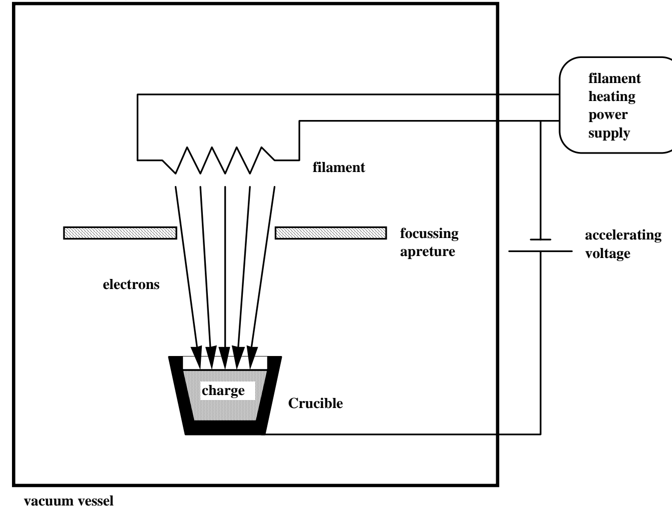

Figure 14.7 A work-accelerated electron beam evaporator.

Several configurations of the work-accelerated electron beam evaporator have been designed and used. In figure 14.8 electrons emitted from the heated cathode are caused to travel in an oval shaped path by the electrostatic negative field applied to the cylindrical focusing electrode. Water cooling is provided via concentric tubes connected thermally to the bottom of the charge. This design has a distinct advantage over that shown in figure 14.7: the evaporant may be directed at a substrate placed above the source without interference by a focusing aperture or filament. Additionally, the focusing aperture and filament do not become heavily overcoated with evaporant.

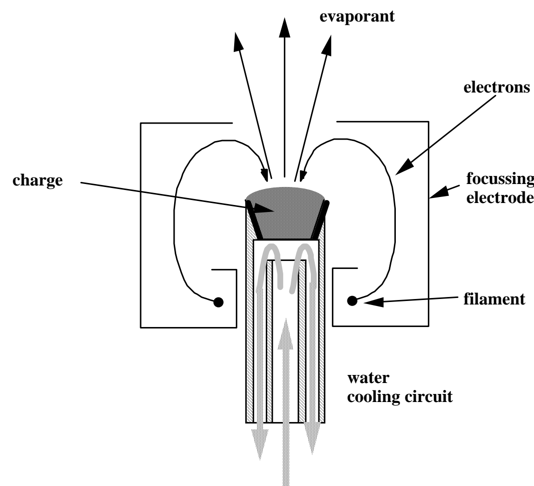

Figure 14.8 Work-accelerated electron beam evaporator with a cylindrical electron focusing electrode.

Some work-accelerated electron beam evaporators use electromagnetic coils to steer and focus the electron beam as shown in figure 14.9. This system provides several

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advantages: source utilization can be controlled and maximized by rastering the electron beam; and the effective source size can be made large by rastering the beam, improving the thickness uniformity and coverage of the substrate. Note that in this design the electrons emitted from the filament impact the backside of the cathode, heating it so that it will in turn emit electrons. The cathode area which emits electrons is hemispherical, which improves the three-dimensional uniformity of the electron beam emitted from it.

Self-accelerated electron beam evaporators use an auxiliary anode to impart kinetic energy to the electrons emitted by the filament (cathode). This allows the evaporation of charge materials which are not electrically conductive, such as calcium fluoride, which is used to coat camera lenses for improved optical performance. A self-accelerated electron beam gun configuration is presented in figure 14.10.

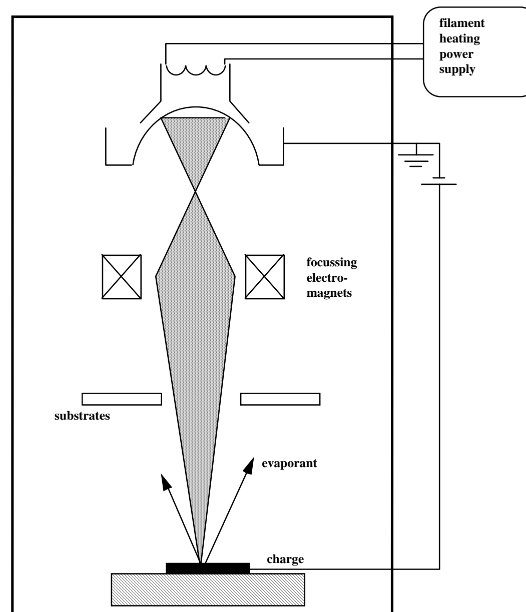

Figure 14.9 Work-accelerated electron beam evaporator electromagnets to focus and steer the electron beam on the charge material.

As was the case with the work accelerated electron beam evaporator in figure 14.9, the self-accelerated gun has a set of electromagnets which may be used to scan the electron beam during evaporation. Automated scan controls for both types of guns are commercially available. These scan controls vary the current in the electromagnets so as to sweep the beam in a "Lissajous" pattern, which is sinusoidal in two dimensions.

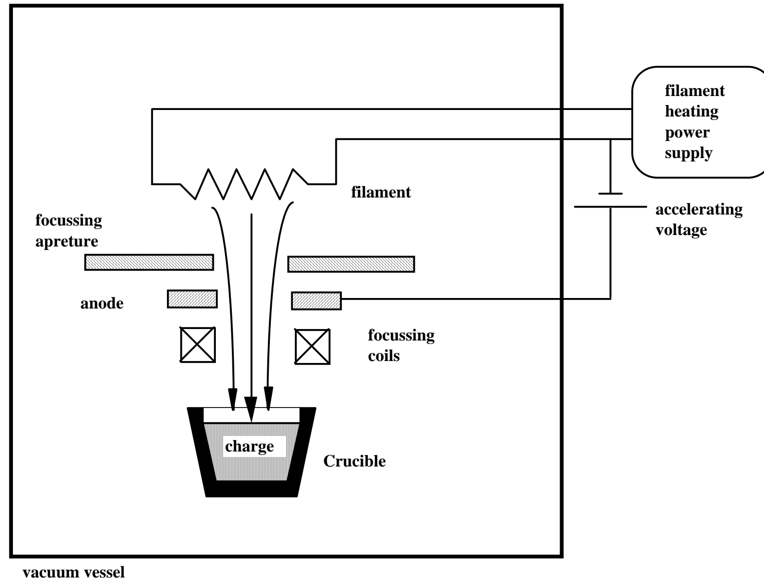

Figure 14.10 Self-accelerated electron beam evaporation.

The majority of commercial electron beam evaporators are of the transverse design, as shown in figure 14.11. These guns use a permanent magnet to steer the electrons emitted from the cathode around 270°, and a set of electromagnets to raster the beam on the charge material. Modern electron beam guns of the transverse design are available with a rotating multiple pocket hearth, which allows deposition of up to five different materials without venting the vacuum vessel. Some of these units also have integral shutters which allow the gun to achieve a stable operating temperature before deposition of the substrate is begun.

Since the process of electron beam evaporation is normally performed under UHV conditions, the evaporant generally travels in straight-line paths from the source to the substrate. To aid in attaining uniform thickness coatings by this technique, substrates are often mounted on "carousels" which rotate, and may wobble or spin individual substrates each revolution (see figure 14.12). Deposition shielding is often placed inside the vacuum vessel to facilitate cleaning. Multiple sets of shields allows for continuous operation of the system: one set may be cleaned while the other set is in operation.

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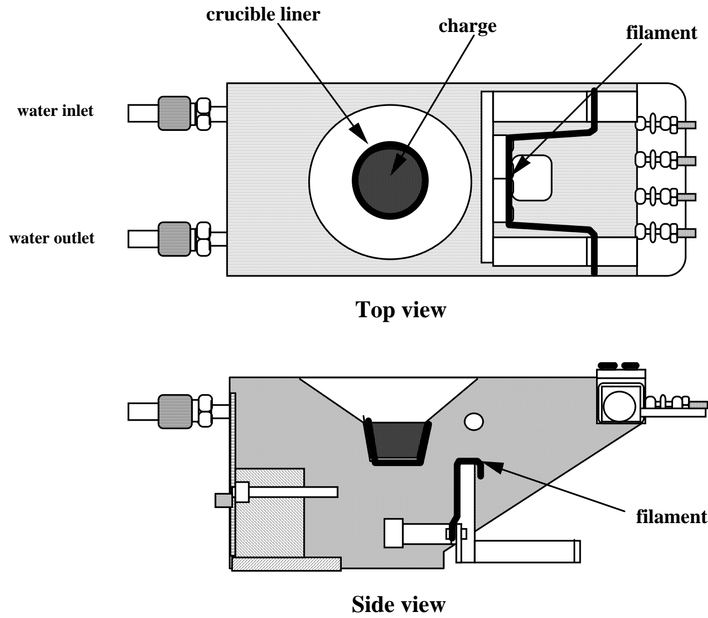

Figure 14.11 Transverse design electron beam evaporation gun.

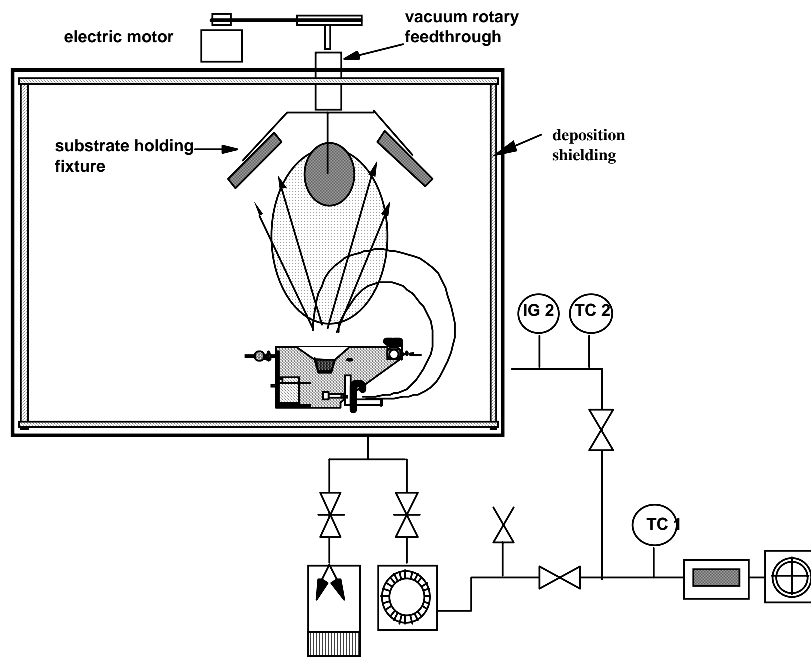

Figure 14.12 Transverse design electron beam evaporation gun mounted in a vacuum vessel.

Plasma Electron Beam Deposition Sources

Instead of using a heated filament to generate the electrons for evaporation of a material, plasma guns utilize the electrons which exist in high temperature gases called plasmas. Two types of plasma sources exist: cold cathode and hot cathode.

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In the cold cathode design (figure 14.13), the cathode is biased negatively from -5 to -20 kV, the rest of the source is at ground potential. Following evacuation of the vacuum system to its base pressure, process gas, such as helium is injected into the system. A partial pressure of from 1 to 100 mTorr is dynamically maintained using flow controllers at the process gas inlet, and adjustable apertures at the high vacuum stack inlet. Electrons emitted by the cathode can ionize process gas atoms, creating positive ions. These ions are accelerated to the cathode, and upon impact, generate a cascade of secondary electrons. Many of these secondary electrons escape the source and are accelerated and focused on the work piece as described in the section on work accelerated electron beam guns.

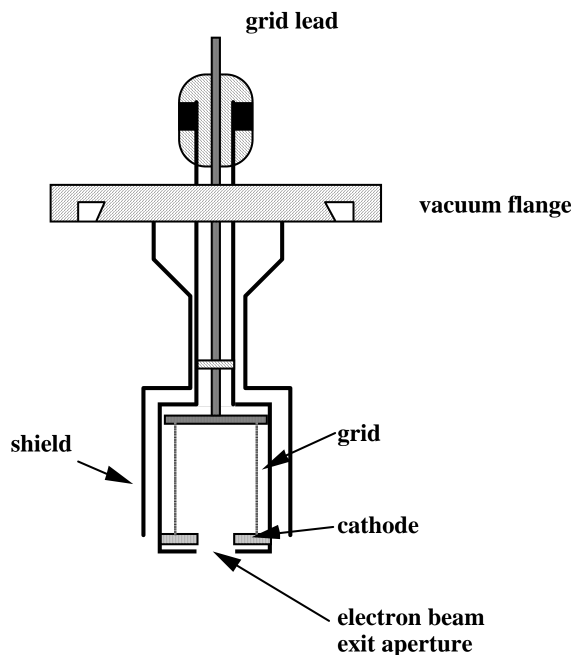

Figure 14.13 Cold cathode electron beam source.

Sample Problems:

14.5 A process gas is used in the operation of the cold cathode electron gun. What effect may this have on the uniformity of the deposited coating?

14.6 List and describe the methods by which electrons can be generated to provide the heat input to conduct a thermal evaporation under vacuum.

14.7 Describe the difference between work accelerated and self-accelerated electron beam evaporation systems.

14.8 What would be the consequences of the presence of a partial pressure of oxygen in an electron beam evaporation deposition system?

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14.9 Calculate the mean-free-path in a vacuum vessel at a pressure of 5×10^{-7} Torr at room temperature. From this data what could you predict about the path vapor atoms and molecules would take between the evaporation source and the substrate? Can you think of situations in which this would be advantageous or disadvantageous?

The Hot Hollow Cathode Electron Beam Gun

Following evacuation to the vessel's base pressure, a process gas, such as Argon is flowed in a controlled manner through the cylindrical hollow cathode. Radio frequency AC electric current is supplied to the cathode of the electron beam source from a low voltage, high current power supply. Ionization of the process gas occurs as a result of the applied electrical power. The cathode operating temperature is very high. For this reason, the cathode is made of a refractory metal, such as tungsten, and must be actively water cooled. Electrons emitted from the hot cathode and from the plasma may be steered and accelerated as described for other electron sources.

Very high deposition rates may be achieved using the hot hollow cathode electron beam source to perform thermal evaporation of materials. The electrons emitted from this source may be work or self-accelerated, and may be magnetically steered.

Sample Problems:

14.10 The operating pressure in a thermal evaporation deposition chamber using a hot hollow cathode electron source is approximately 50 mTorr, with Argon as the process gas. Calculate the mean-free-path at this pressure, and describe the effect this will have on the deposited thin film.

14.11 If you were interested in depositing a thin film of Zirconium Oxide, which of the thermal evaporation deposition processes described would you select? Why?

14.12 Describe methods by which thickness uniformity in thin film coatings can be improved for films made by thermally evaporation.

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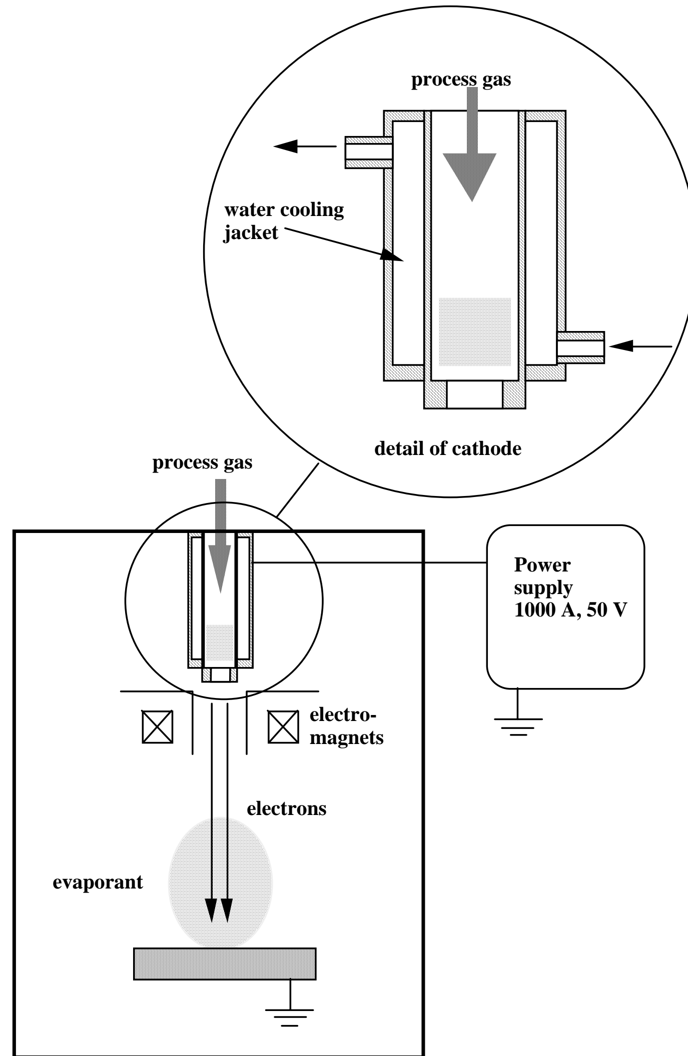

Figure 14.14 Hot hollow cathode electron beam source.
Safety notes for electron beam evaporation systems.

There are several potential dangers associated with the use of high energy electron beams that operators should be aware of:

1) Radiation: A significant amount of X-rays are generated whenever high energy electrons impact materials. The maximum energy of the X-rays is limited by the electrical potential applied to accelerate the electrons. A radiation survey of the exterior of the vacuum vessel, especially viewports, should be conducted to insure a safe working environment.

2) If the beam control systems fail, high energy electrons may be directed to the vacuum vessel walls or to internal fixturing, including water lines. The electron beam sweep pattern should be monitored carefully at the beginning of a deposition run, and periodically during the run. Leaving an electron beam system to run unattended is not recommended.

3) High voltage and high current are used in the process. The vacuum vessel and power supplies should be electrically grounded.

Sputter Deposition of Thin Films

Unlike thermal evaporation, in which a material is heated to increase its equilibrium vapor pressure, in sputter deposition, material is made to go into the vapor phase by the physical interaction of particles impacting the source material (often referred to as the "target"). There is a wide variety of sputtering techniques that are currently used to deposit thin films for use in magnetic storage media (cassette tapes, computer discs), optical thin films and microcircuits. The forms of sputtering which will be described in this section are: diode sputtering, magnetron sputtering, RF sputtering, and ion beam sputtering.

Diode Sputtering

A simple diode sputtering process system is presented in figure 14.15. Following evacuation of the vessel to its base pressure to reduce contamination of the film by atmospheric gases or water vapor, a process gas (most often Argon) is admitted into the vessel. The pressure of this process gas is maintained at a user-selectable pressure between 1-100 mTorr using a set of upstream mass flow controllers to regulate the process gas mix, as well as a downstream throttling gate valve. Such a valve, situated right at the turbpuump, is equipped with a controller that can perform fast, realtime pressure control using a capacitance manometer as a sensor element. An electric bias of from 500 to 5000 V DC is applied to the target. Electrons emitted by the target strike process gas molecules in the vicinity of the target, and may cause the gas to become ionized. The positive ions thus created are accelerated towards the cathode by the applied negative bias. When the positive ions collide with the cathode, the kinetic energy transferred is sufficient to eject atoms of the cathode material. Secondary electrons, ions, and light (IR, visible, UV and X-rays) are also emitted during this collision.

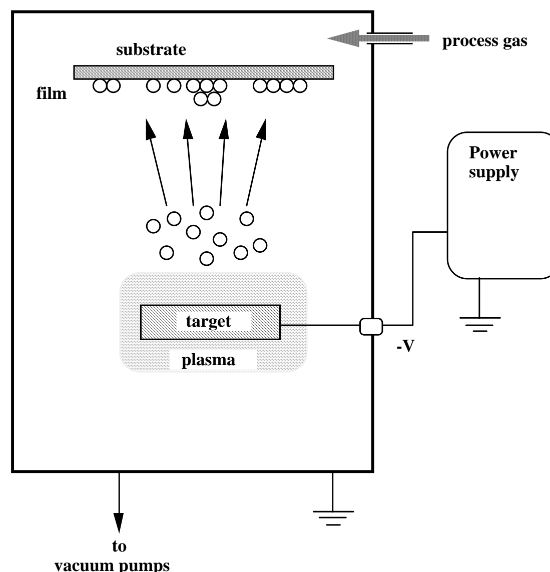

Figure 14.15 Simplified representation of a diode sputter deposition process.

The ejected (sputtered) material travels towards surfaces in the vacuum vessel where it condenses to form films. Since the process gas pressure is in the range of from 1 to 100 mTorr, a significant amount of scattering of the sputtered material by the process gas occurs. In this scattering, sputtered material loses its directional identity, may become neutral, if it was ionized, and also loses kinetic energy. The visible glow that surrounds the cathode during sputtering is called the "glow discharge" or plasma. Visible light of the plasma has a color which is characteristic of the process gas and the material being sputtered. This visible light may be used to monitor the chemical composition of the plasma using a visible light spectrometer (details of this and other deposition monitoring techniques will be covered later in this unit). One requirement of the diode sputtering technique is that the cathode be electrically conductive. Elements and compounds which are insulators can be sputtered by other techniques.

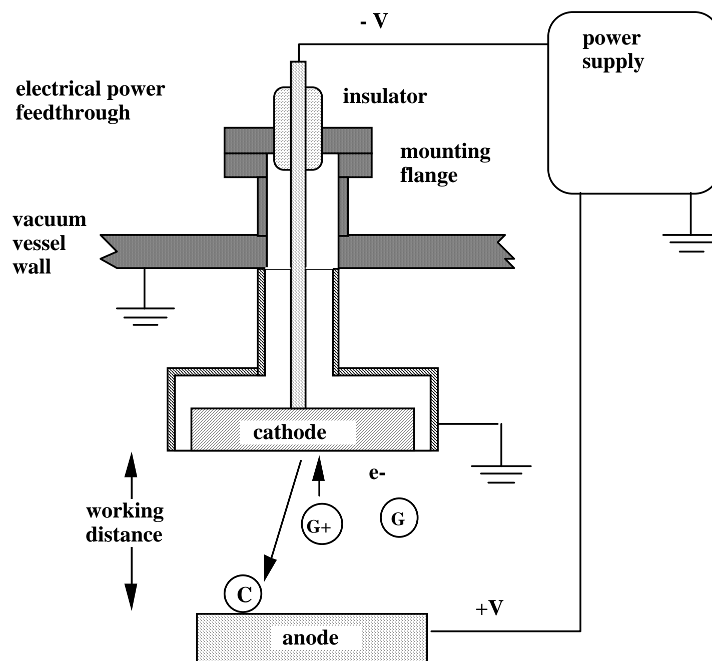

Figure 14.16 Detail of the operation of a diode sputter deposition process.

Typical operating parameters for DC diode sputtering are:

Current density at the cathode:	1 mA/cm^2
Process gas pressure (Argon):	30 to 80 mTorr
Cathode bias:	-3000 VDC
Working distance	2 to 8 cm

Depending on the composition of the cathode, this set of parameters should yield a deposition rate of from 60 to 400 Å/minute. It should be noted that the rate of erosion of

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the cathode is expected to be greater than the rate of deposition at the substrate. Some of the sputtered material condenses on the inner surfaces of the vacuum vessel (a good reason for removable deposition shields), and some may react chemically with residual gases to form volatile by-products which are pumped away.

Now that the concept of diode sputtering has been introduced, a few of the relevant concepts that apply to all sputtering processes will be covered.

Sputtering Yield

The number of target atoms which are ejected from the cathode for every incident ion is called the "Sputtering yield". The magnitude of the sputtering yield is a function of the composition of the process gas and target material, the energy of the incident ions, and the angle of incidence of the ions on the target. In general, the sputter yield is greatest for the following set of conditions:

1) High atomic weight process gas.
2) Low atomic weight cathode material.
3) Low concentration of reactive gas species in the vessel.

The noble gas Argon is the most commonly employed process gas for sputter deposition processes, as it has a high sputter yield for most metals, is chemically inert and non-toxic, and is relatively inexpensive (compared with the other noble gases (Krypton and Xenon).

Table 14.3 Sputter yields for several elements at 200, 600 and 1000 V DC.

Target material	**200 V**	**600 V**	**1000 V**
Silver	1.6	3.4	no data
Aluminum	.35	1.2	no data
Gold	1.1	2.8	3.6
Cobalt	0.6	1.4	no data
Molybdenum	0.40	0.9	1.1
Silicon	0.2	0.5	0.6

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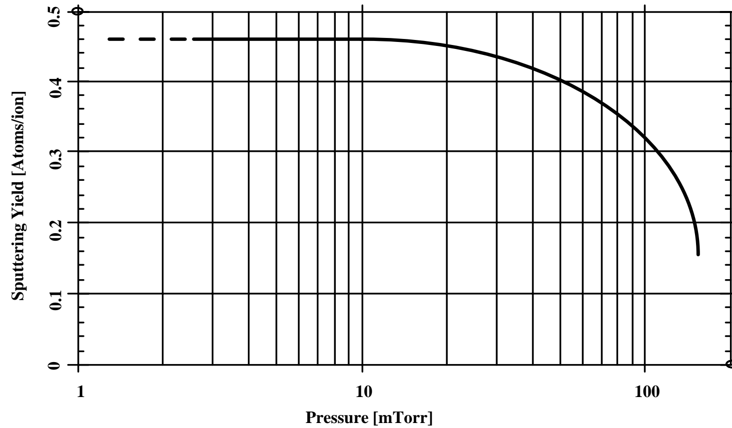

Figure 14.17 Sputter yield as a function of process gas pressure

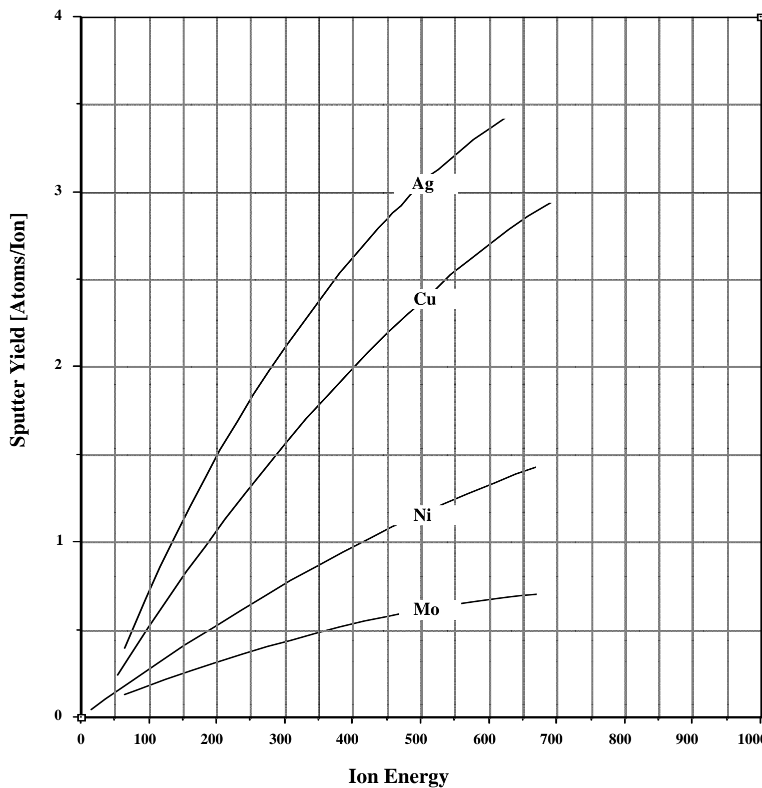

Figure 14.17 Sputter yields for metals sputtered with Argon as a function of ion energy.

It was shown, in the section on thermal evaporation of materials, that there exists a wide range of vapor pressures for materials, the vapor pressure at a given temperature being proportional to the evaporation rate. The magnitude of the variation in sputtering rate is much smaller. Most metals exhibit sputter yields that are between 1 and 5 atoms per ion when sputtered with Argon as the process gas. Prediction of the sputtering behavior of metals is made relatively easy by this fact.

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Sputtering Alloys and Compounds

The deposition of thin films of metallic alloys and some chemical compounds may be accomplished by sputtering. In general, the composition of thin films deposited by sputtering will have the same overall chemical composition as the source (target) after an initial equilibration period. In figure 14.18 is presented a simplified representation of a two component alloy target, made of "A" and "B". Assume that the sputtering yields for material "A" is higher than for material "B". Initially, as material is sputtered from this compound cathode the vapor stream will be higher in concentration of "A" due to its larger sputter yield. After some time, the surface of the cathode will become depleted in "A" and more concentrated in "B". When this occurs, the thin films being deposited will have approximately the same composition as the target.

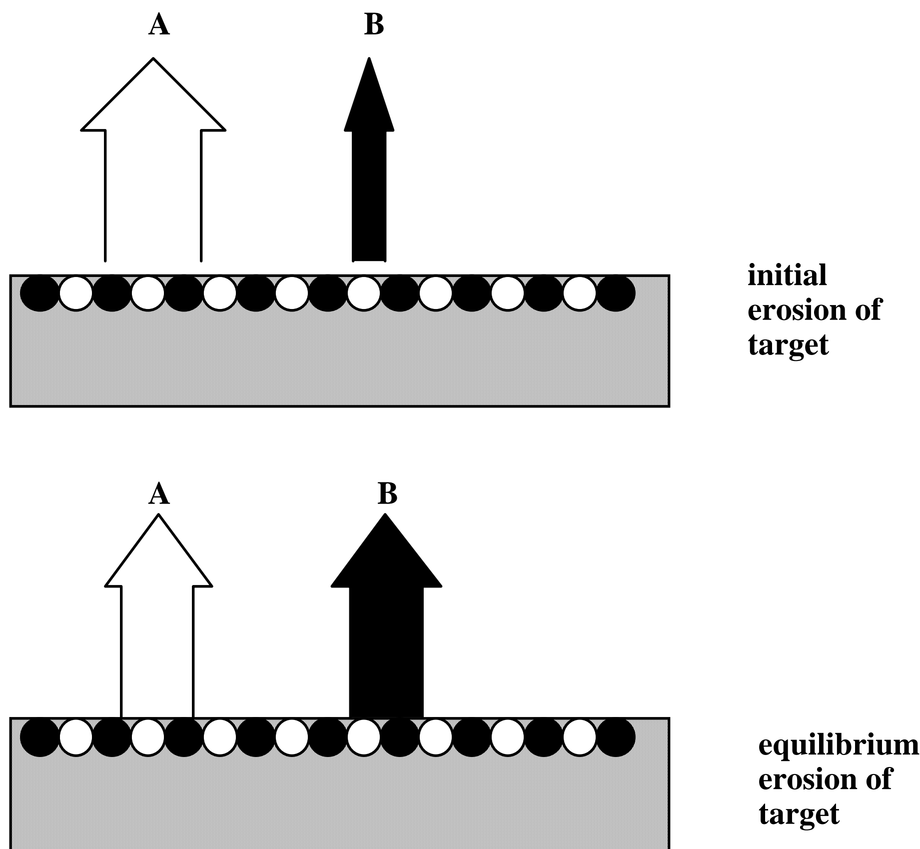

Figure 14.18 Sputtering alloy target made of "A" and "B".

Characteristics of Sputtered Material in the Vapor Phase

The vapor emanating from a sputter deposition source cathode contains neutral atoms, ions (both positive and negative), electrons, neutral clusters of atoms and charged clusters of atoms. Of these, the vast majority are neutral atoms. These atoms have kinetic energies approximately 50 to 100 times that of neutral atoms generated from thermal evaporation sources. This additional energy is thought to be the reason for the

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greater adhesion often observed for sputter deposited films over thermally evaporated films of the same material. Due to the relatively high pressure in an operating sputter deposition chamber, the mean-free path of sputtered species is short. The numerous gas-phase collisions which the sputtered material suffers between the target and substrate tend to reduce the amount of kinetic energy the depositing species have upon arrival. This affects the ad atom mobility and therefore, the density and crystal structure of the thin film. When sputtered atoms lose energy by gas collisions, they are said to be "thermalized", that is, their kinetic energy is reduced to equal that expected for similar atoms at the ambient temperature. A plot of the average distance sputtered tantalum and aluminum atoms can travel before becoming thermalized as a function of process gas pressure is presented in figure 14.19.

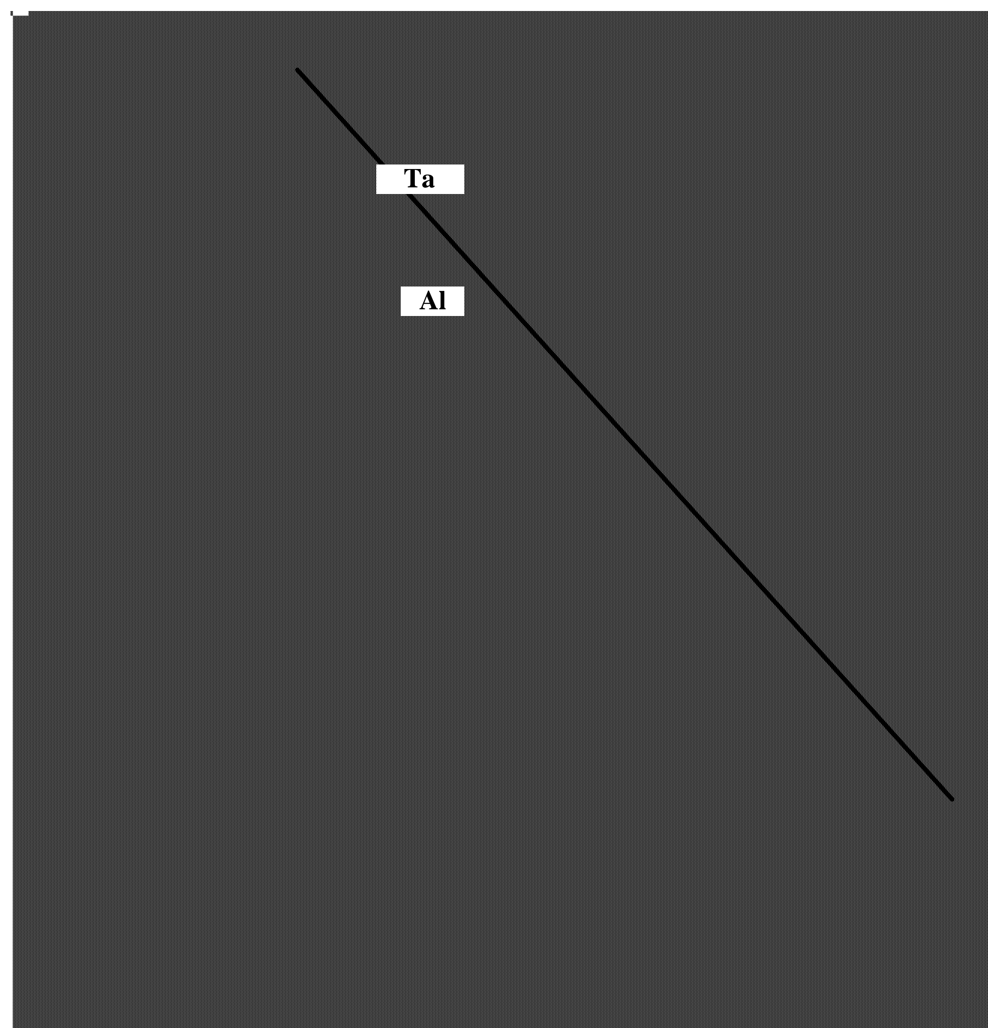

Figure 14.19 Distance tantalum and aluminum sputtered atoms travel before becoming "thermalized" as a function of process gas pressure..

Gas scattering has been used to some advantage in the coating of substrates having complex geometries. By operating at the high end of the pressure range (70-100 mTorr)

the maximum number of gas phase collisions may be induced, effectively reducing the "directionality" of the deposition flux from source. Using this technique the insides of tubes having aspect ratios (length: diameter) of 10:1 have been coated.
Now that some of the features common to all sputtering processes have been described, we will return our attention to the methods of sputtering.

Triode Sputtering

In this process, an auxiliary set of electrodes are employed to enhance the electron emission and increase the ionization of the process gas to generate a higher flux of material sputtered from the cathode.

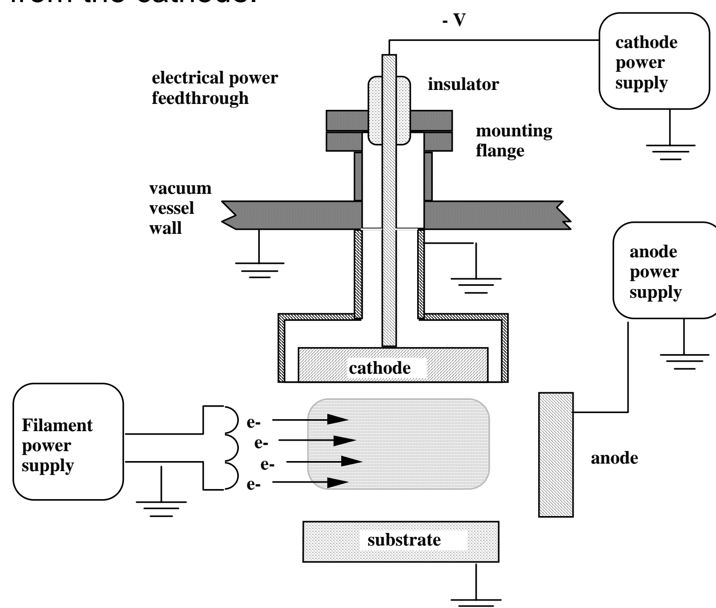

Figure 14.20 Detail of the components of a triode sputter deposition system.

The configuration of electrodes in a triode sputter deposition system is presented in figure 14.20. Three separate power supplies are used: one to resistively heat a filament to emit electrons, one to accelerate the electrons into the anode, and a high voltage power supply to accelerate positive ions towards the cathode. High deposition rates (>1000 Å/ minute) may be achieved using this configuration. Typical ranges for the operating parameters are:

Current density at the cathode:	10-100 mA/cm^2
Process gas pressure (Argon):	0.5 to 30 mTorr
Cathode bias:	-50 to -500 VDC
Filament current	0.05 to 1 amp
Anode bias	+ 50 to 100 VDC
Working distance	2 to 20 cm

Due to the high currents and the intense plasma generated, active water cooling of the sputter target and electron accelerating anode is required. The power supplies should

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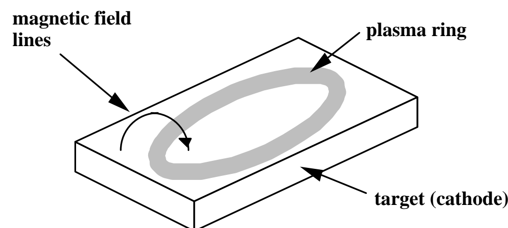
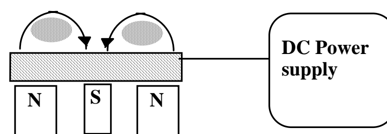
be interlocked with cooling water flow monitors to interrupt power in the event of cooling water loss. Triode sputtering systems have successfully been operated for deposition of a wide range of materials, but the complexity of the process, and the difficulty in scaling the process hardware up in physical size has limited its commercial application.

Magnetron Sputter Deposition

Another technique by which the deposition rate achieved over that of the simple diode sputtering process may be increased is through the use of magnetic fields to constrain the plasma close to the sputter target. Magnets situated beside or underneath the target of a diode sputtering source can be used to constrain the electrons emitted from the cathode to orbit in close proximity of the cathode. The probability that such an orbiting electron will strike a process gas molecule, causing an ionization, is greatly increased without the need to increase process gas pressure. The strength of the magnetic field and placement of the magnets with respect to the cathode is crucial to the proper operation of a magnetron sputter deposition source. In figure 14.21 the plasma ring generated on a planar magnetron as well as a cross section of a magnetron showing the magnetic structure under the cathode. Since the plasma is very localized, the current density can be quite high at the cathode surface, generating a significant heat load, which is very ineffectively dissipated by thermal radiation. Active water cooling of the cathode provides the means to control temperatures, and to prevent demagnetization of the permanent magnets under the cathode. A diagram showing more detail of the design of a magnetron sputter source is presented in figure 14. 22.



Oblique view of planar magnetron cathode.



Side view of planar magnetron cathode showing magnetic structure.

Figure 14.21 Configuration of a planar magnetron sputter deposition source.

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The DC magnetron sputter deposition source has found wide application in industry. Deposition systems using this source range from desktop units for deposition of thin films for prototype electronic devices to 150' long architectural glass coating chambers which use arrays of magnetron sputter guns to coat 4 x 8 foot sheets of glass with several layers in a single pass. Sputtering of all areas of the gun except the cathode is prevented by the use of a ground plane shield (see figure 14.22). This shield, along with the water cooling lines are electrically insulated from the high voltage applied to the cathode through the use of ceramic insulating spacers. It should be noted that in some magnetron sputter guns the water lines (often made from formed stainless steel bellows) are used to conduct high voltage power to the cathode, and are electrically isolated at the vacuum feed through. Under some circumstances, sputtering of these biased lines may occur, which has been known to lead to rupture of the water cooling lines within the vacuum vessel.

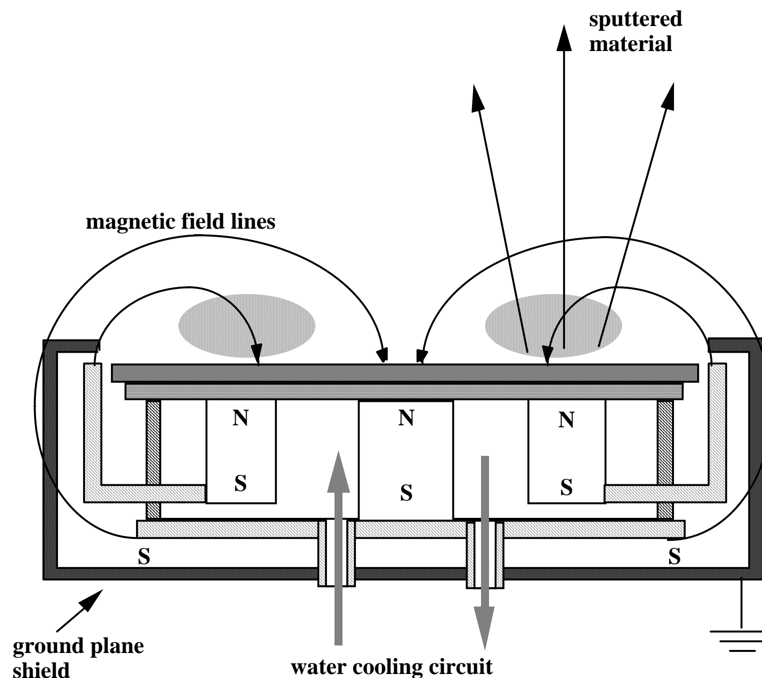

Figure 14.22 Magnetic structure and cooling circuit of a magnetron sputter gun

In addition to the planar magnetron configuration, there has been developed a cylindrical or "Post" magnetron source which may be used to deposit thin films onto the inner surfaces of tubes (see figures 14.22 and 14.23). Electromagnets may be used instead of permanent magnets to generate the magnetic field which constrains the electrons to orbit near the cathode. Using electromagnets it is possible to vary the position of the plasma with respect to the cathode by changing the field strength in sets of magnets. If the magnets are mounted outside the vacuum environment, as shown in figure 14.

Sample Problem:

14.13 What factors influence the sputtering yield? How can the sputtering yield be maximized?
14.14 What is the function of the process gas in diode sputter deposition?

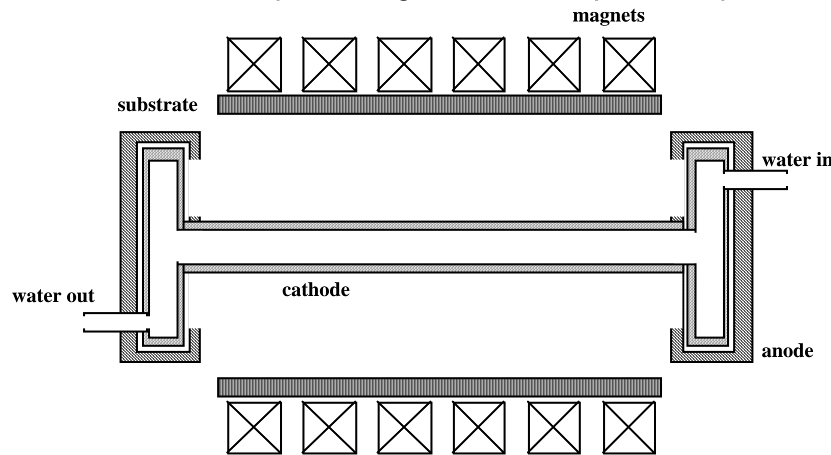

Figure 14.22. Detail of cylindrical post magnetron with electromagnets arrayed outside the tube to be coated.

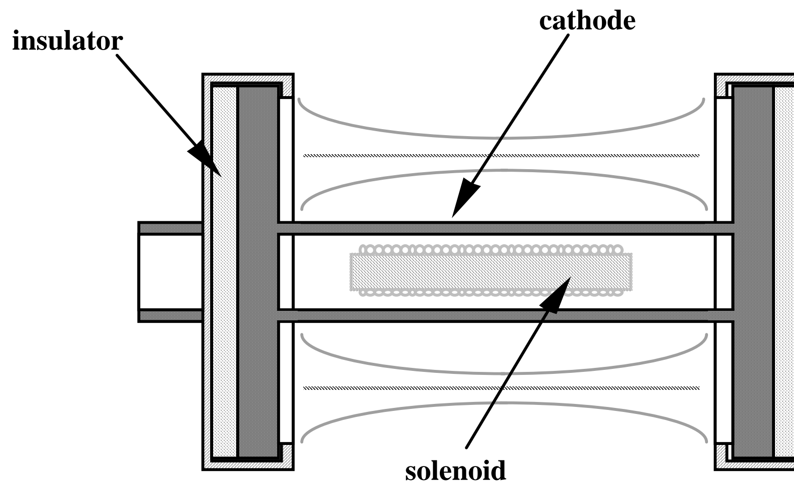

Figure 14.23. Detail of cylindrical post magnetron with electromagnets arrayed inside the cathode.

Typical ranges for the operating parameters of magnetron sputter deposition sources are:

Current density at the cathode:	10-50 mA/cm^2
Process gas pressure (Argon):	3 to 50 mTorr
Cathode bias:	-400 to -2000 VDC
Working distance	2 to 20 cm

Sample Problem:

14.15 What would be the consequences of an interruption in cooling water flow to a magnetron sputter source?

Power Supplies for DC Sputtering

Electric arcs and the resulting high voltage spikes which may occur during warm-up of DC sputter deposition sources can cause damage to power supplies not specifically designed for this application. These arcs are caused by electrical charge buildup on the thin naturally occurring oxide surface layer on the sputter cathode. As the target is bombarded with ionized process gas, the thin oxide layer will sputter away, and arcs should no longer occur. It is strongly recommended that only power supplies designed for sputtering applications be used. These power supplies have internal arc suppression circuitry which is intended to protect the power supply from damage.

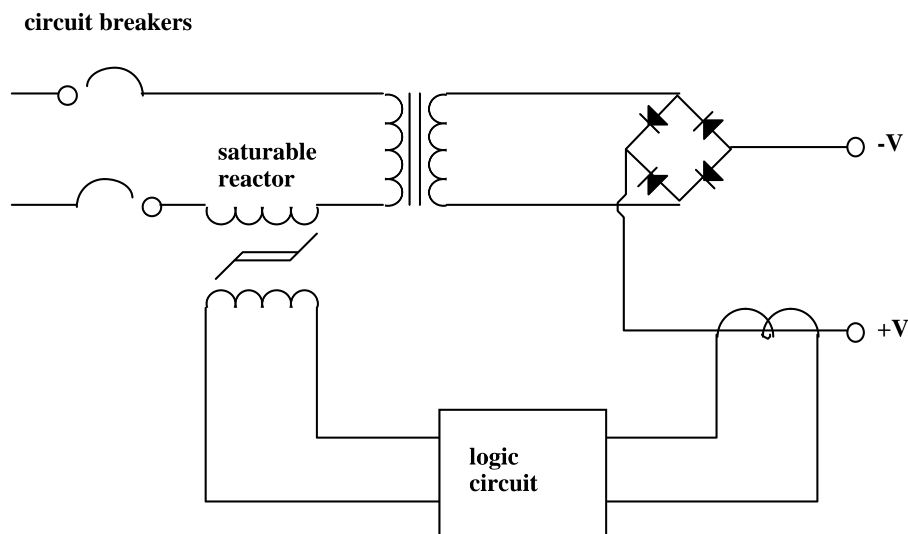

Figure 14.24 Schematic of a power supply designed for DC sputtering applications.

One limitation to the deposition of thin films by any of the DC sputtering techniques just covered is the requirement that the material to be sputtered be electrically conductive. This restriction is not true for radio frequency AC sputtering. In this process an AC power supply is used to apply a voltage that varies sinusoidally with time to a set of electrodes in a vacuum vessel. Attached to one of the electrodes is a target made of the material which is to be sputter deposited. If the material to be RF sputtered is an electrical conductor, an electrical insulator is placed between it and the electrode. When the electrodes are energized, a net negative bias occurs on the sputter target, which is electrically insulated from the electrode. Substrates to be coated may be placed on the other electrode, or mounted elsewhere in the vessel.

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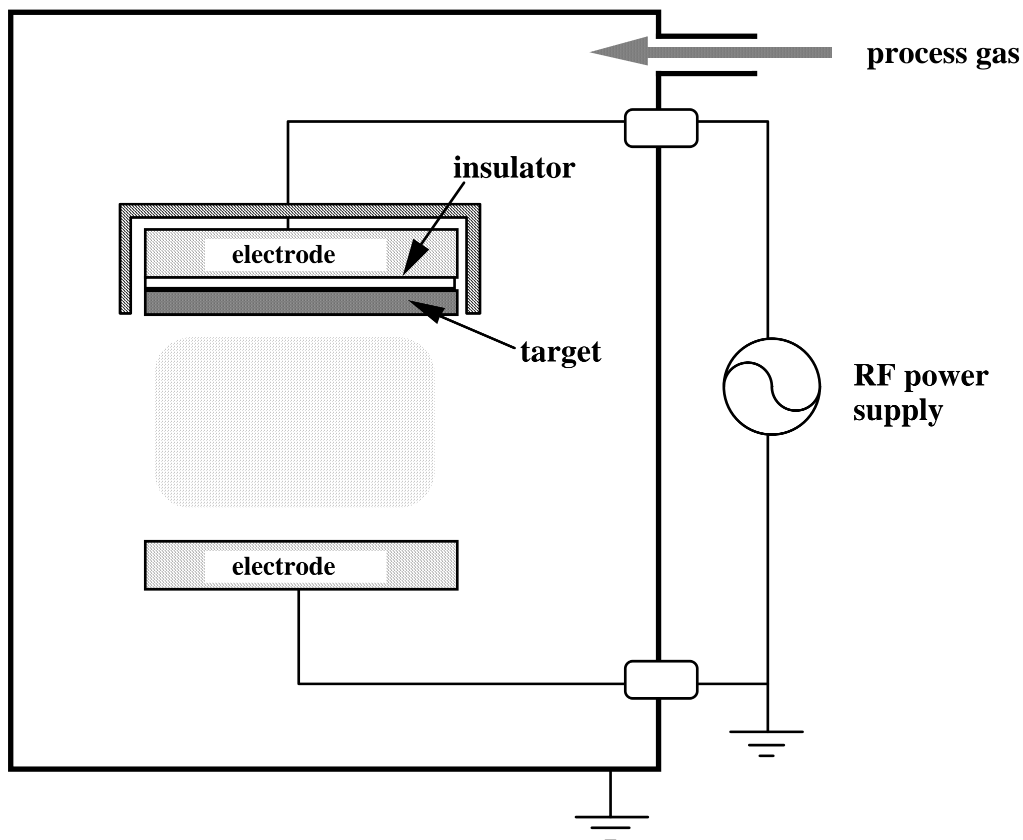

Figure 14.25 Components of an RF sputter deposition system

Materials which can be deposited by RF sputtering include metal oxides (SiO_2, Al_2O_3, ZnO, TiO_2, etc.), mixed oxides (Indium-tin oxide, which forms a transparent, electrically conductive film used to defrost wind shields), plastics, and glass, such as pyrex. In general, the composition of the deposited thin film is very close to that of the cathode, after an initial warm-up period.

As with DC sputtering, special power supplies are required for RF sputtering (see figure 14.26). The radio frequency AC power supply operates at a frequency of 13.56 kHz; many of these supplies output power in the 0.5 to 10 kW range.

Sample Problem:
14.16 Why are special power supplies needed for DC and RF sputtering applications?

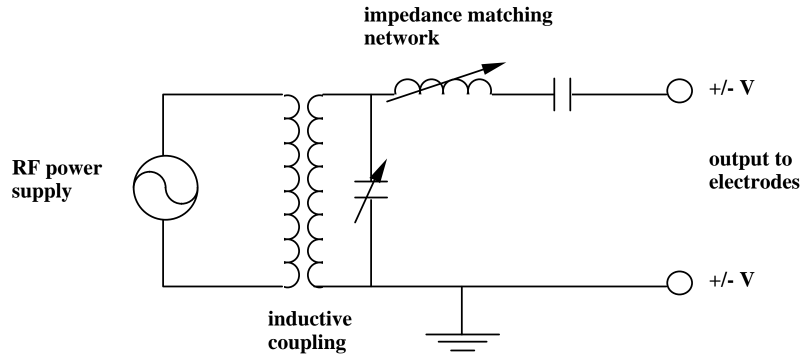

Figure 14.26 Components of an RF sputter deposition system.

Ion-Beam Sputtering

In this sputter deposition process, special ion sources, such as that presented in figure 14.27 are used to generate ions and accelerate these ions towards a sputtering target. The material sputtered from the target by impact of the energetic ions forms the coating on the substrate.

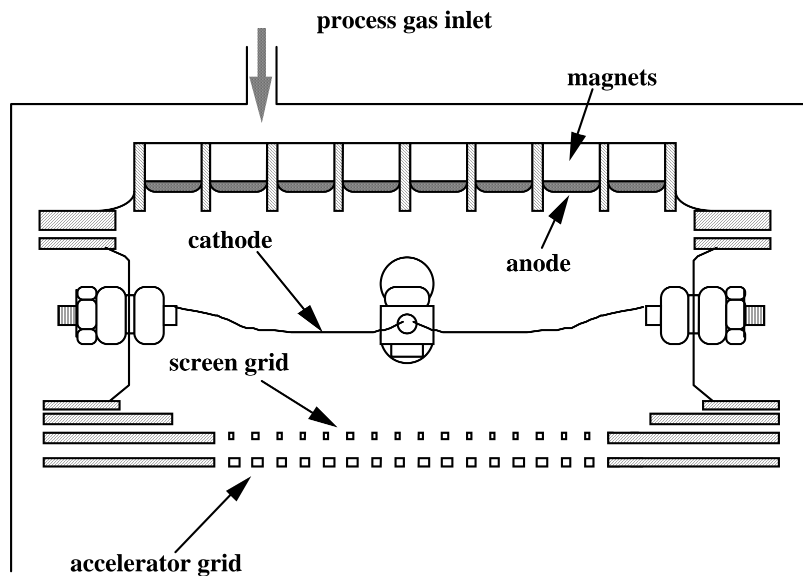

Figure 14.27 Kaufman ion source used in ion-beam sputtering.

In the Kaufman ion source, electrons emitted from the heated filament (cathode) are attracted to the anode, but the strong magnetic field prevents this. Gas molecules impacted by the oscillating electrons become ionized, and, being positively charged, are attracted to the negatively biased accelerator grid. By controlling the bias applied to the screen and accelerator grids, a certain amount of ion beam focusing may be accomplished.

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For a typical 10 cm diameter Kaufman ion source the characteristics of the ion beam are as follows:

Argon ion current: 1 to 2 mA/ cm^2
Argon ion energy: 500 to 2000 eV

Through control of the operating parameters, the ion current density and the ion energy of the beam may be independently varied. Ions generated from a Kaufman source may be used to clean surfaces or to deposit thin films as shown in figure 14.28.

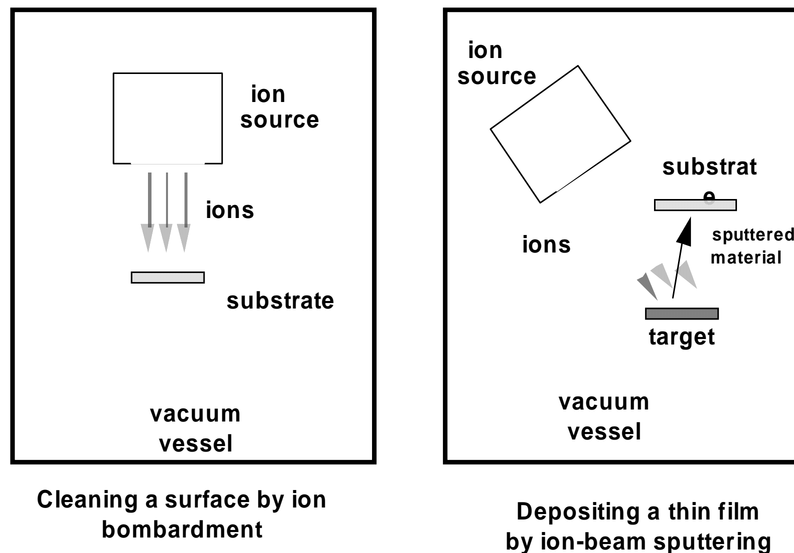

Figure 14.28. Use of ion an ion source to remove material from a surface (left) or to deposit a thin film (right).

Note that since the target used in the ion-beam sputter deposition technique is not a functional electrode, that it need not be electrically conductive. For most commercial applications the deposition rate one can achieve using ion-beam deposition is too low to be practical. Ion-beam sources are used in laboratories to produce high purity thin film coatings for research and development.

Sample Problem:
14.17 What are some of the advantages of RF sputter deposition versus DC sputter deposition?

Arc Deposition

High current, low voltage electric arcs, if constrained, may be used to create evaporant for thin film coatings. There are several techniques used to control the arc, including use of electrically insulating hearths, electrostatic, and magnetic constraints. Arc deposition has been demonstrated to produce thin films having high densities and excellent adherence to the substrate. Normally the arc process is conducted in a UHV environment, but partial pressures of reactive process gases, such as oxygen and nitrogen have been used to deposit metal oxide and nitride thin films. High deposition rates, in the range of from 50 to 500 Å/ second have been reported for the arc deposition process. One current drawback to this technique is the creation of macro-particles along with the atomic and ionic evaporant. These macro-particles have diameters ranging from 0.5 to 50 µm, and are co-deposited in the film, degrading the film's appearance and physical properties. Significant effort has been expended to eliminate the macro-particles without reducing the deposition rate. Once this is accomplished, arc deposition may provide a viable alternative to electroplating.

Laser Ablation

High energy density pulsed laser beams have been used to deposit thin films of a variety of elements, alloys and compounds. In this process, a laser source, external to the vacuum vessel generates a beam which is focused, passed through a viewport and impinges on a target within the vacuum vessel. Sufficient energy is generated to blast (ablate) material from the surface of the target. This ablated material consists of neutral atoms, ions, clusters of atoms and macro particles. The amount of material deposited per laser pulse is very consistent, allowing one to accurately deposit films of a specified thickness. The deposition rate is low compared to other techniques (electron beam evaporation and sputtering, for example). The range of commonly used operating parameters is given below:

laser power:	100 to 500 mJ
spot size at target	0.1 to 0.25 cm^2
power density at target:	400 to 5000 mJ/cm^2
pulse duration:	5 to 50 ns
wavelength:	250 to 308 nm
pulse frequency:	1 to 20 Hz

Laser ablation, as a deposition technique is currently limited to research and development laboratories due to the low deposition rate, the additional safety issues involved with the use of UV lasers and the expense of the equipment. Some of the thin films that have been deposited using laser ablation include super conducting thin films, ceramic coatings, and amorphous metallic layers.

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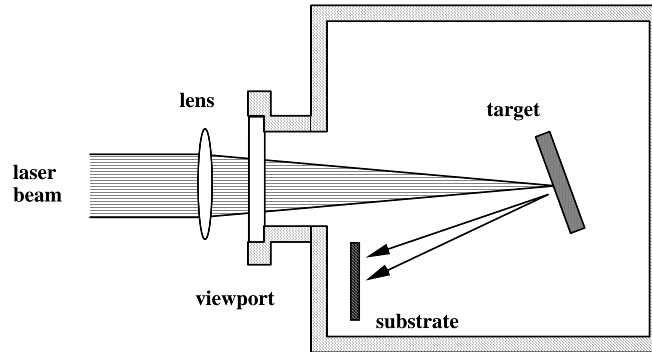

Figure 14.29. Deposition of a thin film by laser ablation.

Characterization of Thin Film Deposition Processes

There are a variety of means by which one can get information about the thin films created by physical vapor deposition processes. Of these techniques, some provide information while the film is being deposited (so-called "in-situ" techniques) while others give information after the deposition process is completed, and the coated part is removed from the vacuum vessel ("ex-situ" techniques). The in-situ techniques provide the means for control of a process during deposition. This real-time information may simply be collected, and used as quality control data, or can be used as input to the process parameters to optimize the process (closed loop operation).

In-Situ Characterization Techniques - Quartz Crystal Microbalance

In this technique a small quartz crystal is caused to oscillate by the application of an electric field. This crystal is placed inside a vacuum vessel, and during deposition of a film, the crystal gains mass due to the material condensing on it. The frequency of this oscillation is reduced as the mass of the film on the crystal increases. Through cross-calibration using a technique such profilometry, the quartz microbalance can be used to accurately monitor film thickness as the film is deposited, and to provide feedback information to control the process (close shutters, increase or decrease power to the deposition source).

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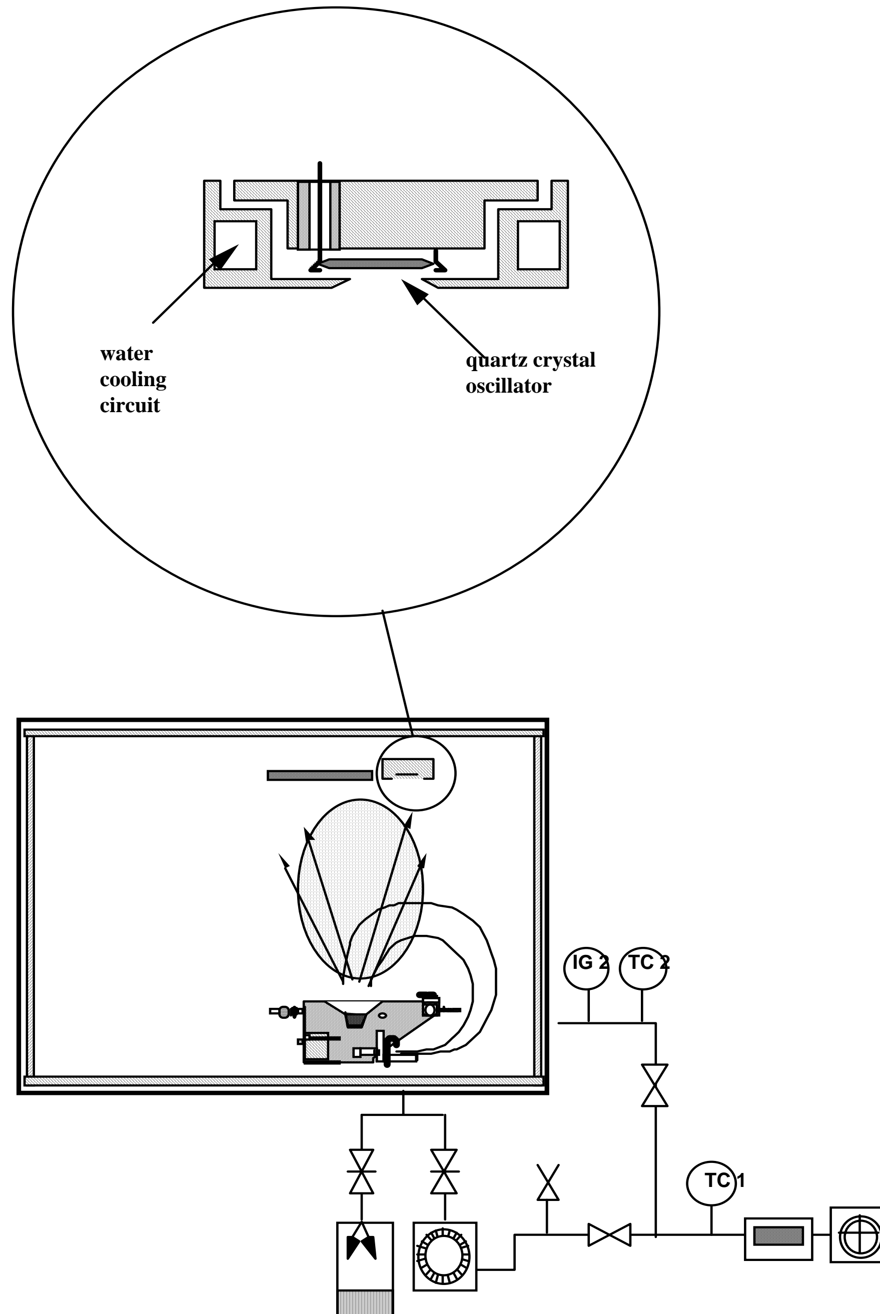

Figure 14.30 Quartz crystal microbalance in an electron-beam evaporation deposition system.

Optical monitoring.

Lenses and mirrors, which are to be coated with a thin film material to enhance their optical performance, are often monitored in the vacuum deposition chamber during the coating process. In-situ optical monitoring typically falls into two broad categories: transmission and reflection. If an optic has a performance specification (% transmission or reflection) at a specific wavelength, it is best to monitor the deposition process with that same wavelength light.

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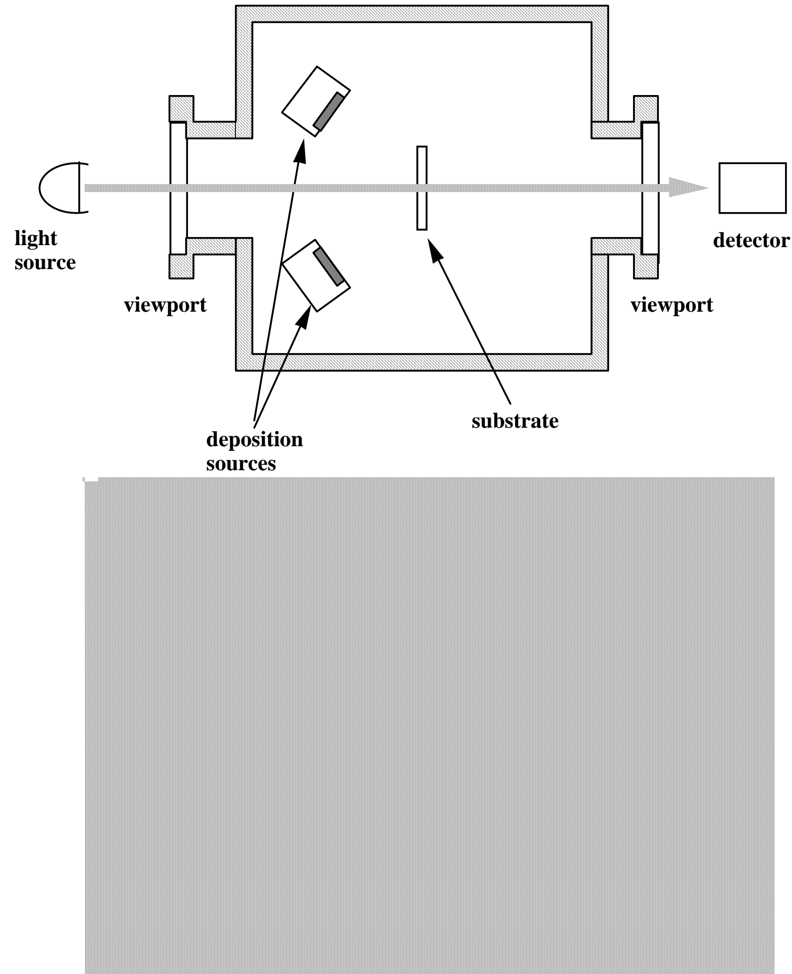

Figure 14.31 In-situ optical monitoring of the deposition process in transmission (top) and reflection (bottom).

Interpretation of in-situ reflectivity and transmission data can become complex. The effect of absorption and reflection of light by the glass viewport can affect the results. It is important to choose viewports having good transmission for the wavelength of light being used for the in-situ monitoring. Both reflection and transmission measurements can be used to monitor film thickness during the deposition. In the reflection method, the intensity of the reflected light will resemble a sine curve as the film grows. This is due to constructive interference between the light reflected from the surface of the film (I_f) and light reflected from the film/substrate interface (I_s).

$$\frac{\lambda}{2} = d\sin(\theta)$$

where:

$\lambda =$ the wavelength of the incident light [Å]

$d =$ the thickness of the thin film coating [Å]

$\theta =$ the angle between the incident beam and the normal to the surface [degrees]

Figure 14.32 Reflection of light from a substrate coated with a thin film.

Optical transmission data may also be used to monitor the thickness of a film during deposition, and can yield quantitative data if an independent thickness calibration is performed.

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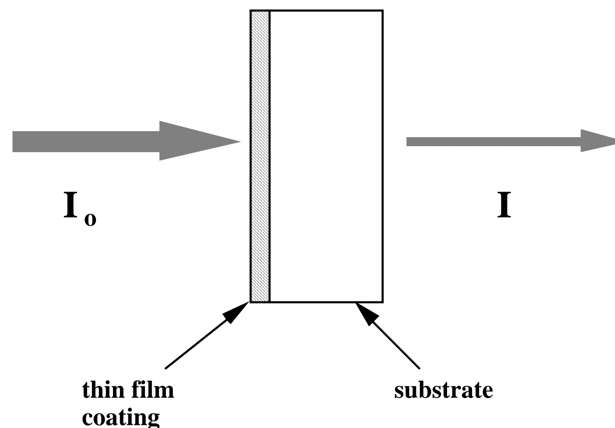

Figure 14.33 Transmission of light through a thin film on a substrate.

$$\frac{I}{I_0} = e^{-ax}$$

where:

$I_0 =$ intensity of the incident beam

$I =$ intensity of the transmitted beam

a = absorption coefficient of the material at the wavelength of interest

x = thickness of the material

The effect of absorption from the substrate must be taken into account in the thickness measurement by optical transmission. This can be accomplished by measuring the

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transmission of the uncoated substrate, and subtracting that value from the transmission measured during deposition.

Film Stress Measurement

Thin films condensing onto a substrate may induce mechanical stress in the film/substrate assembly due to the heat of condensation and the dissimilar coefficients of thermal expansion between the two materials. In-situ thin film stress measurement devices are commercially available, and generally are of the cantilever beam geometry shown in figure 14.34.

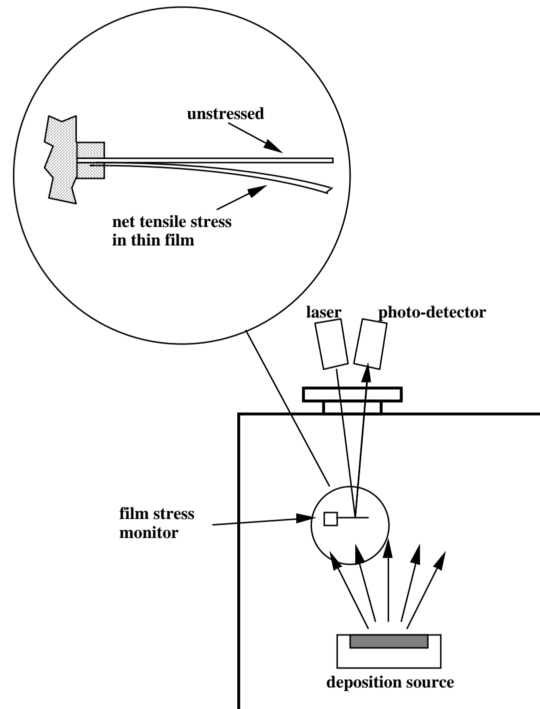

Figure 14.34 In-situ film stress monitor.

If a thin film is in a state of tension, the cantilever film stress monitor will be deflected as shown in the detail of figure 14.34. Alternatively, if the net mechanical stress in the film is compressive, the cantilever beam will bend in the opposite direction (upwards in the detail of figure 14.34).

Many of the thin film deposition techniques described in this unit create a visible plasma which may be analyzed using spectroscopic techniques to determine the chemical composition and state of excitation of species in the plasma.

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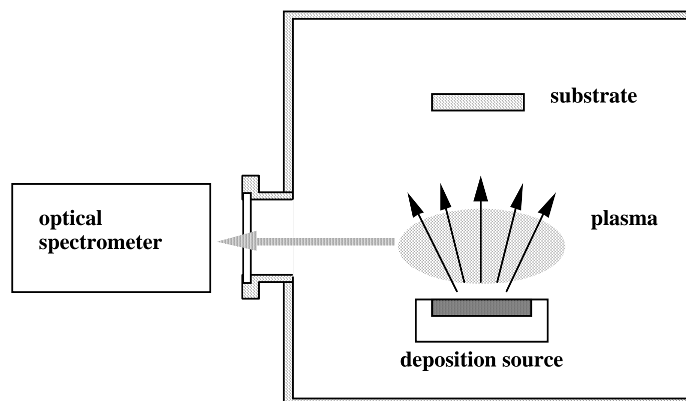

Figure 14.35 Optical spectrometer used to monitor light emissions from the plasma during the deposition of a thin film.

In sputtering processes, for example, the optical emission can be monitored to establish the purity of the sputtering gas, or to determine when the native oxide layer has been sputtered off a target. The intensity of peaks in the spectra observed using this technique are proportional to the concentration of species in the plasma. One can select a spectral line of interest (the line at 4189 Å for oxygen) to monitor intensity as a function of sputtering time. In-situ monitors of this type are commercially available, and can provide feedback to control the deposition process, based on the intensity of a particular spectral feature.

Ex-situ Thin Film Characterization Techniques

Care must be taken to prevent altering the thin film coating in the performance of ex-situ characterization techniques. In general, it is good practice to allow a coated substrate to cool to room temperature before venting the vacuum vessel. This minimizes the possibility of oxidation of the part. Thermal shock, due to rapid cooling caused by premature venting may cause films to tear or delaminate from the substrate, making them virtually useless for physical characterization purposes. Contamination of the coated substrate due to handling and storage should be kept to a minimum for best characterization results.

Contact Profilometry

In this ex-situ characterization technique, the sample is prepared by masking off a portion of a smooth substrate, such as floatglass, then depositing a thin film onto the sample. This creates coated and uncoated areas (a so-called "step slide") on the smooth substrate which can be used to determine film thickness. After the part is removed from the deposition system, a ruby or diamond tipped miniature stylus is scrolled across the sample.

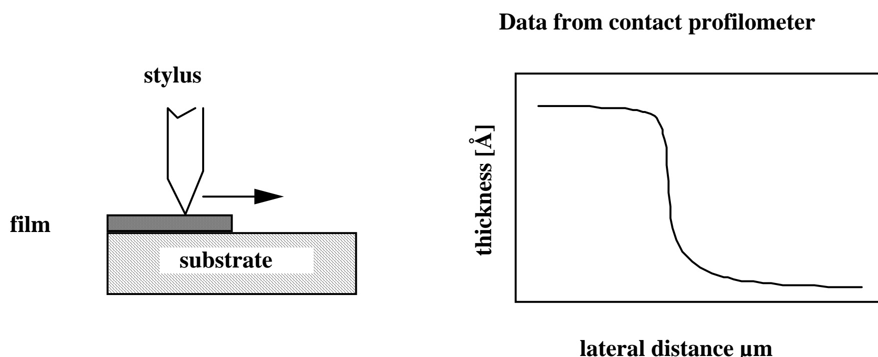

Figure 14.36 Stylus of contact profilometer on a "step slide" sample, and the resulting data.

Contact profilometry is frequently used to establish deposition rates, and as a means of calibration for in-situ rate monitoring techniques, such as the quartz crystal microbalance. Films having thicknesses of from 50 to 100,000Å have been accurately measured using contact profilometry.

Sheet Resistivity

Some thin films are deposited with the goal of attaining a specified electrical characteristic, such as resistivity. These films may be characterized using a commercial instrument called a four-point resistivity probe. Current is passed between each of the points of the probe through the thin film, and is measured. The data is output as sheet resistivity expressed in ohms per square centimeter (Ω/cm^2). Other electrical properties of interest in thin film coatings are the dielectric strength (the ability of a film to prevent high voltage from dissipating to ground through the film), and the critical current density (current density below which a material is super conducting at a given temperature).

Optical Transmission

Ex-situ optical transmission measurements often are more detailed and extensive than the optical monitoring described earlier for in-situ measurements. A single wavelength, or narrow band of wavelengths are often used, along with low resolution spectrometers for the in-situ optical measurements. Optics are often carefully inspected following deposition of a thin film coating to establish the transmission over a broad range of wavelengths.

Compositional analysis

There are a variety of techniques used to determine the chemical composition of thin film coatings following a deposition run. These techniques may be grouped into two broad categories: destructive and non-destructive. Wet chemical analyses, in which the film is dissolved, the solution being analyzed using techniques such as atomic emission spectroscopy are obviously harmful to the coating. If destructive analytical techniques are to be used, it is possible to perform them on "dummy parts" that were coated along with the part of interest. Non-destructive analytical techniques include x-ray techniques (x-ray florescence, x-ray diffraction), electron spectroscopy for chemical analysis (ESCA) and other surface science techniques. Most of these non-destructive analytical

methods are conducted under vacuum in relatively small chambers, which places size limitations on the parts to be analyzed.

Mechanical Testing

Frequently the mechanical property of a thin film coating which is of most interest is its adhesion to a substrate. Simple tests of adhesion include the "scotch tape test" in which a piece of scotch tape is pressed firmly to the coated side of a part, then peeled away. Other more sophisticated tests of adhesion include the Sebastian pull test and the ring shear test. In the Sebastian test, a metal stump is glued to the thin film deposited onto a substrate. The force required to pull the stump away from the substrate is recorded, and plotted as a graph of stress (force per unit area) versus strain (deformation).

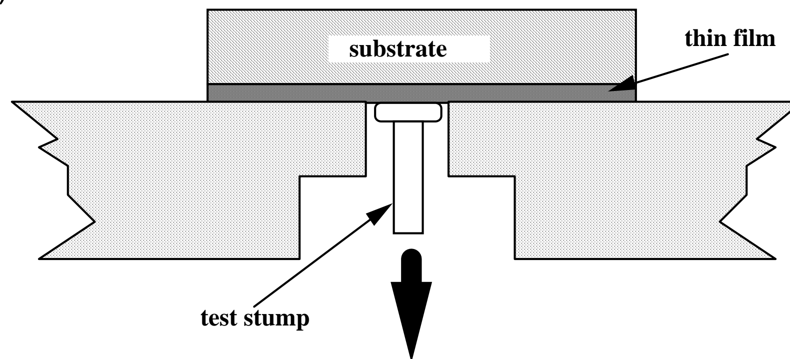

Figure 14.36 Sebastian pull test for adhesion of thin films.

In the ring shear test the circumference of a test cylinder is coated with a thin film. The area coated is subsequently built up by depositing additional material by electroplating. The "ring" is machined to prescribed dimensions, then tested, as shown in figure 14.37.

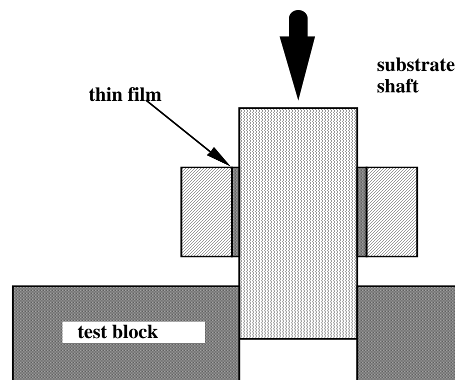

Figure 14.37 Ring shear test for thin film adhesion.

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The amount of force required to push the specimen through a hole in the test block is recorded. The location of failure is also observed. In some cases the thin film is so tenaciously adhered to the substrate that failure occurs in the bulk material.

Reference materials:

Deposition Technologies for Films and Coatings, Bunshah, Roitan, et. al.., Noyes Publications, Park Ridge, New Jersey. 1982, pp. 170-237.

Thin Film Processes, Vossen, John L., Kern, Werner Academic Press, Inc.Orlando, Florida. 1978, pp. 12-62,115-170.

Electron Beam Technology, Schiller, Sigfried, et. al. , Wiley Interscience. New York, New York. 1982,.

Laboratory Activities

Activity 14.1: Selection of physical vapor deposition techniques.

Equipment required: reference literature (deposition technology sales literature).

Procedure: for each of the following components to be coated with the specified thin film, describe in detail the deposition technique that would be appropriate. In the description include drawings of the hardware required, showing locations and relative sizes of components. Also include in your description the in-situ or ex-situ characterization techniques you would use to monitor the quality of the thin film deposits.

1) Deposit magnetic thin film coatings (iron and chromium oxides onto strips of plastic tape (12" wide by 100' long) for use in the manufacture of cassette recording media.

2) Deposit a reflective coating of aluminum onto a concave glass mirror 12" in diameter.

3) Deposit a gold corrosion barrier (10,000Å thick) on the inside surface of an aluminum tube 10" in diameter and 4' long.

4) Deposit an electrically insulating thin film onto a circuit board.

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Experiment 14.1 Deposition of a thin film by electrical resistance heated thermal evaporation.

Equipment required: small vacuum vessel or bell jar vacuum system with Feed throughs and internal hardware for deposition of thin films by resistance heated thermal evaporation (see figure 14.3). A low voltage power supply (5 to 40 VDC, 10 to 50 A) with power leads compatible with power Feed throughs on the vacuum vessel.

Procedure: Assemble the equipment as shown in figure 14.3. Make certain the electrical connections are secure, and that the vessel and power supply are both safely grounded. Prior to any experimental work, have the laboratory instructor inspect the vacuum vessel and power supply. Before pump down mount at least one substrate (glass microscope slides work well for this) on the opposite side of the shutter from the deposition source. Load the source with an appropriate amount of charge material (aluminum). Evacuate the vessel and conduct the thermal evaporation. Allow the filament to cool for 30 minutes after the power is turned off prior to venting the vessel. Upon completion of experimental work shut down the vacuum system safely and write a lab report describing your experiment. Include any characterization of the thin film you perform.

Experiment 14.2: Deposition of a thin film by DC diode sputtering.

Equipment required: small vacuum vessel or bell jar vacuum system with electrical power feed throughs; a diode sputtering power supply with appropriate power leads; a copper cathode and an aluminum cathode; a bottle of compressed Argon with regulator; a leak valve.

Procedure: Assemble the equipment as shown in figure 14.38. Make certain the electrical connections are secure, and that the vessel and power supply are both safely grounded. Prior to any experimental work, have the laboratory instructor inspect the vessel for safety. Evacuate the vessel to a pressure of less than 5×10^{-5}Torr. Before proceeding, turn off the ion gauge. The next task is to throttle the high vacuum pump to control the flow of process gas through the system. If an iris valve is installed between the inlet of the high vacuum pump and the vessel, close it approximately 95%. In the absence of an iris valve, close the gate valve almost completely. Use the leak valve to inject process gas into the vessel. The goal here is to achieve a steady pressure of between 5 to 50 mTorr of Argon.

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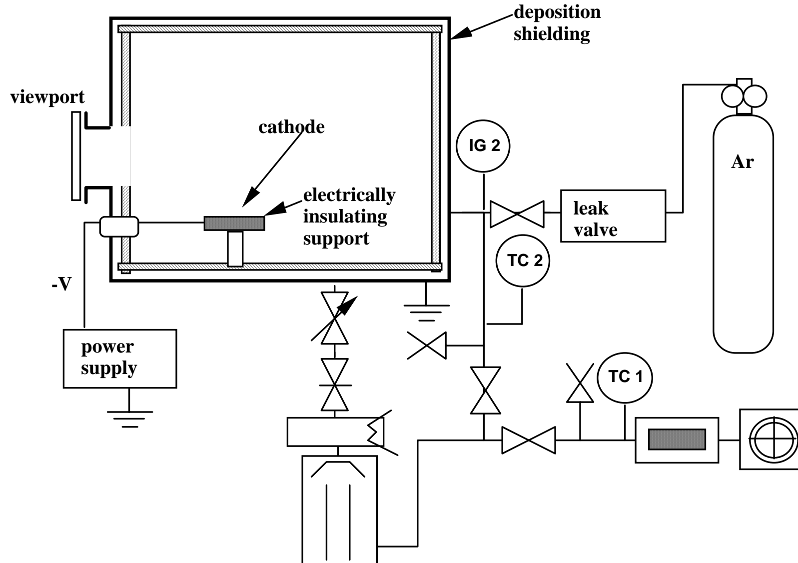

Figure 14.38 Experimental setup for DC diode sputtering.

Once a stable process gas pressure is established, turn on the sputter source power supply and slowly increase the negative bias applied to the cathode. A glow discharge should appear at an applied bias of from 500 to 800 V. Note the color and distribution of the plasma. Allow the cathode to sputter for ten minutes. Turn the sputter source power supply down to zero volts and shut it off. Allow the system to cool for 30 minutes. Vent the vessel and inspect the cathode and substrate. Exchange the copper cathode for the aluminum cathode and repeat the sputtering experiment. Upon completion of all experimental work, shut the vacuum system down safely, venting all pumps. Write a report of your observations.

Discussion questions:

1. What causes the plasma to be a different color when different cathodes are sputtered?
2. Why does the cathode heat up during sputtering?
3. What useful applications can you imagine for this process?

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