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PVD Processes: Heating & Temperature Measurement

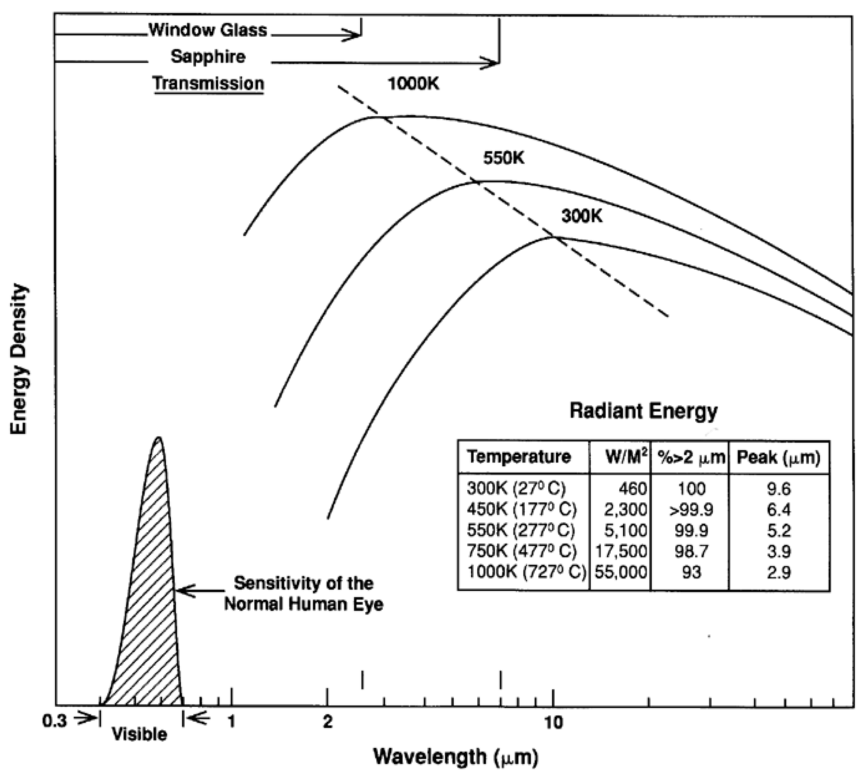
The substrate temperature during film deposition is often an important processing parameter. Raising the substrate temperature and monitoring the temperature are important process considerations.

Heat is transferred by conduction, radiation and convection. In a good vacuum, the gaseous particle density is too low for convection to be an important process. Conduction of heat to or from a surface can be done by placing it in good thermal contact with a hot (to heat) or cold (to cool) surface. Mechanical contacting, such as clamping, often does not provide good and reproducible thermal contact, so a compliant conductive material should be used. A soft, vacuum-compatible material, such as indium or silver, can be used, or something obtained by soldering (<450 °C) or brazing (>450 °C) the surfaces together. For contact heating, the heater surface can be heated by an embedded electric heater, by circulating hot oil, by electron bombardment or by radio frequency (rf) heating.

If a moving fixture is used to hold the samples, contact heating may be difficult, so radiant heating can be used. Radiant heating is usually done by resistively heated tungsten-quartz radiant heaters, where a hot tungsten filament produces both visible and infrared (>0.7 micron wavelength) radiation. A quartz (fused silica) envelope around the filament allows most of the radiant energy to escape and can withstand the high temperatures generated. Care must be taken when handling the quartz heaters, because salt from fingerprints can diffuse into the quartz at high temperatures, causing devitrification of the fused silica. Resistively heated tungsten-halogen lamps and xenon arc and flashlamps are also used for heating. Some heaters have reflectors to direct the radiant energy, but generally, the entire inside of the vacuum chamber is heated. Radiant heating does not work well if the surface to be heated is a good infrared reflector, such as gold.

Electron heating can be accomplished by having the holder (or substrate) at a positive potential with respect to an electron-emitting surface, such as a hot tungsten, a thoriated-tungsten, or LaB_6 surface. Electron heating has the advantage that it can be confined to the area of interest. Rf heating uses a low-frequency (*e.g.*, 400 kHz) electromagnetic radiation that is adsorbed by an electrically conductive material (susceptor) or by the substrate directly. Carbon is a common susceptor material in CVD technology. Heating can be accomplished by ion bombardment and is often combined with sputter cleaning. This has the disadvantage that the two operations are not independently controllable.

Cooling a surface in a vacuum is accomplished by having the substrate



Temperature	W/M²	%>2 μm	Peak (μm)
300K (27° C)	460	100	9.6
450K (177° C)	2,300	>99.9	6.4
550K (277° C)	5,100	99.9	5.2
750K (477° C)	17,500	98.7	3.9
1000K (727° C)	55,000	93	2.9

Fig. 1—Radiation from a black body surface at various temperatures, along with the optical sensitivity of the human eye.

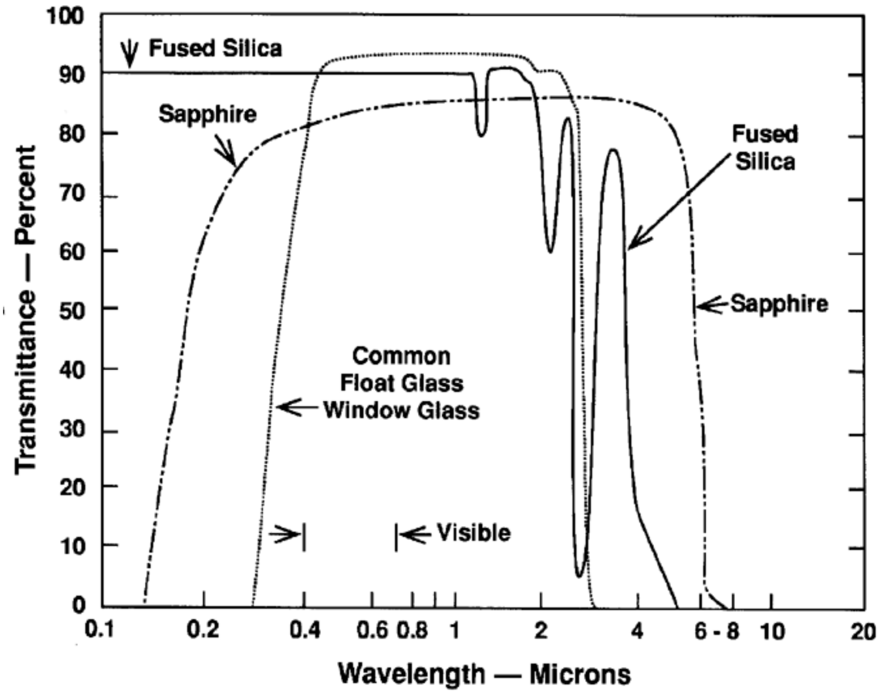


Fig. 2—Transmission of various window materials over the visible and near-infrared spectral regions.

in good thermal contact with a cold surface. The cold surface is generally cooled by a cold liquid, such as chilled water or oil, water/ethylene glycol mixture (25 °C), dry ice/ acetone (-78 °C), refrigerants (~ -150 °C) or liquid nitrogen (-196 °C). Helium, because of its low mass, is sometimes used for convective cooling at sub-atmospheric pressures.

A material radiates energy (E) at a rate given by $E=\sigma AT^4$, where σ is the emissivity, A is the surface area, and T is the temperature in degrees Kelvin. If the emissivity is equal to one, the material is called a "black body." Radiation from a cavity in a surface approaches the radiation spectrum of a black body. Figure 1 shows the black body radiation spectrum at several temperatures. The peak radiation intensity in microns from a hot surface is about equal to 3000 divided by the temperature in degrees Kelvin. The area under the curves represents the total energy radiated (watts/m^2).

The temperature of a surface is close to the same temperature as the bulk of the material, unless the heating energy is being introduced into the surface and the thermal conductivity of the material is low. When the thermal conductivity is low, there can be an appreciable temperature difference between the surface and the interior of the material, or from one part of the material to another.

The temperature of the bulk of the material can be measured using embedded thermocouples. The surface temperature can be measured by thermocouples attached to the surface, or even by thermocouples deposited on the surface. Thermocouples are made by joining dissimilar materials to form a thermocouple junction. The temperature of this junction generates a voltage (about one millivolt per degree), which is measured with relation to a reference junction at 0 °C (or some other temperature) and related to the calibration for those particular junction materials. Examples of thermocouple materials are: Platinum/10% rhodium; platinum : platinum/13% rhodium; iron : constantan; chromel : P-alumel; and copper : constantan. The thermocouple junction may be bare or may be contained in a protective sheath.

Thermocouples can present mounting problems. In the cases of moving fixtures, it is difficult to transmit the millivolt readings through sliding contacts. The surface temperature can be determined in a non-contacting manner by using optical pyrometry. Optical pyrometry is usually performed by measuring the total radiant energy intensity over a portion of the emittance spectrum of the hot surface. Because the energy intensity is proportional to T^4, the measurement can be quite sensitive to temperature. In some cases—particularly when observing surfaces through plasmas—a specific wavelength, such as 3.8 microns, may be used to avoid non-thermal background radiation.

The surface to be measured should fill the field-of-view of the pyrometer, so focused beams are used when monitoring temperatures at a distance. The optical pyrometer calibration can be adjusted to account for a non-unity, emissivity. Often, however, the instrument is used in a comparative mode, and accurate temperature is not important. Calibration of the optical pyrometer can be set by referring the reading to: (1) a surface at a known temperature, (2) an area of the surface that has been coated with a high-temperature, dull black paint with an emissivity near one or for high temperatures, (3) the radiation from a hole in the surface with a depth-towidth aspect ratio greater than six. Optical pyrometers can be set up so as to read only the maximum temperature it sees. This is useful when measuring the temperature of surfaces on a moving fixture that passes in front of the pyrometer.

Effects that affect the temperature reading, or that can cause drift with time, are an unknown or changing emissivity of the hot surface, or adsorption of the radiation before it enters the sensor. In many cases, the radiation is carried to the sensor by optical fibers or light pipes, or transmitted through windows. If the window or fiber bundle becomes coated with material that adsorbs or reflects radiation, the temperature readings will be in error. When depositing a coating on a surface, the emissivity often changes, indicating an abrupt (but false) change of temperature. If this occurs, the pyrometer should be calibrated periodically by referring to a surface that has a known temperature, such as an area on a wall or fixture where the temperature is monitored by a thermocouple.

It is important to have windows and fiber optics materials that will transmit the radiation with little adsorption. Figure 2 shows the transmission curves for several window materials. A common, rather inexpensive material is sapphire (Al_2O_3), which has good visual transmittance (from 0.2 micron) and good infrared radiation transmittance (to 6 microns). Common "float glass" window glass is poor, because it adsorbs radiant energy above 2 microns. Fused silica (fused quartz) is also not very good, even though it transmits to about 4 microns. Other materials with good infrared transmission are: Calcium fluoride (CaF_2), (visible to 8 microns), and silicon (>1.1 microns). A rather inexpensive source of sapphire windows is sapphire watch crystals, which are used for their scratch resistance.

Optical pyrometers are relatively expensive. A less expensive, non-contacting instrument for reading temperatures is the infrared thermometer. In this instrument, the infrared radiation is focused on a thermocouple, and the output of the thermocouple is measured. There is no power source needed.

The maximum temperature that a surface has seen can be measured in a passive manner by observing melting of sharp corners of a material with a known melting point, a phase change, a color change, or the subsequent outgassing behavior of the surface. Such passive temperature monitoring techniques are not used very often. For high temperatures, the color of the surface can be used to get an approximate temperature. At approximately 500 °C, a hot surface can be seen as a dull red color against a black background, and grows brighter as the temperature increases. The color becomes a yellowish-red at about 1000 °C and "white hot" at about 1400 °C. P&SF