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A New Method for the Determination of Thin Film Porosity

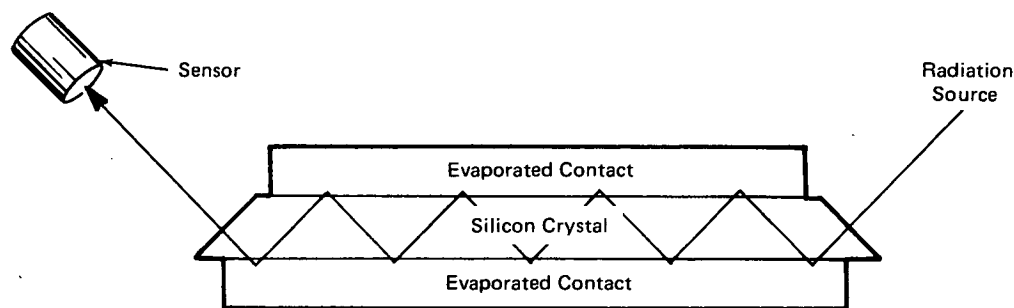


Figure 1. Multiple Internal Reflection Effect

The problem:

The porosity of thin films is usually determined with mercury porosimeters, by the KET method, or from gas flow measurements. These methods, however, are not adequate for the determination of small (less than 200 Å) pores.

The solution:

Internal reflection spectroscopy may be used to determine the presence of water in thin film pores. The presence of water in such pores is a function of the relative humidity and the pore size. Thus, by controlling the humidity, one can determine the pore size.

How it's done:

Infrared radiation entering a single crystal of silicon at a critical angle is internally reflected at the surface of the crystal. As the radiation travels through the crystal, reflected from surface to surface, it actually extends several wavelengths outside the crystal at every reflection. An appropriate substance placed on the surface of the crystal will absorb some of the radiation. Figure 1 shows how material adjacent to a crystal may absorb internally reflected infrared radiation along its path through the crystal.

To measure the porosity of a thin film, the film is evaporated onto both sides of a single crystal of silicon. The surrounding humidity is raised to a point at which water condenses in the pores. At this humidity the O-H bond stretch (occurs at a frequency of 3400 cm^{-1}) will absorb infrared radiation.

The size of the pores is determined by progressively increasing the humidity in the atmosphere surrounding the sample. The relative humidity at which water condenses in the pores will be indicated by an absorption peak at 3400 cm^{-1} . The relative humidity may be related to the pore size through the Kelvin equation:

$$RT \ln \frac{P}{P_0} = \frac{2 \alpha V}{r}$$

where R = gas constant (in ergs/deg mole)
 T = Kelvin temperature
 P = vapor pressure of water in pore
 P_0 = vapor pressure of water on flat surface
 (P/P_0) = relative humidity at which condensation will occur in a pore of radius r
 α = surface tension of the liquid (dynes/cm)
 r = pore radius (cm)
 V = molar volume (cc/mole)

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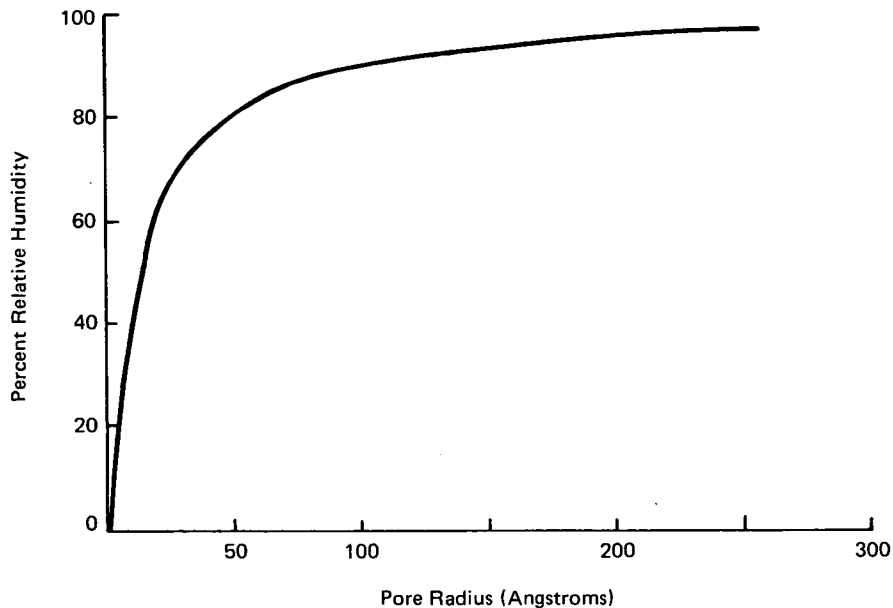


Figure 2. Pore Size Required for Capillary Condensation

Figure 2 shows the results of an experiment in which the porosity of a three micron layer of silver was determined. The first water absorption peak at a relative humidity of 85% corresponds to a minimum pore size of about 65 Å.

At a given humidity, the water content of a sample increases very little. Thus, an increase in absorbance with an increase in humidity will be due, almost totally, to the presence of additional larger pores, and gives an indication of the pore size distribution in the sample.

Fluids with a surface tension different from that of water can be used to detect pores, the radii of which range over a different set of sizes. The fluid need only have a functional group that absorbs in the infrared region.

Note:

Requests for further information may be directed to:
 Technology Utilization Officer
 NASA Headquarters
 Code KT
 Washington, D. C. 20546
 Reference: B73-10286

Patent status:

NASA has decided not to apply for a patent.

Source: Theodore R. Beck, Charles J. Bishop, and
 W. F. Springgate of
 The Boeing Company
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