



Fig. 6. Exploded assembly showing stainless ring, platinum foil, and sample geometry.

vapor through this extruded material is apparently so slow that it does not interfere with our experiments.

The sample comes out as a lenticular wafer .004"-.015" thick, weighing 1-10 milligrams. It is usually hard and coherent. Under the microscope its crystalline nature is usually evident. Grain size is usually 1-10 microns, so that some optical properties can be determined microscopically. Moderately good X-ray patterns can usually be obtained on a Norelco recording X-ray spectrometer. These X-ray determinations provide the easiest and most unequivocal means of identifying the crystalline phases present in the sample. It has been found that grinding the sample in a mortar before X-raying results in general in a better pattern. This is presumably due to the development of preferred orientation due to the shear stress and/or strain present in the sample under pressure.

PROCEDURE

Successful use of this apparatus requires appropriate quenching to preserve the sample in the crystal phase which it had attained at stabilized temperature and pressure. Quenching is normally done by swinging the furnace open and directing an air blast directly at the pistons. A cooling curve is shown in figure 7. Even this rapid rate of cooling is not sufficient to prevent recrystallization of some samples. Whenever the presence of water vapor or other volatiles is not necessary to preserve the crystal phase present during the run, the pistons and holders can be removed rapidly and water quenched. The sample in the stainless ring and platinum foil capsule usually separates readily from the piston, and quenching is essentially instantaneous on contact with water. The time to get the sample from test conditions into the water is less than 10 seconds.