4.4 SPECIMEN PREPARATION FOR KNOOP HARDNESS TESTS. The procedures which follow have been found most useful for preparing, mounting, grinding and polishing metal pecimens for Knoop hardness testing where indenter loads of 50 or 100 grams are used.

4.4.1 Sample Preparation. Small diameter wire and parts. It is most desirable to obtain a longitudinal section for testing and, since diameters may range from .001" to .049", it has been found that the most successful method of preparation is that shown in Fig. 4-6, "A". The wire is first bent in opposite 90° angles at each end, taking care to make the bends in the same plane (to insure flatness), and then bent again at 90°. These last bends serve a retention pins.

In all formed parts it is customary to check hardness in the "contact area." This is best accomplished by mounting in the manner shown in Fig. 4-6, "B", as a longitudinal section of the "contact area" is exposed for hardness measurements and/or microscopic structural study.

Large diameter wire and parts. Diameters of .05" and over can be prepared either as longitudinal or transverse sections. For most alloys there is a very small difference in the hardness values obtained on either section, but for reference purposes the section should be specified (Fig. 4-6, "C", shows a transverse section).

Fig. 4-6. Specimens prepared for mounting.

A — small diameter wire
B — small diameter wire parts
C — large diameter wire
D — strip stock
E — part made from strip
F — headed part

Strip stock and parts. Prepared as shown in Fig. 4-6, "D", exposing a longitudinal section of the thickness. Strip in the range of .002" thick may buckle under the pressure required to make the mount. The bends help to give rigidity, thus minimizing the tendency to buckle. Keeping the width (depth) of sample to a minimum will also help in this respect. However, if this condition should occur, it may be possible to obtain a true section by grinding sufficiently deep to get beyond the buckled area. Strip parts are prepared in a manner similar to strip (exposing a long section of the thickness in the· "contact area"). See Fig. 4-6, "E".

Headed parts. Headed parts with a radius on the head (Fig. 4-6, "**F**") should have a "flat" filed on the radius to minimize the tendency of "floating" in the mounting medium. If a "flat" were not filed, the mounting plastic would frequently flow under the part and lift or float it from the face of the mold block.

4.4.2 Mounting Procedure. Prepared parts are placed on the face of a mold block and inspected for flatness. If the entire area of prepared surface does not contact the face of the mold block, parts should be filed or bent until it does. A sketch may be drawn at this stage identifying particular lots or parts by shape or orientation. Finely sifted Bakelite powder is now dusted over the parts, as it adapts more readily and insures that all parts are firmly imbedded in the mount. (It also helps to hold small or delicately balanced parts when remaining powder is poured over them.) The mold sleeve is put in place after having covered part with the fine powder and remaining charge of Bakelite is added. The ram is inserted and the mold is ready for curing, which is a matter of pressure and temperature, according to the molding plastic being used. For Buehler' #1380, a pressure of about 1000 psi is maintained until the temperature reaches 80°**C** (176°**F**), then pressure is raised to about 4000 psi until the temperature reaches 130°**C** (266°**F**), at which the heat source is removed . Pressure is maintained until the mold cools to 60°**C** (140°**F**) and the mount is removed. It is preferable that the ram have a convex end. This gives a concave bottom on the mount, which makes for easier and faster levelling of the mount. It should also be noted that a porous or granular mount results if pressure, temperature cycle is not correct.

4.4.3 Grinding and Polishing. Grinding is best accomplished if the abrasive papers are placed on a smooth, flat surface. If a grinding table is not available, plate glass will serve as well. Either dry emery paper or waterproof silicon carbide paper, wet with tap water, are excellent grinding media. If emery is used, it is necessary to follow a sequence of # 1, #0, #00, #000, and #0000 grits. After each grinding step, the sample must be rotated 90° and all scratches from the previous paper removed before proceeding to a smaller grit paper.

If silicon carbide paper is used, most of the grinding can be accomplished with 600 grade paper. The faster cutting of this abrasive, coupled with the fineness of the grit, permits the use of one grinding step on a carefully prepared mount prior to polishing. Caution must be exercised if coarser silicon carbide paper are used, as it is possible to grind through a small diameter wire in a few passes on the paper.

After the final grinding step, the base of the mount must be ground parallel with the surface to be indented, if the hardness tester is not equipped with a paralleling device. The surfaces should be parallel within ± .0005".

Polishing should be performed in accordance with recommended metallographic procedure . Small diameter samples and thin gage strip stock of

soft materials have a tendency toward edge rounding. Therefore, polishing should be held to the minimum necessary to produce a satisfactory surface finish.

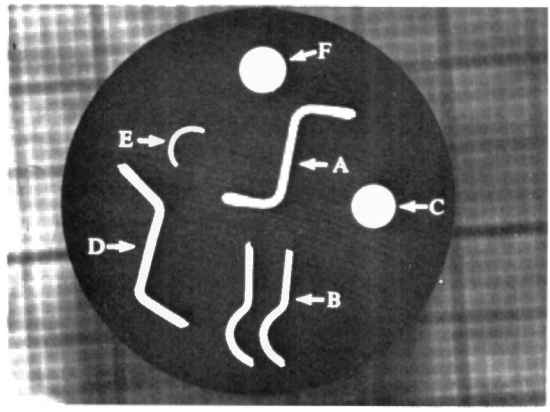

Fig. 4-7. Ground mount of parts shown in Fig. 4–6.

One of the techniques that has proven most successful for noble metal alloys is the use of Microgrit® **WCA** lapping compounds in water suspension on a motor driven wheel covered by a micro-polishing cloth. The solution should contain a soap or wetting agent for lubrication. This poltshing procedure is done in two steps. Microgrit **WCA** #12 is used ·on a medium speed (approximately 600 rpm) wheel for one to two minutes. This is followed by polishing with Microgrit **WCA** #9 on a slow speed (approximately 600 rpm) wheel for one to two minutes. This is followed by polishing with Microgrit **WCA** #9 on a slow speed (approximately 250 rpm) wheel. Time for finishing will vary, depending on the hardness of the material. One to two minutes are sufficient for the harder noble metal alloys (300 to 400 **KHN**), but two to five minutes may be necessary for alloys between 100 to 250 **KHN**.

4.4.4 Indentation. Whenever possible, indentations should be made in the "contact area" and away from any bends made in preparation of sample for mounting. On wire and strip it has been found best to make impressions parallel to the longitudinal axis. Impressions made in this manner are less likely to be affected by any rounding of sample which may have occurred during grinding and polishing. To avoid effects of cold working introduced by indentation, the space between impressions should be at least three times the width of the indentations.

This space between indentations is great enough to prevent the cold working surrounding an impression from having any effect on a subsequent indentation.

4.4.5 Materials List. The following materials have been found satisfactory

Molding Compound: Buehlers #1380 A. B. Bakelite (Black)

Emery Paper: Buehler's #1425 A. B. Emery Polishing Paper

Silicon Carbide Paper: Wetordry Tri-M-ite #600,
Minnesota Mining &Mfg. Co.

Polishing Compounds: Microgrit WCA #12 and WCA #9,
MicroAbrasives Corp., Westfield, Mass.