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**SPECIMEN MOUNTING METHODS
FOR METALLOGRAPHY**

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SPECIMEN PREPARATION TECHNIQUES FOR OPTICAL AND ELECTRON METALLOGRAPHY

Program

23-24 September 1973

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D. V. Miley and A. Calabra, Dow Chemical Company, Golden,
Colorado.

ABRASIVE CUTTING IN METALLOGRAPHY—J. Nelson and R.
Westrich, Buehler, Ltd., Evanston, Illinois.

USE OF WIRE SAWS FOR METALLOGRAPHIC SECTIONING—H.
McLaughlin, Laser Technology Inc., No. Hollywood, California.

ELECTRICAL DISCHARGE MACHINING—Joseph Barrett,
IMANCO, Monsey, New York.

LAPPING IN METALLOGRAPHY—N. J. Gendron, General Elec-
tric Company, Schenectady, New York.

DIAMOND ABRASIVES FOR METALLOGRAPHIC POLISHING—
J. L. McCall and R. D. Buchheit, Battelle-Columbus Laboratories,
Columbus, Ohio.

ALUMINA ABRASIVES FOR METALLOGRAPHIC POLISHING—F.
Charvat, Union Carbide Corporation, San Diego, California.

**A NEW UNIVERSAL POLISHING TECHNIQUE FOR METALLO-
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Company, Palo Alto, California.

**TECHNIQUES OF SPECIMEN PREPARATION FOR METALLOGRAPHY
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—K. Roth and D. M. R. Taplin, University of Waterloo, Water-
loo, Ontario, Canada.

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Ridge, Tennessee.

**PREPARATION OF SAMPLES FOR ELECTRON MICROSCOPY BY
ION MICROMILLING**—R. Kossowsky and G. P. Sabol, Westing-
house Research Laboratories, Pittsburgh, Pennsylvania.

**SPECIMEN PREPARATION TECHNIQUES FOR SCANNING ELEC-
TRON MICROSCOPY**—N. Hodgkins, Micrographics, Newport
Beach, California.

EPITAXIAL ETCHING—R. J. Gray, Oak Ridge National Labora-
tory, Oak Ridge, Tennessee.

FERROFLUID ETCHING—R. J. Gray, Oak Ridge National Labora-
tory, Oak Ridge, Tennessee.

CATHODIC ETCHING—F. Cain, Burrell Corporation, Pittsburgh,
Pennsylvania.

**PREPARATION OF THIN SECTIONS OF LUNAR ROCKS, METEOR-
ITES, FOSSILS AND VARIOUS BRITTLE MATERIALS FOR OPTICAL
MICROSCOPY**—R. Beauchamp, Battelle-Pacific Northwest Lab-
oratories, Richland, Washington.

CERAMOGRAPHIC SPECIMEN PREPARATION TECHNIQUES —
Speaker to be announced.

**SPECIMEN PREPARATION TECHNIQUES FOR SEMICONDUCTOR
MATERIALS**—D. Medellin, Rockwell International, Anaheim,
California.

PLUTONIUM METALLOGRAPHY—E. Cramer, Los Alamos Scien-
tific Laboratory, Los Alamos, New Mexico.

METALLOGRAPHIC TECHNIQUES FOR COMPOSITE MATERIALS—
T. J. Bertone, The Aerospace Corporation, Los Angeles, Cal.

**PREPARATION OF THIN FILMS FOR ELECTRON MICROSCOPY
USING ELECTROPLATING METHODS**—C. Hays, Texas A & M,
College Station, Texas.

**MICROSTRUCTURAL CHARACTERIZATION OF RADIOACTIVE MA-
TERIALS**—H. Rosenbaum, General Electric Company, Vallejo
Nuclear Center, Pleasanton, California.

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INTRODUCTION

Metallography is one of the oldest and also one of the most modern branches of metallurgy. It has been variously described as an art and as a science, and undoubtedly it has been considered by some to be a "black art". Its recognition as a distinct area of study goes back at least as far as 1916 when the ASTM Committee on Metallography was first established (1).

No matter how sophisticated our new techniques become, though, there will always be a need to perform optical metallography, either solely or in conjunction with other techniques. And of initial interest to the optical metallographer will be selection of an appropriate method of mounting the sample so it may be properly prepared. It is the purpose of this paper to briefly review the development of mounting methods, and to discuss in some detail several of the more common methods being used. Characteristic advantages and disadvantages will be pointed out. Retention of sample edges for examination is essential in many investigations, and a separate section will be devoted to the many techniques devised to achieve this goal.

HISTORY OF MOUNTING METHODS

Before beginning our discussion of the more modern mounting techniques, it might be interesting to take a brief look back and see how the art of specimen mounting has progressed.

As might be expected, the very earliest "mounts" were simply hand-held specimens, normally for examination of fracture surfaces. The use of the microscope to study fracture surfaces was reported before 1800 (2). In the years immediately following, there were some studies of ground-and-etched metals, and quite a bit of interest in the microscopic examination of meteorites (2).

Henry Clifton Sorby, considered by most to be the father of modern metallography, was a wealthy Englishman who conducted studies in many areas of the natural sciences (3). One of his areas of interest was petrography, and he is credited with preparing the first petrographic specimens in 1849 (4). He used Canada balsam to mount 1-in. square specimens on glass, and it is interesting to note that this is still one of the principal methods of petrographic preparation almost 125 years later.

By the early 1860's, Sorby's interests had expanded to include the examination of metallic structures. It seems only natural that he would adapt his earlier petrographic methods for use with metals. He cut his samples with a saw, then ground or filed them to a convenient thickness. They were then affixed to a piece of glass with Canada balsam, and the exposed surface was prepared by polishing and etching. A thin cover glass was then applied over the prepared surface, again using Canada balsam (3). Sorby's historic first publication of a metal microstructure appeared in 1864.

At about the same time in Germany, Adolph Martens was also developing similar techniques. He was apparently unaware of Sorby's 1864 publication, and his first papers appeared in 1878 (2). According to Smith (3), Martens was not strong on metallurgical theory, but he was a devoted technical metallographer. He also mounted his specimens on glass for preparation.

Similar work was also going on in France during the late 1800's, where Osmond used Canada balsam to mount thin metal slices to glass for preparation (3).

The turn of the century saw the beginning of the plastic age with the production of the first synthetic plastic, phenolic (5). The phenolic resins exhibited many desirable properties, and entered the commercial market in 1909 (6). The widely-used phenolic, bakelite, is named for its developer, Dr. Leo H. Baekeland.

It was apparently some time, however, before bakelite became significant as a metallographic mounting medium. Some of the more common materials used during the early 1900's included paraffin wax, sealing wax, sulfur, various plasters and cements, and low-melting point alloys (7-11). (See Appendix I for compositions of various low-melting point alloys.) A paper by Ragatz in 1929 (9) gave a comprehensive review of some of the mounting materials in use at that time including Lipowitz alloy, iron cement, litharge and glycerin cement, plastic magnesia cement, and sulfur. He also referred to a 1927 paper on the use of glass for mounting of fine tungsten filaments. (A tungsten-in-glass technique was reported more recently in 1959 (12).)

All of these early mounting materials were generally undesirable for one reason or another. They resulted in difficulties such as rapid clogging of grinding papers, undesirable etching effects, corrosion of the sample, inadequate hardness, poor grinding and polishing characteristics, water absorption, and poor adherence. However, as recently as 1968 a procedure using a low-melting alloy, Wood's Metal, was reported (13) for mounting metallic sodium.

By the early 1930's, the use of bakelite as a mounting material was becoming more common, and one paper described it as "the most satisfactory mounting material to be had" (14). And by standards of that time, it probably was, since it avoided most of the difficulties mentioned above.

Bakelite gained wider acceptance and usage during the 1930's, and pressure-molding techniques were developed (8,14). Also during this decade, more synthetic plastics became commercially available. These included the vinyls, polystyrene, polyethylene, and the acrylates (8,15). Some of these plastics were evaluated for use as mounting materials by Wyman (15), and although he

mentions the use of casting resins, the emphasis at that time remained on pressure-molding techniques..

Even though the epoxies were developed in the mid-1940's (5), their use in metallography did not occur for some years. During the '40's, the mainstay materials included bakelite, lucite, and tenite. However, Kehl and Church (16) recognized the value of using a castable resin rather than pressure molding for some types of samples, and they devised a rather involved technique for casting of methyl methacrylate (lucite). The development of many castable resin systems and epoxies followed during the 1950's and 1960's, bringing us up to the commonly-used products of today.

Most of the important aspects of high-quality metallographic preparation were recognized by the metallographers of the early 1900's. One of the primary subjects of concern then, as now, was that of edge retention and preservation of surface features. References to the use of electroplating for edge retention go back as far as 1922 in ASTM Method E-3 (17), and this has remained one of the most acceptable techniques. Additions of various filler materials to the

mounting medium in order to control mount hardness and polishing characteristics has also been widely used. These additions include such diverse materials as wood and mineral flours (15), iron grit (18), ground glass (19), and pelletized alumina (20,21).

And so we see that metallographic mounting methods have been extremely varied over the years, making use of some materials and techniques which today seem rather primitive. Nevertheless, the basic principles and objectives of metallographic mounting were established using these early techniques, and with all of our modern technology, we still don't have one universal mounting method for all applications - we still resort to a variety of techniques. And that is what we shall attempt to cover in the rest of this presentation.

MODERN MOUNTING TECHNIQUES

REQUIREMENTS

As an introduction to our discussion of the commonly-used mounting techniques, it might be worthwhile to briefly consider some of the requirements that should be kept in mind. The desirable characteristics of mounting materials have been discussed by several authors (6,7,11,15,16,21,22), and it is from these sources that the following remarks are taken.

The primary reason for mounting samples is simply to facilitate ease in handling. Sharp corners are eliminated, thereby increasing the safety to the metallographer and avoiding damage to the papers and cloths used for preparation. Also it is obviously very convenient to have all samples of a uniform size and shape, for automatic preparation equipment, viewing systems, and storage facilities.

The first concern in selecting a mounting medium has to be protection and preservation of the sample. Both physical damage to fragile or delicate specimens and structural

changes due to excessive heat or pressure must be considered. Also, it may be necessary to retain components in a specific relation to one another; surface scales, coatings, etc. must be preserved.

The mount should have sufficient hardness, and it should, ideally, have grinding and polishing characteristics similar to the specimen. (Hardness is not always an indication of abrasion characteristics.) It should resist physical distortion due to the heat generated during grinding, polishing, etching, and washing, and it should not clog grinding papers rapidly. The mount should also be chemically inert to the variety of lubricants, solvents, and etchants which are used.

The mount material should be capable of penetrating small pores, crevices, convolutions, and other surface irregularities. It should exhibit good adhesion to the sample surface, and not form a shrinkage gap between the sample and the cured mount. It should retain the specimen edge without rounding.

Depending on the type of sample, the mount material may need to be either electrically conductive or insulating. The

mount material must not cause unwanted etching effects from chemical or electrochemical reactions.

The mounting medium should be simple and fast to use and convenient to store. It should not be prone to formation of defects in the cured mount, such as cracks or voids. Often it is advantageous for the mount to be transparent. The mount material should present no health hazards, and it should be readily available at a reasonable cost.

This is a pretty formidable list of requirements, and no one mounting technique will fulfill them all. However, by judicious selection of materials and methods, it should be possible to obtain a mount which does achieve the most critical requirements for a given situation.

MECHANICAL MOUNTS

Numerous references to the mounting of samples by mechanical devices can be found in the literature (e.g., 9,11,17,22,23,24). Normally these techniques will be employed for specimens of regular shape and sturdy character. Although steps can be taken to achieve a degree of edge retention, this is not normally an advantage to mechanical methods. These techniques can be quite rapid, and if for some reason plastic mounts cannot be used, mechanical means may be the only alternative. In cases where subsequent examinations require an unmounted sample, removal from a mechanical clamp is obviously much easier than removal from a plastic mount.

Normally the specimen, or a stack of specimens, is clamped between suitable end plates (Fig. 1a & b) or is held within a cylinder or other convenient shape by a set-screw (Fig. 1c). Ideally, the end plates or surrounding member should have abrasion and polishing characteristics similar to the specimen material. Also, the mechanical mount should either be inert to the solvents and etchants used or have the same reactivity as the specimen.

The clamping pressure used to hold the sample within the mechanical device can be very critical. If insufficient pressure is used, there can be gaps remaining which may trap grit particles or cause staining due to seepage of lubricants and etchants. If the clamping pressure is excessive, physical damage to the specimen may result.

Various materials may be used as spacers if a stack of samples is being prepared. For maximum edge retention, a spacer with the same or slightly lower abrasion and polishing rates than the specimen would probably be selected. However, care must be exercised in selecting the spacer material so that undesirable galvanic reactions are avoided. If edge staining is a problem, the use of softer spacers, such as a soft metal or plastic sheet, would help. Coating of the individual samples with a thick layer of lacquer or phenolic or epoxy resin can also be effective. Immersion in molten paraffin may also be an effective means of filling pores and reducing the absorption of reagents. Reagent absorption and seepage can also be reduced by using alternate layers of dense blotting paper, but the paper may pick up abrasive particles, thus causing more problems than it cures.

PRESSURE MOUNTS

The various plastics used for metallographic mounting may be classed in several different ways, according to the technique used and the properties of the material. It is often convenient to divide these plastics into one group which requires the application of heat and pressure and another group which is castable at room temperature, and this is the basis for classification that we shall use here. Thermosetting and thermoplastic materials are in the class which requires the application of heat and pressure for curing. Let us start this discussion with some remarks which are applicable to pressure molding of both the thermosetting and the thermoplastic resins (7,11,15,17,22,23,25,26).

It was mentioned earlier that pressure-molding techniques using various plastic resins were developed during the 1930's. For about three decades, these methods were the most widely used, since they overcame many of the difficulties inherent in the earlier techniques. Even with the advent of the castable resins ~~and~~^{with} their advantages, the pressure-molding methods have remained very popular. Good quality mounts can be prepared rapidly and simply, and they are

characterized by relatively high hardness and good chemical resistance to a wide variety of reagents and solvents. These materials are fairly versatile and are available in transparent form as well as in a wide range of colors. They are all sold as uncured powder, and bakelite can also be purchased as a preformed pellet. Filler materials may be added to improve abrasion resistance or to achieve electrical conductivity. (Figure 2)

Mounting of the thermoplastic and thermosetting resins is accomplished in a hydraulic-type press, which may range from a simple, homemade design to an elaborate automatic unit. (Figure 3) Temperatures up to about 300°F and pressures up to 4200 psi are required. The press should have a high capacity heater surrounding the specimen mold so that curing times are held to a minimum. A means of rapidly cooling the mold is also required for the thermoplastic materials. The press should also have an automatic temperature controller, a mold ejection device, and a timer. Standard mold diameters are 1", 1-1/4", and 1-1/2".

Proper mounting technique starts with a sample which is clean and dry, and at least 1/2 in. smaller than the mold diameter. Unless they are necessary for examination of

the sample, all sharp corners and edges should be eliminated. In order to avoid early partial curing of the resin, the die set should be reasonably cool when the resin is added. Alternatively, pressure must be applied without delay. Sufficient plastic must be used to cover the specimen to prevent damage to the top die and to maintain sufficient molding pressure. Pressure control is not overly critical, as long as the required minimum for the resin is exceeded. Excessive pressure may damage some samples. Temperature control is more critical; the required minimum must be reached without exceeding the maximum. If temperature is too high, the thermosetting resins may char and the thermoplastic resins may become so fluid that they are squeezed out through the mold parts. Good surface finish and close tolerances on the mold parts are important.

With these similarities in mind, let's look now at some of the differences between the thermosetting and thermoplastic resins.

Thermosetting Resins

The two most widely used thermosetting resins are bakelite and diallyl phthalate. Melamine has also been used, but is rather brittle when used alone. These resins actually undergo a chemical change at the molding temperature and pressure, and after sufficient time at the appropriate conditions they are fully cured. They may be ejected from the mold while hot. Several types of molding defects are common with these resins, and they are illustrated in Figure 4.

Table I summarizes typical properties of bakelite and diallyl phthalate (11,17,22,23,25). The data from Table I show that both of these resins have similar curing cycles, so one mounting press can be used for either type. The heat distortion temperature listed in the table probably has more meaning in relation to the curing cycle than to subsequent distortion after curing. The thermosetting resins are normally considered to undergo no actual softening at high temperatures, but rather to char if the temperature is too high.

The coefficient of thermal expansion of these resins is relatively high compared to most metals, which are normally about $1-3 \times 10^{-5}$ in/in/°C. According to Samuels (22) this large difference is desirable in most cases, since the greater shrinkage of the plastic during cooling tends to hold the sample more tightly. This is an advantage because with these resins there is little or no actual adhesion to the sample surface. However, for certain sample shapes, this large difference in shrinkage can be a big disadvantage. For example, a tube section may be gripped tightly on the outside diameter, but on the inside, the shrinkage tends to pull the mount material away from the sample. This is demonstrated in Figure 5. The differential in thermal expansion can be altered somewhat by adding wood or mineral fillers to the powdered resin. These resins are normally purchased with the desired filler already added.

The abrasion and polishing rates of the thermosetting resins can also be altered by changing the type of filler material. For example, glass fibers are often added to diallyl phthalate instead of asbestos, and this lowers the abrasion rate and improves edge retention.

The thermosetting resins are decomposed by strong oxidizing acids and strong alkalies. They are resistant to strong reducing acids, organic acids, weak alkalies, mild etchants, alcohols, and organic solvents (15,22).

The major advantages of the thermosetting resins are that they are inexpensive, quickly and simply prepared, reasonably hard, and chemically resistant to most metallographic reagents. Their disadvantages include possible damage or alteration to fragile or heat-sensitive samples, gaps between sample and mount due to lack of adhesion and differential thermal contraction, and usually less than optimum edge retention properties.

Thermoplastic Resins

Several types of molding resins are classed as thermoplastics. These include polystyrene, polyvinyl chloride (PVC), polyvinyl formal (formvar), and methyl methacrylate (lucite, transoptic). Of these, methyl methacrylate is probably most widely used. The thermoplastic resins differ from the thermosetting in that they do not cure at the molding temperature. Rather, they become fluid at this temperature, and then must be cooled under pressure to produce the cured mount. Common mount defects, their causes, and remedies are illustrated in Figure 6 (7,22,23,25).

The properties of several thermoplastic resins are summarized in Table II (11,15,17,22,23,25). The heat and pressure required during the molding cycle are similar to that required by the thermosetting resins. With the addition of a mold cooling device, the mounting presses used for thermosetting resins may also be used for the thermoplastics. One advantage of most thermoplastic resins over the thermosetting types is that maximum pressure need not be applied until the resin is completely molten, thus avoiding damage to some specimens. Another advantage over the thermosetting resins is the transparent mount that can be obtained with the methyl methacrylate and formvar resins.

From Table II, we see that the thermoplastic resins are characterized by fairly low heat distortion temperatures. This results in softening and distortion of the mount if frictional heating during grinding and polishing is not properly controlled. Damage to the mount can also occur if insufficient heat filters are used when viewing small specimens under high intensity illumination, as Richardson has pointed out for PVC (23).

The thermoplastic resins possess fairly high coefficients of thermal expansion - generally somewhat higher than the thermosetting materials. As previously discussed, this high coefficient may be an advantage or a disadvantage, depending on sample geometry. Samuels points out that epoxies are the only mounting resins which show true adherence to the sample (22). Therefore, the thermoplastics, like the thermosetting resins, must rely on mechanical gripping which can be either enhanced or limited by differential thermal contraction. In extreme cases of mount shrinkage, whole sections of the mount may loosen and possibly fall out. For example, a longitudinal section of tubing may result in this type of mount defect. Richardson reports a simple remedy for this situation (23). By drilling one or more holes through the tube wall, additional bonding and support is provided for the portion of mount material on the tube I.D.

The abrasion and polishing rates for the thermoplastic resins are generally lower than they are for the thermosetting resins. Of the thermoplastics, formvar and PVC seem to have the best polishing characteristics (22,27).

The chemical resistance of the thermoplastic resins is good, except most are attacked by strong acids such as nitric and acetic, and some are at least partially soluble in organic solvents, such as acetone or chlorinated hydrocarbons. All show good resistance to the usual etching reagents, dilute acids, and alcohol.

Probably the major advantage of the thermoplastics is that some of them produce clear, transparent mounts. Their other properties, in general, are not significantly better than those of the thermosetting resins, and the thermoplastics do require a controlled cooling cycle which may be inconvenient. The thermoplastics do not completely solve the problems of edge retention and gaps between sample and mount.

CASTABLE MOUNTS

The use of castable resins, or "cold mounting" plastics, has been growing in popularity for about 15-20 years.

These resins offer certain advantages over the pressure molding resins, and possess properties which expand the mounting capabilities of the laboratory (6,7,20-22,29-39).

These resins are normally two-component systems, liquid-liquid, liquid-solid, or solid-solid (which must be melted for use). One component is the resin and the other component is variously known as the hardener, activator, or catalyst. Resins available for cold mounting applications include polyesters, polystyrenes, acrylics, and epoxides.

(Polystyrenes and acrylics were also discussed under Pressure Mounts.) (Figure 7)

The castable resins are readied for use by thoroughly mixing the required amount of resin and hardener. Pot life of the mixture will vary from a few minutes to many hours, depending on resin type and temperature of storage if the resin is not to be used immediately. Mounts are prepared by placing the sample into a re-usable mold, and then filling the mold with the resin. The mount is cured at room temperature or slightly above.

A variety of materials are used for molds, including glass, aluminum, brass, steel, teflon, and silicone rubber. Mold release agents such as vacuum grease or silicone oil are often required, depending on the mold material and the type of resin. Epoxies adhere to most materials, so epoxy mounts are probably most difficult to remove from the mold. However, silicone rubber molds have proved very convenient to use with the epoxies as well as the other castable resins (22, 23, 28, 38). Although molds are normally made in the same range of standard sizes used for pressure mounts, the size and shape of the mold and resulting mount are limited only by the capabilities of the grinding and polishing equipment available. Mount defects common to the castable resins are illustrated in Figure 8 (7).

The castable resins have a wide range of applicability in situations where pressure mounting is impossible, inconvenient, or ineffective. The mounting of very fragile or delicate samples is easily accomplished with the castable resins. Vacuum impregnation is often used to improve mount quality for these specimens (7, 20, 21, 22, 29-32). Several authors have reported on apparatus and techniques to accomplish vacuum impregnation, (22, ²³30, 32), but the simplest method is probably to use a small chamber such as an oven connected to a

mechanical vacuum pump (20, 21). Re-impregnation after successive grinding steps is sometimes necessary to insure specimen integrity or to eliminate voids on the mount face.

Relatively low curing stresses for the castable resins are an advantage in mounting delicate specimens. However, some specimens, because of their geometry, may be damaged by the curing stresses that are present. Crouse and Gray (37) investigated brazed T-joints which cracked after being mounted in castable resins. They were able to reproduce the cracks using several different resins, and attributed the defects to the curing stresses. To alleviate cracking in the joint, the specimens were nickel-plated prior to mounting. The joints did not crack, but the nickel plating was separated from the specimens, leaving an undesirable gap. Pelletized alumina particles added to the resins was the most promising approach to reducing the stress on the sample while maintaining integrity of the mount-to-specimen interface.

Castable resins have found wide use in the nuclear field, where the simple casting techniques are easily adaptable to hot cell or glovebox work (31-36). Chang, et al., (34) have reported a technique for minimizing the amount of resin

which must be cured inside a glovebox. They make a pre-mold outside the glovebox which leaves a small conical or pyramidal cavity which is subsequently filled inside the glovebox with the sample and a small amount of uncured resin. This eliminates the use of dies inside the glovebox and minimizes contamination of the box atmosphere by gas released during curing.

The castable resins are also convenient to use when it is desired to alter the mount properties by addition of a filler material. Ground glass has been reported as an effective filler (19), and recent interest has centered on various grades of pelletized alumina (20, 21). Fillers are very effective in improving abrasion and polishing characteristics, which are not especially good for the cold mounting plastics.

The epoxides exhibit many characteristics which make them excellent resins for use in castable mounts. They adhere well to most other materials, therefore, they need not rely on differential shrinkage to grip the specimen. Of the castable resins, they have the lowest shrinkage factor. Some epoxies cure rapidly, but others require a much longer curing time than the other castable resins. However, this is a definite advantage when vacuum impregnation is necessary.

Curing can be hastened by heating to about 60-70°C. Use of a radio frequency oven to accelerate curing has also been reported (39). Some epoxies can be stored under refrigeration for several weeks after the hardener and resin have been mixed. The excellent bonding characteristics of the epoxies makes them practical to use for double mounting or re-impregnation techniques. Some epoxies show excellent transparency. Chemical resistance of the epoxies is very good except in concentrated nitric or glacial acetic acid. The epoxies are sensitive to slight variations in the resin to hardener ratio. The resin and hardener can be purchased in pre-measured packets to minimize mixing problems. Some systems are toxic or may cause skin irritation, so adequate ventilation is required and skin contact should be avoided.

The primary advantage of the acrylics is their rapid cure rate. Consequently, they do possess a relatively high exotherm, which may be undesirable. The curing exotherm may be controlled somewhat by the mount thickness. Also because of their short curing time, they are not very applicable to vacuum impregnation. They show the highest shrinkage of the castable resins. Acrylic mounts may be clear to opaque. The acrylics are attacked by strong acids and are somewhat soluble in some organic solvents. They

are probably less sensitive than the epoxies to variations in the mixture ratio. Breathing of the vapors or prolonged skin contact with the resins should be avoided.

The polyester resins exhibit less volume shrinkage than the acrylics, but they do rely on shrinkage to grip the specimen rather than bonding to the specimen. The polyesters cure fairly rapidly, but generally have longer curing times than the acrylics. They should therefore be more applicable to vacuum impregnation than the acrylics. They are not very sensitive to slight variations in the mixture. They show good chemical resistance to the normal metallographic reagents.

Probably the biggest advantage of the various castable resins is their versatility. By use of appropriate filler materials, the mount can be customized to the sample. Also, a wider variety of specimens, especially fragile or delicate items, can be mounted with castable resins than can be with the pressure-mounting techniques. On the other hand, the casting plastics must be measured fairly accurately, and generally require a longer curing time than the pressure-molding resins. They also exhibit relatively high shrinkage, which must be compensated for when mounting some types of specimens. The castable resins probably present a greater health hazard due to their vapors and possible skin irritant effects. However, these hazards are not great if reasonable care is used.

SPECIAL TECHNIQUES

In the foregoing sections, we have discussed the general procedures for mechanical, pressure, and castable mounts with only passing mention of some special problems which may be encountered. In this section, we will describe some of the techniques which have been reported for specific mounting requirements. These techniques will include mounting of small-diameter wires and thin sheet, edge retention, conductivity, and mount marking.

Mounting of Wire and Sheet

The mounting of small-diameter wire and thin sheet specimens can be rather challenging. Obtaining a truly accurate cross section, undistorted by tipping of the specimen, is the biggest problem. Good edge retention on these specimens is usually required also. Several authors have reported a variety of methods to achieve these ends (12,22,23,34, 41-46), and their remarks are summarized here. (Figure 9)

Double mounting techniques are very effective for both wire and sheet specimens. For example, holes or slots which are just larger than the specimen dimensions can be machined

into a preformed blank of cured or uncured resin. The specimen is inserted into the preform, and depending on the type of resin which was originally used, is held in place by one of several means. If the preform is of a thermosetting resin, additional resin may be added and the curing cycle repeated. For thermoplastic resins, simply repeating the molding cycle without the addition of more resin will suffice. Epoxy resin can also be used to bond the sample into the preform.

Another double mounting technique starts by mounting the specimen horizontally in any desired resin. This first mount is then cut to reveal the cross-section of the sample. The sectioned mount is then re-mounted, with the specimen in the vertical position.

A mineral-filled epoxy with a putty-like consistency can also be used. The samples are simply pushed into the mixture in the desired orientation, and after this mount cures, it can be re-mounted into a standard size if necessary.

Pyrex or quartz glass has also been used successfully as an initial mounting medium for wires. In the case of tungsten wires, the glass capillary can be heated until it collapses onto the wire, insuring intimate contact. For other materials, a capillary is chosen with an inside diameter just larger than the wire diameter. After inserting the wire into the glass capillary, the composite is mounted in either a pressure-type or castable resin.

Sheet specimens can often be made self-supporting by bending them into an "S" or "Z" shape. Also, they are relatively easy to support with curved pieces of shim stock or small coils or spring clips. Stacks of sheet specimens are usually mounted with spacers between them. The stack may be bolted together before mounting, or the spacers may be cemented into place between the sheets. A thin lead strip bent around and between the specimens can also be used. Usually it is advisable to arrange the spacers so they do not appear on the face of the mount after it has been ground and polished. If the sheet specimens have friable surface layers, these layers can often be protected from damage by alternating the samples between sheets of thin plastic, such as 0.004-inch ethyl cellulose. After clamping the stack, it is mounted

in acrylic resin. If an epoxy mount is preferred, polychlorotrifluoethylene film should be used.

Mounting for Edge Retention

Retention of specimen edges and the preservation of surface features has long been an area of concern to the metallographer. The techniques we shall discuss here cover about 50 years of effort devoted to the examination of specimen edges (7,9,11,12,17-23,25,31,35,40,47-52).

The quality of sample edge retention can be judged by the variation in light between the sample and the mount. This variation should not exceed the depth of field of the objective lens. So, for low-magnification viewing where depth of field is greater, more sample rounding can be tolerated than for high-magnification viewing. This may be an important consideration in determining which edge retention technique will be used. Samuels (22) has presented a comparison of the depths of field for typical objectives. His data are summarized in Table III.

In addition to specific methods which may be used to improve edge retention, there are factors which must be considered during specimen preparation which will affect sample

rounding (22). During grinding, the abrasion rate of the plastic mount will almost always be greater than the rate for a metallic sample. The plastic will grind away faster leaving the metal edges exposed and rounded off. Finer abrasives will produce less edge rounding than the coarser grits, and fixed abrasive laps cause less rounding than the abrasive papers. Also, the abrasion rates of the various plastics differ and affect the degree of rounding. Samuels (22) rates formvar and PVC as best, followed by the allyl plastics, and epoxy and the phenolics as worst. The abrasion rate of the sample is another consideration, and generally the lower the abrasion rate of the sample, the more edge rounding may be expected.

After grinding, the specimen will normally stand in relief above the mount, unless it is a very soft metal specimen. The relative polishing rates of the sample and the plastic will have a bearing on whether the relief and edge rounding become greater or less. A napless polishing cloth will help to improve edge retention, but it may also cause heavier scratching of the sample. Unidirectional polishing by the "trailing edge" technique may improve edge retention, but this is not normally recommended. It is very important to avoid rocking of the sample during all stages of grinding and polishing.

With these considerations in mind, let us now discuss improvement of edge retention in more specific terms. One of the simplest methods of edge retention, and one that is fairly effective, is the use of mechanical devices such as those described earlier in this presentation. If clamping devices and spacers between specimens are selected to closely match the abrasion and polishing rates of the specimen, reasonable edge retention can be achieved. These methods can be used with or without plastic mounting resins. When plastic mounts are used, a common practice is to simply include a metal ring which surrounds the sample in the mount.

One of the oldest techniques, and still considered by many to be the best, is to plate the sample either by electrolytic (Figure 10) or electroless^(Figure 11) deposition of metal. The plated sample can then be mounted in any convenient plastic resin. The metals commonly electrodeposited include copper, iron, nickel, and zinc. Copper may be plated from a cyanide or sulfate bath; the cyanide bath is often used to form a flash coating, with heavier deposition being made from the sulfate bath. Deposition of iron can be accomplished from a ferrous chloride solution, but is reportedly difficult to accomplish. Electrolytic nickel from a nickel sulfate-

nickel chloride bath is effective on ferrous alloys, nickel alloys, and metals of fairly high melting point. Zinc is plated onto zinc alloys from a cyanide solution. Compositions and operating conditions for several plating baths are presented in Appendix II..

Electroless plating can be accomplished with a variety of metals, such as copper, nickel, and silver. (See Appendix II). They have the advantage of being usable on nonconductive samples, and often they are used as an initial layer prior to electrodeposition. A commercially-available electroless nickel solution known as Edgemet is distributed by Buehler Ltd.

The biggest problem with electrodeposition is in obtaining a sufficiently clean specimen surface to insure adequate adhesion of the plating. Surface treatments used in industrial applications of electroplating are normally too harsh for metallographic samples and they may remove the features of interest. It may be possible to sufficiently clean the surface with detergent, solvents, or mild etchants if damage to the surface layers can be avoided.

Adhesion to the sample is also related to the internal stresses which are present in most electrodeposited metals. The residual stresses are surprisingly high, and may range from a few thousand psi compression to 100,000 psi tension, depending on the type of deposit. Kushner (49) has reported the approximate values of stress in various electrodeposited metals, and some of these data are presented in Table IV. It was also shown that residual stress is significantly affected by thickness of the plating and the type of plating bath that is used.

Some plating baths also have limited throwing power, and produce poor penetration of the coating into fine surface features. A thin electroless deposit can help alleviate this problem.

Electroless deposits do avoid some of the difficulties encountered with electrolytic deposits. They are reportedly stress-free, and they normally penetrate surface irregularities quite well. Also, any type of specimen may be plated, whether or not it is electrically conductive.

An additional advantage of metallic coatings is that they can help to improve the contrast between surface films and the mount material. Vapor deposition has also been used to apply coatings for improved contrast (48).

Another general method employed to improve edge retention is to make additions of a filler material to the mounting resin. The object is to alter the abrasion and polishing characteristics of the mount to more closely match the sample. An added benefit of the filler technique is that when some types of fillers are used, they effectively reduce the amount of shrinkage in the mount and help to eliminate the troublesome gap between the sample and the plastic.

Over the years, many types of filler materials have been used. Wood flour is usually added to bakelite, and mineral fibers are often added to diallyl phthalate. However, these additions are not especially effective in improving edge retention. Probably the most effective additive for thermosetting resins is short glass fibers, successfully used with diallyl phthalate. ^(Figure 12) Ground glass has been an effective additive to epoxy resins (19). The glass tube fused to tungsten mentioned earlier as an aid to mounting fine wires also served to improve edge retention of the wire (12).

Iron grit was advocated as a filler for edge retention (18), but there were difficulties with its use, and more effective fillers are now available. The grit had to be present in thin layers so the mounting plastic could fully penetrate it, and it could not be in contact with the specimen, or undesirable etching effects resulted when electrolytic methods were used. Also, the grit had a tendency to fall out of the mount if much grinding was required.

Probably the most popular filler now in use for edge retention is pelletized alumina. This filler is available in three hardness grades and in two mesh sizes for matching to the sample hardness and shape. This material is now available in black as well as white. When the black alumina is used in conjunction with a mounting resin to which black dye has been added, it is very effective in reducing reflected light from the mount surface. This is especially helpful in improving contrast of specimens being examined under polarized light. ^(Figure 13) Optimum wetting of the alumina by the resin is best accomplished by vacuum impregnation.

The use of pelletized alumina does slow the grinding and polishing steps, and additional abrasive is required. For

this reason, a double mounting technique is sometimes recommended in which the initial mount surrounds the area immediately adjacent to the sample with alumina. The second mounting step adds pure resin, so that the resulting final mount has a relatively small area of alumina exposed.

Mounting for Conductivity

The plastics used for metallographic mounts are normally electrical insulators, so special steps are necessary if electrolytic preparation methods are to be used. We will describe several methods of physically achieving electrical contact with the specimen and methods to make the mount itself electrically conductive (22,23,26,53,54). (Figure 14)

Contacting the sample surface with a probe results in uneven current distribution, so this simple approach is not always satisfactory. A better method is to drill a hole through the back of the mount and into the specimen. A screw is then self-tapped into the hole to act as the electrical contact. This method is not always satisfactory, however, since the sample may be damaged or electrical contact may not be sufficient. Also, it is possible to apply enough pressure

when inserting the screw to actually pop the specimen partially out of the mount. If castable resins are to be used, leads may be attached to the specimen before mounting and left protruding from the back of the mount.

Another approach is to place metal balls or rods on the back surface of the specimen prior to mounting. After the mounting plastic is cured, the back of the mount can be machined to expose the balls or rods. A variation of this technique was described by Hoffman (53). His mounts were prepared in just the reverse order to those described above. A thick metal disc, which will eventually become the back of the mount, is placed into a mold, and the sample placed on top of the disc. Mounting resin is then poured into the mold to cover the specimen. After curing, the mount is ground to expose the specimen. This technique is supposed to work even if the specimen is a fine powder. A further modification reported by Richardson (23) includes the use of a conductive cement to insure positive contact of the specimen to the metal disc. In addition to providing the necessary electrical contact, the metal disc can serve as a clamping base for a magnetic chuck for hot cell use.

For some applications, the most satisfactory solution is to make the mount itself conductive. According to Samuels (22) a resistance of about 100 ohms from front to back of the mount is desirable. It is possible to achieve satisfactory conductivity by mixing approximately 10 volume percent metal with the molding plastic. However this method is probably the least reliable. Good results can be obtained if the individual plastic particles are coated with a conductor. For example, PVC can be milled with carbon black to produce a conductive mounting material (22).

Another combination of conductor and mounting resin is the copper-filled diallyl phthalate powder which is commercially available. This material can be used by itself or in conjunction with straight diallyl phthalate in a technique described by Ladroga (26). In this method, a small tube is used to confine the copper-filled resin to the area adjacent to the sample. Un-filled resin makes up the remainder of the mount. This technique is effective if a completely conductive mount face is not desired. For example, in a vacuum cathodic etcher, arcing can occur if the entire mount is conductive. This application of the technique was also described by Dobbins (54).

Mount Marking

It may be appropriate to conclude our discussion of mounting methods with a few brief comments on the marking of mounts for identification. Hand scribes and vibrating-point tools are used extensively for this purpose. If the resulting markings are difficult to see, their visibility can be improved by inking over the scribe lines. Ink will adhere well to the roughened scribed areas, but it usually will not wet the smooth surfaces of most plastic mounts.

Bartell (55) reported that 2 ml of ethyl alcohol added to 1 oz. of India ink acts as an effective wetting agent. Any portion of the mount can then be written on with the ink mixture.

A more permanent type of mount identification can be achieved if a clear mounting resin is used (56). In this case a small metal tag or a scrap of paper with the identification can be included in the mount with the sample. The identification is then permanently visible and protected with the sample.

A similar technique was reported by Clayton (57) for preserving the polished and etched surface of mounted specimens with a

thin layer of lucite (or any other clear resin, presumably). The protected surface can still be examined at magnifications up to about 100X. This technique could be of value in cases where repeated examination is required, such as in the classroom.

CONCLUSION

If there is any one conclusion that can be drawn from the material presented here, it is that there is a mounting technique available for virtually any type of specimen. And the metallographer's first concern must be to select the technique which will best permit him to preserve the specimen and to prepare it for thorough examination. We have seen that each mounting technique and each mounting material has advantages and disadvantages, and no single material can fulfill every desirable requirement. However, by intelligent selection of materials and techniques, with proper diligence and care, and sometimes with a bit of imagination, this first step of metallographic preparation can foster success in the other steps that follow.

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FIGURE 1

Sketches of several types of mechanical mounts.
(Art work to be done)

FIGURE 2

Examples of mounts made with various
thermo setting resins

20-1341

FIGURE 3
Examples of commercially available mounting presses.

DEFECT	CAUSE	REMEDY
Partial Split	Too large a section in the given mold area. Sharp cornered specimens.	Increase mold size. Reduce specimen size.
Edge Shrinkage	Excessive shrinkage of plastic away from sample.	Decrease molding temperature. Cool mold slightly prior to ejection.
Circumferential Splits	Absorbed moisture. Entrapped gasses during molding.	Preheat powder or premold. Momentarily release pressure during fluid state.
Burst	Too short a cure period. Insufficient pressure.	Lengthen cure period. Apply sufficient pressure during transition from fluid state to solid state.
Unfused	Insufficient molding pressure. Insufficient time at cure temperature. Increased surface area of powdered materials.	Use proper molding pressure. Increase cure time. With powders — quickly seal mold closure and apply pressure to eliminate localized curing.

FIGURE 4
Mold defects in mounts made with thermosetting resins.
(Art work to be done)

FIGURE 5
Photomicrograph to be taken at approx 100-500 X
showing occurrence of shrinkage gap on a tube I.D.

DEFECT	CAUSE	REMEDY
Concave	Powdered media did not reach maximum temperature. Insufficient time at maximum temperature.	Increase holding time at maximum temperature.
Cracking	Inherent stresses relieved upon or after ejection.	Allow cooling to a lower temperature prior to ejection. Temper mounts in boiling water.

Acrylics:

DEFECT	CAUSE	REMEDY
Bubbles	Too violent agitation while blending resin and hardener.	Blend mixture gently to avoid air entrapment.

FIGURE 6

Mold defects in mounts made with thermoplastic resins.
(Art work to be done)

FIGURE 7

Examples of mounts made with castable resins

Epoxides:

DEFECT

Cracking

Bubbles

Discoloration

Soft Mounts

CAUSE

Insufficient air cure prior to oven cure.
Oven cure temperature too high.
Resin to hardener ratio incorrect.

REMEDY

Increase air cure time.
Decrease oven cure temperature.
Correct resin to hardener ratio.

Too violent agitation while blending resin & hardener mixture.

Blend mixture gently to avoid air entrapment.

Resin to hardener ratio incorrect.
Oxidized hardener.

Correct resin to hardener ratio.
Keep containers tightly sealed.

Resin to hardener ratio incorrect.
Incomplete blending of resin-hardener mixture.

Correct resin to hardener ratio.
Completely blend mixture.

Polyesters:

DEFECT

Cracking

Discoloration

Soft Mounts

Tacky Tops

CAUSE

Insufficient air cure prior to oven cure.
Oven cure temperature too high.
Resin to hardener ratio incorrect.

Resin to hardener ratio incorrect.
Resin has oxidized.

Resin to hardener ratio incorrect.
Incomplete blending of resin-hardener mixture.

Resin to hardener ratio incorrect.
Incomplete blending of resin-hardener mixture.

REMEDY

Increase air cure time.
Decrease oven cure temperature.
Correct resin to hardener ratio.

Correct resin to hardener ratio.
Keep containers tightly sealed.

Correct resin to hardener ratio.
Completely blend mixture.

Correct resin to hardener ratio.
Completely blend mixture.

FIGURE 8

Mold defects in mounts made with castable resins

FIGURE 9

Miscellaneous methods of supporting wire, sheet, and tube specimens for mounting.

FIGURE 10

Photomicrograph to be taken at about 100-500x showing an example of sample edge retention by use of electrolytic surface deposit.

FIGURE 11

Photomicrograph to be taken at about 100-500x showing an example of sample edge retention by use of an electroless surface deposit.

FIGURE 12

Photomicrograph to be taken at about 100-500x of an example of sample edge retention using glass-filled diallyl phthalate

Mounted in Epoxy & White
Alumina Filler

Mounted in Epoxy & Black
Alumina Filler

FIGURE 13

Comparative photomicrographs using polarized light showing difference in contrast using white and black alumina filler. (From authors' previous paper, reference 21)

FIGURE 14
Examples of mounting to achieve sample conductivity.

TABLE I
TYPICAL PROPERTIES OF THERMOSETTING MOLDING RESINS⁽¹⁾

Resin	Molding Conditions			Heat Distortion Temp. - °C (2)	Coeff. of Thermal Expansion in/in/°C	Abrasion Rate µm/min (3)	Polishing Rate µm/min (4)	Trans- parency	Chemical Resistance
	Temp. °C	Press. psi	Time min						
Bakelite (wood filled)	135-170	2500-4200	5-12	140	$3.0-4.5 \times 10^{-5}$	100	2.9	Opaque	Attacked by strong acids & alkalis
Diallyl phthalate (asbestos-filled)	140-160	2500-3000	6-12	150	$3-5 \times 10^{-5}$	190	0.8	Opaque	Attacked by strong acids & alkalis

- Notes:
1. Data from references 11,17,22,23,25.
 2. Determined by method ASTM D648-56 (22).
 3. Specimen 1 cm² area abraded on slightly worn 600 grit SiC under load of 100 g. at rubbing speed of 10⁴ cm/min. (22).
 4. One-inch diameter mount on a wheel rotating at 250 rpm covered with synthetic swede cloth and charged with 4-8 µ diamond (22).

TABLE II
TYPICAL PROPERTIES OF THERMOPLASTIC MOLDING RESINS⁽¹⁾

Resin	Molding Conditions						Heat Distortion Temp. - °C (2)	Coeff. of Thermal Expansion in/in/°C	Abrasion Rate (3)	Polishing Rate (4)	Trans- parency	Chemical Resistance
	Heating			Cooling								
	Temp. °C	Press. psi	Time min	Temp. °C	Press. psi	Time min						
Methyl methacrylate	140-165	2500-4200	6	75-85	max	6-7	65	$5-9 \times 10^{-5}$	-	7.5	Water white to clear	Not resistant to strong aci & some solver
Polystyrene	140-165	2500	5	85-100	max	6	65					
Polyvinyl formal	220	4000	-				75	$6-8 \times 10^{-5}$	20	1.1	Light brown, clear	Not resistant to strong aci
Polyvinyl chloride	120-160	100	nil	60	4000		60	$5-18 \times 10^{-5}$	45	1.3	Opaque	Resistant to most acids ar alkalis

- Notes: 1. Data from references 11,15,17,22,23,25
2. Determined by method ASTM D648-56 (22).
3. Specimen 1 cm² area abraded on slightly worn 600 grit SiC under load of 100 g. at rubbing speed of 10⁴ cm/min. (22).
4. One-inch diameter mount on a wheel rotating at 250 rpm covered with synthetic swede cloth and charged with 4-8 μ diamond (22).

TABLE III
 DEPTHS OF FIELD OF TYPICAL MICROSCOPE OBJECTIVES⁽¹⁾

Final Magnification	Objective Lense		Type	Depth of Field, μm
	Magnification	Numerical Aperture		
100	5.6	0.20	Dry	20
250	8.0	0.40	Dry	3
500	21.0	0.65	Dry	1
750	41.0	0.85	Dry	0.4
1000	58.0	0.95	Dry	0.1
1000	50.0	1.0	Oil	0.6
1500	75.0	1.4	Oil	0.2

1. Data from Samuels (22).

TABLE IV
APPROXIMATE RESIDUAL STRESS IN
ELECTRODEPOSITED METALS⁽¹⁾

Metal	Stress in Electrodeposit (2) psi
Cd	-500 to -300
Zn	-1000 to -2000
Al	0
Ag	+2000
Au	+1500
Cu	+2000
Ni	+20,000
Fe	+40,000
Cr	+60,000
Rh	+100,000

(1) Data from Kushner (49)

(2) Approximate relative order of magnitude
for thin films.

APPENDIX I
SEVERAL LOW MELTING POINT ALLOYS FOR
SPECIMEN MOUNTING

1. Woods Alloy: melting point 70°C

Bi	50.0
Pb	25.0
Sn	12.5
Cd	12.5

2. Lipowitz Alloy: melting point 70°C

Bi	50.0
Pb	27.0
Sn	13.0
Cd	10.0

3. Cadmium-Zinc Eutectic: melting point 265°C

Cd	82.5
Zn	17.5

4. Lead-Tin Eutectic: melting point 183°C

Pb	38.1
Sn	61.9

5. Cerrolow*117: melting point 47°C

Bi	44.7
Pb	22.6
Sn	8.3
Cd	5.3
In	19.1

*Trademark, Cerro Corp., New York

6. Cerrolow*136: melting point 58°C

Bi	49.0
Pb	18.0
Sn	12.0
In	21.0

7. Cerrobend*: melting point 70°C

Bi	50.0
Pb	26.7
Sn	13.3
Cd	10.0

*Trademark, Cerro Corp., New York

APPENDIX II

SOLUTIONS FOR ELECTROLYTIC AND ELECTROLESS DEPOSITION OF METALS

1. Copper, cyanide bath (Ref. 22. See also refs. 11 and 23)

Solution: CuCN 20g/l
 NaCN 30g/l
 NaOH 1.5-3 g/l

Temperature: 45-60°C

Voltage: 4-6 volts

Current Density: .5-1 ma/cm²

Agitation: mild stirring

Anode: copper

2. Copper, acid bath (Ref. 22. See also ref. 11)

Solution: CuSO₄ · 5H₂O 170g/l
 H₂SO₄ (conc.) 60g/l

Temperature: 15-50°C

Voltage: 1-4 volts

Current Density: 10-20 ma/cm²

Agitation: mild stirring

Anode: Copper

3. Iron (Ref. 22. See also ref. 11)

Solution: FeCl_2 288g
NaCl 57g
 $\text{H}_2\text{O}(\text{dist.})$ 1l
Filtered for use
Temperature: 70-100°C
(Must make up for evaporation loss
with distilled water.)
Current Density: .5-4 a/dm²
Agitation: Specimen (cathode) suspended and
rotated @ 50 rpm.
Anode: Ingot iron plate

4. Nickel (Ref. 22. See also ref. 23)

Solution: $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ 300g/l
 $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ 60g/l
Boric acid 40g/l
pH 4
Add 1 part 30% H_2O_2 per 200 parts
solution once a day
Temperature: 40-70°C
Voltage: 1-3 volts
Current Density: 30 ma/cm²
Agitation: Vigorous stirring
Anode: Anode nickel

5. Zinc (Ref. 22)

Solution: $\text{Zn}(\text{CN})_2$ 60g/l
 NaCN 23g/l
 NaOH 53g/l

Temperature: Room temperature

Voltage: 1-4 volts

Current Density: 10-15 ma/dm²

Anode: Zinc

6. Brashear Process for Silvering (Ref. 22. See also ref. 51)

Stock Solutions: A. Silver nitrate 20g
 Water to 300 ml

 B. Potassium hydroxide 14g
 Water to 100 ml
 (Make fresh or store in polythene)

 C. Ammonium hydroxide, s.g. 0.880

 D. Dextrose 6.5g
 Water to 100 ml
 (Make fresh)

Mixing the
Silvering
Solution:

To 3 volumes of A, add C until precipitate is just redissolved. Add A by drops until solution is straw-colored. Add 1 volume of B with stirring. Add C until solution clears. Add A until permanent precipitate forms. Filter and use same day. Dispose of unused solution the same day to avoid formation of explosive compound.

Silvering:

Mix 3 volumes of silvering solution with 1 volume of D. Immerse specimen immediately. Silver for about 3 minutes, rinse in distilled water, apply second coat. Rinse and transfer immediately to desired electroplating bath.

7. Electroless Nickel (Ref. 47)

Solution:

$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ 30g/l

$\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$ 10g/l

$\text{Na}_3\text{C}_6\text{H}_2\text{O}_7 \cdot \text{H}_2\text{O}$ 100g/l

NH_4Cl_2 50g/l

NH_4OH to adjust pH to 8-10

Dissolve in order given in warm distilled water.

Plating:

Bring solution to low boil, immerse specimens. Plating time about 1-1/2 hours.