~~Designation:E3–95~~

~~An American National Standard~~

~~Standard Practice for~~ Designation: E 3 – 01 (Reapproved 2007)~~ε1~~ ~~Preparation~~ of Metallographic Specimens[1]

This standard is issued under the fixed designation E 3; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

ε[1] NOTE—Section 13, Precision and Bias was editorially removed from the standard in March 2009.

1. Scope

1.1 The primary objective of metallographic examinations is to reveal the constituents and structure of metals and their alloys by means of ~~the~~a light optical or scanning electron microscope. In special cases, the objective of the examination may require the development of less detail than in other cases but, under nearly all conditions, the proper selection and preparation of the specimen is of major importance. Because of the diversity in available equipment and the wide variety of problems encountered, the following text presents for the guidance of the metallographer only those practices which experience has shown are generally satisfactory; it cannot and does not describe the variations in technique required to solve individual specimen preparation problems.

NOTE 1—For a more extensive description of various metallographic techniques, refer to Samuels, L. E., *Metallographic Polishing by Mechanical Methods*, American Society for Metals (ASM) Metals Park, OH, 3rd Ed., 1982; Petzow, G., *Metallographic Etching, ASM, 1978; and VanderVoort, G., Metallography: Principles and Practice* ~~, McGraw Hill, NY, 1984.~~ , McGraw Hill, NY, 2nd Ed., 1999.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

~~E7 Terminology Relating to Metallography~~

A 90/A 90M Test Method for Weight [Mass] of Coating on Iron and Steel Articles with Zinc or Zinc-Alloy Coatings

E 7 Terminology Relating to Metallography

E 45 ~~Practice~~ Test Methods for Determining the Inclusion Content of Steel

E 340 Test Method for Macroetching Metals and Alloys

E 407 ~~Test Methods~~ Practice for Microetching Metals and Alloys

E 768 Guide for Preparing and Evaluating Specimens for Automatic Inclusion Assessment of Steel

E 1077 Test Methods for Estimating the Depth of Decarburization of Steel Specimens

E 1122 Practice for Obtaining JK Inclusion Ratings Using Automatic Image Analysis

E 1245 Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis

E 1268 Practice for Assessing the Degree of Banding or Orientation of Microstructures

E 1558 ~~Guide to Electrolytic Polishing of Metallographic Specimens[2]~~ Guide for Electrolytic Polishing of Metallographic Specimens

E 1920 Guide for Metallographic Preparation of Thermal Sprayed Coatings

3. ~~Significance and Use~~

~~3.1 Microstructures have a strong influence on the properties and successful application of metals and alloys. Determination and control of microstructure requires the use of metallographic examination.~~

~~3.2 Many specifications contain a requirement regarding microstructure; hence, a major use for metallographic examination is inspection to ensure that the requirement is met. Other major uses for metallographic examination are in failure analysis, and in research and development.~~

~~[1] This practice is under the jurisdiction of ASTM Committee E-4 on Metallography and is the direct responsibility of Subcommittee E04.01 on Sampling, Specimen Preparation, and Photography.~~

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[1] This guide is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.01 on Specimen Preparation . Current edition approved July 1, 2007. Published September 2007. Originally approved in 1921. Last previous edition approved in 2001 as E 3 – 01.

~~3.3Proper choice of specimen location and orientation will minimize the number of specimens required and simplify their interpretation. It is easy to take too few specimens for study, but it is seldom that too many are studied.~~ Terminology

3.1 *Definitions*:

3.1.1 For definitions used in this practice, refer to Terminology E 7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *castable mount*—a metallographic mount generally made from a two component castable plastic. One component is the resin and the other hardener. Both components can he liquid or one liquid and a powder. Castable mounts generally do not require heat and pressure to cure.

3.2.2 *compression mount*—a metallographic mount made using plastic that requires both heat and pressure for curing.

3.2.3 *planar grinding*—is the first grinding step in a preparation procedure used to bring all specimens into the same plane of polish. It is unique to semi or fully automatic preparation equipment that utilize specimen holders.

3.2.4 *rigid grinding disc*—a non-fabric support surface, such as a composite of metal/ceramic or metal/polymer charged with an abrasive (usually 6 to 15μm diamond particles), and used as the fine grinding operation in a metallographic preparation procedure.

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5. Selection of Metallographic Specimens

~~4.1The~~5.1 The selection of test specimens for metallographic examination is extremely important because, if their interpretation is to be of value, the specimens must be representative of the material that is being studied. The intent or purpose of the metallographic examination will usually dictate the location of the specimens to be studied. With respect to purpose of study, metallographic examination may be divided into three classifications:

~~4.1.1~~5.1.1 *General Studies or Routine Work*—~~Specimens from locations that are~~ Specimens should be chosen from locations most likely to reveal the maximum variations within the material under ~~study should be chosen.~~ study. For example, specimens ~~should~~could be taken from a casting in the zones wherein maximum segregation might be expected to occur as well as specimens from sections where segregation ~~should~~could be at a minimum. In the examination of strip or wire, test specimens ~~should~~could be taken from each end of the coils.

~~4.1.2~~5.1.2 *Study of Failures*—Test specimens should be taken as closely as possible to the fracture or to the initiation of the failure. Before taking the metallographic specimens, study of the fracture surface should be complete, or, at the very least, the fracture surface should be documented. ~~Specimens~~ In many cases, specimens should be taken ~~in many cases~~ from a sound area for a comparison of structures and properties.

~~4.1.3~~5.1.3 *Research Studies*—The nature of the study will dictate specimen location, orientation, etc. Sampling will usually be more extensive than in routine examinations.

~~4~~5.2 Having established the location of the metallographic samples to be studied, the type of section to be examined must be decided.

5.2.1 For a casting, a section cut perpendicular to the surface will show the variations in structure from the outside to the interior of the casting.

5.2.2 In hot-worked or cold-worked metals, both transverse and longitudinal sections should be studied. Special investigations may ~~at times~~ require specimens with surfaces prepared parallel to the original surface of the product.

5.2.3 In the case of wire and small rounds, a longitudinal section through the center of the specimen proves advantageous when studied in conjunction with the transverse section.

~~4.3Cross~~5.3 Transverse sections or ~~transverse~~cross sections taken perpendicular to the main axis of the material are ~~more suitable~~often used for revealing the following information:

~~4~~5.3.1 Variations in structure from center to surface,

~~4~~5.3.2 Distribution of nonmetallic impurities across the section,

~~4~~5.3.3 Decarburization at the surface of a ferrous material (see Test Method E 1077),

~~4.3.4Depth of surface imperfections,~~

~~4.3.5Depth of corrosion,~~

~~4.3.6Thickness of protective coatings, and~~

~~4.3.7Structure of protective coating.~~

~~4.4Longitudinal sections taken parallel to the main axis of the material are more suitable for revealing the following information:~~

~~4.4.1Inclusion content of steel (see Practice E45~~

5.3.4 Depth of surface imperfections,
5.3.5 Depth of corrosion,
5.3.6 Thickness of protective coatings, and
5.3.7 Structure of protective coating. See Guide E 1920.
5.4 Longitudinal sections taken parallel to the main axis of the material are often used for revealing the following information:
5.4.1 Inclusion content of steel (see Practices E 45, E 768, E 1122, and E 1245),
~~4~~5.4.2 Degree of plastic deformation, as shown by grain distortion,
~~4~~5.4.3 Presence or absence of banding in the structure (see Practice E 1268), and
~~4.4.4The quality~~5.4.4 The microstructure attained with any heat treatment.
~~4.5The~~5.5 The locations of surfaces examined should always be given in reporting results and in any illustrative micrographs. A suitable method of indicating surface locations is shown in Fig. 1.

~~5.~~

6. Size of Metallographic Specimens

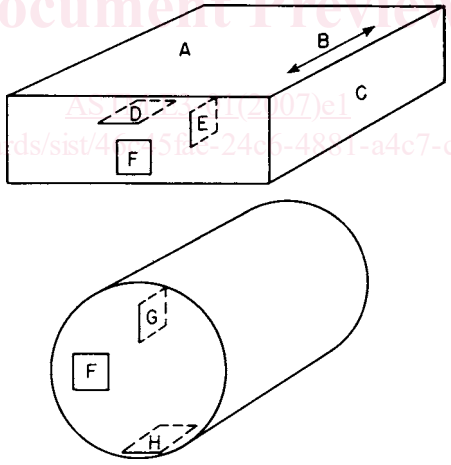
~~5.1The specimens to be polished for metallographic examination are generally not more than about 12 to 25 mm (0.5 to 1.0 in.) square, or approximately 12 to 25 mm in diameter if the material is round. The height of the specimen should be no greater than necessary for convenient handling during polishing.~~

~~5.2It is not always possible to secure specimens having the dimensions given in 5.1, when the material to be examined is smaller than the ideal dimensions. For example, in the polishing of wire, strip, and other small articles, it is necessary to mount the specimens because of their size and shape.~~

~~5.2.1Larger samples may be mounted or not, as the available equipment dictates. However, the larger the specimen, the more difficult it is to prepare, especially by manual methods.~~

~~5.2.2Specimens that are too small to be handled readily during polishing should be mounted to ensure a surface satisfactory for microscopical study. There are, based on technique used, three fundamental methods of mounting specimens (see Sections 7-9~~

6.1 For convenience, specimens to be polished for metallographic examination are generally not more than about 12 to 25 mm (0.5 to 1.0 in.) square, or approximately 12 to 25 mm in diameter if the material is cylindrical. The height of the specimen should be no greater than necessary for convenient handling during polishing.



Symbol in Diagram	Suggested Designation
A	Rolled surface
B	Direction of rolling
C	Rolled edge
~~D~~	~~Longitudinal (or lengthwi se) section parallel to rolled sur- face~~
D	Planar section
E	Longitudinal section perpendicular to rolled surface
F	Transverse section
G	Radial longitudinal section
H	Tangential longitudinal section

FIG. 1 Method of Designating Location of Area Shown in Photomicrograph.

6.1.1 Larger specimens are generally more difficult to prepare.

6.1.2 Specimens that are, fragile, oddly shaped or too small to be handled readily during polishing should be mounted to ensure a surface satisfactory for microscopical study. There are, based on technique used, three fundamental methods of mounting specimens (see Section 9).

~~6.~~

7. Cutting of Metallographic Specimens

~~6.1In~~7.1 In cutting the metallographic specimen from the main body of the material, care must be exercised to minimize altering the structure of the metal. Three common types of sectioning are as follows:

~~6~~7.1.1 Sawing, whether by hand or machine with lubrication, is ~~easy and~~ easy, fast, and relatively cool. It can be used on all materials with hardnesses below approximately ~~35 HRC.~~350 HV. It does produce a rough surface containing extensive plastic flow that must be removed in subsequent preparation.

~~6.1.2An~~7.1.2 An abrasive cut-off ~~wheel~~blade will produce a smooth surface often ready for fine grinding. This method of sectioning is normally faster than sawing. The choice of cut-off ~~wheel,~~blade, lubricant, cooling conditions, and the grade and hardness of metal being cut will influence the quality of the cut. A poor choice of cutting conditions can easily ~~overheat~~damage the specimen, producing an alteration of the microstructure. ~~As a general rule,~~ Generally, soft materials are cut with a hard bond ~~wheel~~blade and hard materials with a soft bond ~~wheel.~~blade. Aluminum oxide abrasive ~~wheels~~blades are preferred for ferrous metals and silicon carbide ~~wheels~~blades are preferred for nonferrous alloys. Abrasive cut-off ~~wheels~~blades are essential for sectioning metals with ~~hardnesses~~hardness above about ~~35 HRC.~~350 HV. Extremely hard metallic materials and ceramics may be more effectively cut using diamond-impregnated cutting ~~wheels.~~blades. Manufacturer's instructions should be followed as to the choice of ~~wheel and speeds.~~

~~6.1.3Flame cutting completely alters~~ blade. Table 1 lists the ~~structure of the metal at the flame cut edge. If flame cutting~~ suggested cutoff blades for materials with various Vickers (HV) hardness values.

7.1.3 A shear is ~~necessary to remove the specimen, it should be cut sufficiently large so that it can be recut to~~ a type of cutting tool with which a material in the ~~proper size by some other method that will not substantially alter the structure. Exercise care to ensure that the region of interest~~ form of wire, sheet, plate or rod is ~~not altered by the heat of the cutting flame.~~ cut between two opposing blades.

~~6.2Other~~7.2 Other methods of sectioning are permitted provided they do not alter the microstructure at the plane of polishing. All cutting operations produce some depth of damage, which will have to be removed in subsequent preparation steps.

~~7.~~8. Cleanliness

~~7.1Cleanliness (see~~

8.1 Cleanliness (see Appendix X1~~.~~)) during specimen preparation is essential. All greases, oils, coolants and ~~oils~~ residue from cutoff blades on the specimen should be removed by some suitable organic solvent. Failure to clean thoroughly can prevent cold mounting ~~castable~~ resins from adhering to the specimen surface. Ultrasonic cleaning ~~is particularly~~may be effective in removing the last traces of residues on a specimen surface.

~~7.2Any~~8.2 Any coating metal that will interfere with the subsequent etching of the base metal should be removed before polishing, if possible. If etching is required, when studying the underlying steel in a galvanized specimen, the zinc coating should be removed before mounting to prevent galvanic effects during etching. The coating can be removed by ~~digestion~~dissolving in cold nitric acid (HNO_3, sp gr 1.42), in dilute sulfuric acid (H_2SO_4) or in dilute hydrochloric acid (HCl). The HNO_3 ~~method requires care to prevent overheating, since large samples will generate considerable heat. By placing the cleaning container in cold water during the stripping of the zinc, attack on the underlying steel will be minimized.~~

~~7.3Oxidized or corroded surfaces may be cleaned as described in~~ method requires care to prevent overheating, since large samples will generate considerable heat. By placing the cleaning container in cold water during the stripping of the zinc, attack

TABLE 1 Cutoff Blade Selection

Hardness HV	Materials	Abrasive	Bond	Bond Hardness
up to 300	non-ferrous (Al, Cu)	SiC	P or R	hard
up to 400	non-ferrous (Ti)	SiC	P or R	med. hard
up to 400	soft ferrous	Al_2O_3	P or R	hard
up to 500	medium soft ferrous	Al_2O_3	P or R	med. hard
up to 600	medium hard ferrous	Al_2O_3	P or R	medium
up to 700	hard ferrous	Al_2O_3	P or R&R	med. soft
up to 800	very hard ferrous	Al_2O_3	P or R&R	soft
> 800	extremely hard ferrous	CBN	P or M	hard
	more brittle ceramics	diamond	P or M	very hard
	tougher ceramics	diamond	M	ext. hard

P—phenolic
R—rubber
R&R—resin and rubber
M—metal

on the underlying steel will be minimized. More information may be found in Test Method A 90/A 90M.

NOTE 2—Picral etchant produces little or no galvanic etching effects when used on galvanized steel.

NOTE 3—The addition of an inhibitor during the stripping of Zn from galvanized coatings will minimize the attack of the steel substrate. NEP (polethylinepolyamine) or $SbCl_3$ are two useful inhibitors.

8.3 Oxidized or corroded surfaces may be cleaned as described in Appendix X1.

~~8.~~

9. Mounting of Specimens

~~8.1There~~9.1 There are many instances where it will be advantageous to mount the ~~specimens~~specimen prior to grinding and polishing. Mounting of the specimen is usually performed on small, ~~flimsy,~~fragile, or oddly shaped specimens, fractures, or in instances where the specimen edges are to be examined.

~~8~~9.2 Specimens may be either mechanically mounted, mounted in plastic, or a combination of the ~~two can be used to provide optimum results.~~ two.

~~8.3~~9.3 *Mechanical Mounting*:

~~8~~9.3.1 Strip and sheet specimens ~~are frequently~~may be mounted by binding or clamping several specimens into a pack held together by two end pieces and two bolts. ~~Clamp mounting generally affords a means of rapid mounting with very good edge retention.~~

~~8.3.2The~~9.3.2 The specimens should be tightly bound together to prevent absorption and subsequent exudation of polishing materials or etchants.

~~8.3.3The~~9.3.3 The use of filler sheets of a softer material alternated with the specimen may be used in order to minimize the seepage of polishing materials and etchants. Use of filler material is especially advantageous if the specimens have a high degree of surface irregularities.

~~8~~9.3.4 Filler material *must* be chosen so as not to react electrolytically with the specimen during etching. Thin pieces of plastic, lead, or copper are typical materials that are used. Copper is especially good for steel specimens since the usual etchants for steels will not attack the copper.

~~8~~9.3.5 Alternatively, the specimens may be coated with a layer of ~~phenolic or~~ epoxy resin before being placed in the clamp in order to minimize the absorption of polishing materials or etchants.

~~8.3.6The~~9.3.6 The clamp material should be similar in composition to the specimen to avoid galvanic effects that would inhibit etching. The specimen will not etch if the clamp material is more readily attacked by the etchant.

~~8.3.7The~~9.3.7 The clamp should preferably be of similar hardness as the specimens to minimize the rounding of the edges of the specimens during grinding and polishing.

~~8~~9.3.8 Exercise care in clamping the specimen. Excessive clamping pressure may damage soft ~~specimens; however, good sealing is required to prevent absorption of polishing materials or etchants.~~ specimen.

~~8.4~~9.4 *Plastic Mounting*:

~~8.4.1Specimens may be embedded in plastic to protect them from damage and to provide a uniform format for both manual and automatic preparation. This is the most common method for mounting metallographic specimens. Mounting plastics may be divided into two classes—compression mounting and castable.~~

~~8.4.2When mounting specimens in plastic, exercise care in order to avoid rounding of specimen edges during the grinding operation. There are several methods available that prevent rounding. The specimens may be surrounded by hard shot, small rivets, rings, etc., of approximately the same hardness or, when using casting resin, a slurry of resin and alumina may be poured around the specimen to prevent rounding. The specimens may also be plated before mounting (see Section 9).~~

~~8.4.3~~

9.4.1 Specimens may be embedded in plastic to protect them from damage and to provide a uniform format for both manual and automatic preparation. This is the most common method for mounting metallographic specimens. Mounting plastics may be divided into two classes—compression and castable.

9.4.2 The choice of a mounting compound will influence the extent of edge rounding observed during the grinding and polishing operations. There are several methods available that minimize rounding. The specimen may be surrounded by hard shot, small rivets, rings, etc., of approximately the same hardness or, when using a castable resin, a slurry of resin and alumina may be poured around the specimen. The specimen may also be plated before mounting (see Section 10). Many mounting procedures result in sharp edges on the mount corners. The corners should be beveled to remove any plastic mounting flash.

9.4.3 *Compression Mounting*~~—Thermosetting plastics require the use of a mounting press providing heat (up to approximately 160°C) and pressure (up to approximately 30 MPa). *The finished mounts can be ejected hot but the best results are obtained when the finished mount is cooled under pressure.* There are three types of thermosetting compression mounting plastics used predominantly in the metallographic laboratory. Regardless of the resin used to compression mount specimens, the best results are obtained when (~~—*There are four types of compression mounting plastics used predominantly in the metallographic laboratory (see Table 2). These plastics require the use of a mounting press providing heat (140-180°C) and force (27-30 MPa). Thermosetting plastics can be ejected hot but the best results are obtained when the cured mount is cooled under pressure. Thermoplastic compounds do not harden until cooled and therefore should not be ejected while hot. Regardless of the resin used, the best results*

TABLE 2 Characteristics of Hot-Compression Mounting Compounds

Type of Compound	Characteristics
Acrylic	thermoplastic, cure time 10-15 min, optically clear, moderate shrinkage, low abrasion resistance, degraded by hot etchants
Diallyl phthalate[A]	thermosetting, cure time 5-10 min, opaque, minimal shrinkage, good resistance to etchants, moderate abrasion resistance
Epoxy[A]	thermosetting, cure time 5-10 min, opaque, very low shrinkage, good resistance to etchants, high abrasion resistance
Phenolic[A] (Bakelite)	thermosetting, cure time 5-10 min, opaque, moderate shrinkage, degraded by hot etchants, moderate abrasion resistance

[A] These compounds may be filled with wood flour, glass fiber or mineral particulate.

are obtained when (1) the ~~specimens are~~specimen is clean and dry, and (2~~) the cured mount is cooled under full pressure to below 30°C before ejection from the press.~~

~~8.4.3.1Wood-filled bakelite resins cure in 5 to 10 min, are relatively inexpensive, can be obtained in several colors, and are opaque. These resins have a tendency to pull away from the specimen leaving a crevice, which will trap liquids that later can smear, stain, and obscure a portion of the specimen.~~

~~8.4.3.2Diallyl phthalate resins are less likely to shrink and are more resistance to attack by etchants. They are more expensive than the phenolic resins with about the same hardness.~~

~~8.4.3.3Filled dry epoxy resins provide minimal shrinkage. Commercial resins intended for metallography are usually filled with hard material, minimizing edge rounding during preparation. These resins are the most expensive of the three types of thermosetting plastics. Cost can be reduced by first adding a layer of filled epoxy resin and filling up the remainder of the press cavity with phenolic resin.~~

~~8.4.3.4Resins are used in a similar fashion. Because of the adhesive characteristics of the resins, a mold release agent should be applied to the surface of the mold. Do not apply the release agent to the specimen. The specimen is placed in a heated mold face down (the surface to be ground). The appropriate amount of resin is poured over the specimen, the mold is closed, and pressure is applied. The pressure is released at the end of the cure, the mold opened, and the finished mount ejected. As noted in 8.4.3, shrinkage can be minimized by cooling to room temperature under pressure. Modern automated mounting presses can apply pressure and heat, time the cure, and cool the mount under pressure.~~

~~8.4.3.5Acrylic thermosetting resins produce transparent mounts. They require cooling under pressure. Heat and pressure must be carefully applied to avoid formation of "cotton ball" defects in the center of the mount.~~

~~8.4.4~~*) the cured mount is cooled under full pressure to below 40°C before ejection from the press. This will ensure minimal shrinkage gap formation.*

9.4.4 *Castable Plastics*—~~Castable resins are used at room temperature. Some may require an external heat source or applied pressure in order to cure. These resins consist of two or more components which must be mixed just prior to use. There are three kinds of castable plastics in common use:~~

~~8.4.4.1Acrylic resins consist of a powder and liquid, and cure rapidly (from 8 to 15 min) to a moderate hardness. These resins exhibit low abrasion resistance and a tendency to pull away from the specimen. They also tend to give off an unpleasant odor and enough heat during curing to alter the microstructure of some as-quenched steels.~~

~~8.4.4.2Polyesters consist of two liquids, and cure to form water-clear mounts with little heat evolution, low shrinkage, and low hardness. The cure takes 1 to 3 h and the mixing ratio is critical. They are more expensive than the acrylic resins.~~

~~8.4.4.3Epoxy resins have the best properties concerning transparency, heat generation, shrinkage, adhesion to the specimen, and hardness of the three castable resins. They are expensive. Cure times vary broadly, from 1 to 1½ h for some formulations to 4 to 8 h for others. Some formulations require cooling and others heating.~~

~~8.4.4.4The molds for castable plastics are simple cups that hold the resin until it cures. They may be reusable or not; the choice is a matter of convenience and cost. Handling castable resins requires care. They all can cause dermatitis. Manufacturers' recommendations for mixing and curing must be followed to obtain best results.~~

~~8.5~~*—Castable mounts are usually prepared at room temperature. Some may require an external heat source or applied pressure in order to cure. These resins consist of two or more components which must be mixed just prior to use. There are four kinds of castable plastics in common use (see Table 3).*

9.4.5 The molds for castable plastics are often simple cups that hold the resin until it cures. They may be reusable or not; the choice is a matter of convenience and cost. Handling castable resins requires care. They all can cause dermatitis. Manufacturers' recommendations for mixing and curing must be followed to obtain best results.

TABLE 3 Characteristics of Castable Mounting Compounds

Type of Compound	Characteristics
Acrylic	Cure time 8-15 min, moderate shrinkage, peak curing temperature can reach 90-120°C during polymerization, low abrasion resistance, opaque to transparent
Polyester-acrylic (quartz-filled)	Cure time 8-15 min, very low shrinkage, peak curing temperature can reach 90-120°C during polymerization, high abrasion resistance, opaque
Polyester	Cure time 30-60 min, high shrinkage, peak curing temperature can reach 90- 120 C during polymerization, moderate abrasion resistance, transparent
Epoxy	Cure time ½-20 h, very low shrinkage, good adhesion, low heat generation during polymerization, moderate abrasion resistance, low viscosity (good for vacuum impregnation), transparent