

1

3,187,444

SPECIMEN MOUNTING

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The present invention relates to means for preparing mountings for specimens to permit a scientific study thereof. More particularly, this invention is concerned with new and improved specimen mounting compositions, with new and improved methods for mounting, grinding, and polishing specimens contained in such mounting compositions, and with the new and improved specimen mounts provided by such compositions and methods.

A wide variety of scientific investigations require the preparation of suitable mountings for specimens which are to be observed or processed with scientific instruments and apparatus of various sorts. The science of metallography, for example, essentially involves the study of the structural characteristics or constitution of metals or alloys in relation to their physical and mechanical properties. Two significant phases of this study are known as macroscopic and microscopic examination. Macroscopic examination involves the visual observation of the rather gross structural details of a metal or an alloy either by the unaided eye or with the assistance of low power microscopes or binoculars. The more important of the two phases, however, is the microscopic examination of the prepared metal specimen. Generally, this is accomplished by employing magnifications with optical instruments of from 25 times to as high as 3,000 times. It has been through the diligent microscopic study of metals that many perplexing problems of physical metallurgy have been solved.

The preparation of specimens, such as metallographic specimens, for example, for a microscopic study remains a skilled technical art. In general, the procedure for such a study comprises first obtaining a flat polished surface on the specimen by means of grinding with a series of abrasive papers containing abrasive particles of decreasing grit size, or by grinding on suitable abrasive laps followed by a fine finishing step employing an abrasive paper. Final polishing is generally accomplished on cloth covered lap wheels. Ultimately, these grinding and polishing operations provide a flat, scratch free, mirror like surface on the specimen. This is, of course, the required condition of the specimen before it may be chemically etched and the metallographic structure appropriately revealed.

Since a number of specimens which are to be microscopically examined are in many cases either too small or too awkwardly shaped to handle conveniently during grinding and polishing, they must be appropriately mounted in a suitable material. In the past, a great number of mounting materials have been employed for mounting metallographic specimens, but unfortunately all have had their limitations. Examples of materials which have been used in the past are sulfur, woods metal, dental plasters, various plastics, and a variety of low melting alloys. However, a specimen mounting material possessing the required strength and solvent resistance which are necessary in the art of successful metallographic specimen examination and which will not mar or scratch the specimen as it is abraded during polishing has heretofore not been available in the art. Specifically, the materials possessing the required strength necessary to hold the specimen in place during the grinding and polishing operation would break off during the polishing to cause scratches on the surface of the metallographic specimen which was

2

to be examined. In addition, it has been impossible, or at least extremely difficult to mount materials such as, for example, foam metals, graphite, porous materials or specimens with cracks and voids, with the mounting materials heretofore employed in the art.

Accordingly, it is the primary object of the present invention to provide a new and improved means for mounting a specimen to permit a scientific study thereof.

Another object of the present invention is to provide a new and improved specimen mounting composition.

A further object of the present invention is to provide a new and improved method for mounting a specimen.

Yet another object of the present invention is to provide a new and improved specimen mount.

Briefly stated, these and other objects which will appear hereinafter may be attained in accordance with the present invention by preparing a new and improved specimen mounting composition comprising from about 10 to about 30 parts by weight of powdered abrasive material and from about 80 to about 120 parts by weight of a curable resin.

The new and improved method for mounting a specimen in accordance with the present invention comprises the steps of admixing from about 10 to about 30 parts by weight of a powdered abrasive material with from about 80 to about 120 parts by weight of a curable resin to form a homogeneous mixture, positioning the specimen in a receptacle, adding the mixture to the receptacle to envelop said specimen, and curing the resin to provide a solid matrix body containing the specimen.

The new and improved method for preparing a specimen mount for observation in accordance with the present invention comprises the steps of admixing from about 10 to about 30 parts by weight of a powdered abrasive material with from about 80 to about 120 parts by weight of a curable resin to form a homogeneous mixture, positioning the specimen in a receptacle, adding the mixture to the receptacle to envelop the specimen, curing the resin to provide a solid matrix body containing the specimen, and grinding a surface of the solid matrix body to expose at least a portion of the specimen.

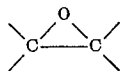
As an article of manufacture, the new and novel mount of the present invention comprises a solid matrix body comprising from about 80 to about 120 parts by weight of a cured resinous material having dispersed therein from about 10 to about 30 parts by weight of an abrasive powder, and a specimen embedded in said solid matrix body.

In general, a wide variety of abrasive materials may be employed in accordance with the present invention. Among the abrasive materials which are preferred in the grinding and polishing of metallographic specimens, however, are those having a Knoop hardness value of at least 900, such as, for example, chromia, zirconia, beryllia, topaz, garnet, tungsten carbide alloys, zirconium boride, titanium nitride, tungsten carbide, tantalum carbide, zirconium carbide, alumina, beryllium carbide, titanium carbide, silicon carbide, aluminum boride, boron carbide, boron nitride, diamond and other hard compounds of this nature.

Depending upon a specific requirement, the particle size of the powdered abrasive materials used in accordance with the present invention may vary within wide limits. However, abrasive powders having a particle size of 100 mesh or less have generally proven to be satisfactory in the preparation of most metallographic specimens with particles of less than 0.1 micron in size being preferred.

The curable resin used in accordance with the present invention may, in general, comprise any resinous material capable of being cured into a solid, thermosetting mass.

Among the preferred materials of this type, however, are the epoxy materials having the grouping



Examples of such resins are given in U.S. Patents 2,324,483 and 2,444,333 to Castan, and British Patents 518,087 and 579,698. Generally, the epoxy resins described therein are the reaction products of an epichlorohydrin such as epichlorohydrin and a phenol having at least two phenolic hydroxy groups such as bis-(4-hydroxyphenol)-2,2-propane. U.S. Patents 2,494,295 to Greenlee, 2,500,600 to Bradley, and 2,511,913 to Greenlee further describe epoxy resins which may be used in conjunction with the present invention. The epoxy resins used therein have more than one epoxy group per molecule and may be obtained by reacting a polyhydroxy alcohol or phenol such as hydroquinone, resorcinol, glycerine, condensation products of phenols with ketones, such as, for example, bis-(4-hydroxyphenol)-2,2-propane with epichlorohydrin. Such epoxy resins are sold under the name of Epon by Shell Chemical Corporation, under the name Araldite by the Ciba Company, as Epi-Rez resin by Devoe-Raynolds, and ERL resins by the Bakelite Company. The data given below for such resins is representative.

TABLE 1

Epoxy Resin	Epoxide Equivalent	Melting Point, ° C.
Epon 828.....	192	9
Epon 834.....	225-290	20-28
Epon 1001.....	450-525	64-76
Epon 1004.....	905-985	97-103
Epon 1007.....	1,600-1,900	127-133
Epon 1009.....	2,400-4,000	145-155
Epon 1062.....	140-165	Liquid
Epon 1064.....	300-375	40-45
Araldite 6010.....	192	Liquid
Araldite 6020.....	200-205	Liquid
ERL-2774.....	175-200	Liquid
Epi-Rez 510.....	175-200	Liquid

As is well known to those skilled in the art, such epoxy resins may be cured to a thermosetting infusible mass by the addition of a variety of curing agents, such as amines, for example, diethylene triamine, dicarboxylic acid anhydrides, and boron trifluoride-amine complexes.

In order that those skilled in the art may better understand how the present invention may be practiced, the following example is given by way of illustration and not by way of limitation. All parts and percentages are by weight unless otherwise noted.

Example 1

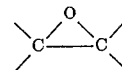
160 grams of powdered alumina (Linde B polishing powder) of size less than 0.1 micron were added, with stirring, to 800 grams of ERL-2795 resin, a bis-phenol-A-epichlorohydrin diluted with butyl glycidyl ether, and the stirring continued until a smooth, homogeneous slurry was obtained. 96 grams of a diethylene triamine curing agent (Hysol hardener #C1) were added to this slurry, and the resulting mixture allowed to set until a thermal reaction was initiated.

The interior portions of brass molds having an inside diameter of 1¼" and a height of about 1" were then coated with a silicone release agent (Dow Corning 7 compound), and the molds placed on film sheets coated with "Spraylat" manufactured by the Spraylat Corporation, New York, N.Y.

The specimens which were to be examined were then placed into the molds, and the resin-abrasive slurry poured thereover to fill the mold and to completely envelop the specimen. After standing for about 2-3 hours, the resin had cured and the formed mounts were removed from the molds and heated for several minutes at about 200° F. to insure completion of curing. Thereafter, each mounting

was ground until a suitable surface of the specimen was revealed which surface was then polished with finishing paper to provide a mirror like finish ready for etching and observation. A slight "flaking" of the abrasive occurred during the grinding and polishing steps that assists in obtaining a high degree of polish on the specimen surface being prepared.

While the above example has been performed with only a single curable resin and a single abrasive material, it should be understood that from 80 to 120 parts by weight of any curable resin, and preferably a resin containing the grouping



may be admixed with from about 10 to about 30 parts by weight of a powdered abrasive material to provide the new and improved specimen mounting composition in accordance with the present invention. In addition, although the example illustrates the use of a specific amount of a curing catalyst, it should be understood that varying amounts of curing catalyst may be employed depending upon the specific resin and the specific catalyst chosen. Of course, in certain instances it may be advantageous to cure the resin in the absence of a catalyst, such as for example by heating. Preferably, however, from 0.1 to over 10 percent by weight of catalyst, based upon the weight of the resin, may be employed, and the mixture heated to from 150-350° F. to assure a complete cure of the resin.

As will be appreciated by those skilled in the art, the present invention provides a new and distinctly improved means for preparing a specimen mount which aids rather than harms the polishing of the specimen. In particular, the technique employed eliminates the need for costly mounting presses and saves considerable "mounting time." Moreover, no limit is imposed upon the size of the specimens that may be mounted in accordance with the invention. As noted above, the new and improved mounting composition of the present invention provides a particularly strong matrix in which the specimen is to be contained, which matrix is exceptionally heat and solvent resistant. Also, the nature of the mounting composition of the present invention is such that it may be used to mount specimens otherwise not mountable, such as, for example, foam metals, graphite, porous materials, and specimens with cracks and voids, by employing a vacuum impregnation technique.

It should be understood, that although the new and improved mounting means of the present invention have particular utility in the mounting of metallographic specimens, they may also be advantageously employed in the mounting of a variety of specimen materials. Further, it should be understood that the present invention is not to be considered limited to any of the specific embodiments herein described, but may be used in other ways without departure from the spirit of the invention and the scope of the appended claims.

What is claimed is:

1. The method of preparing a specimen mount for observation which comprises the steps of admixing from about 10 to about 30 parts by weight of a powdered abrasive material having a Knoop hardness value of at least 900 with from about 80 to about 120 parts by weight of a curable resin to form a homogeneous mixture, positioning said specimen in a receptacle, adding said mixture to said receptacle to envelop said specimen, curing said resin to provide a solid matrix body containing said specimen, and grinding a surface of said solid matrix body to expose at least a portion of said specimen.

2. The method for preparing a specimen mount for observation which comprises the steps of admixing from about 10 to about 30 parts by weight of a powdered abrasive material having a Knoop hardness value of at least

5

900 with from about 80 to about 120 parts by weight of a curable resin to form a homogeneous mixture, coating the interior surface of a receptacle with a release agent, positioning the specimen to be observed in said receptacle, adding said mixture to said receptacle to envelop said specimen, curing said resin to provide a solid matrix body containing said specimen, and grinding a surface of said solid matrix body to expose at least a portion of said specimen.

3. An improved specimen mount comprising a solid matrix and a specimen, the matrix comprising a mixture of from about 10 to 30 parts by weight of a powdered abrasive material having a Knoop hardness value of at least 900, with from about 80 to about 120 parts by weight of a curable resin, the specimen having a ground exposed surface suitable for microscopic observation.

6

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