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METHOD OF MAKING A SPECIMEN ASSEMBLY

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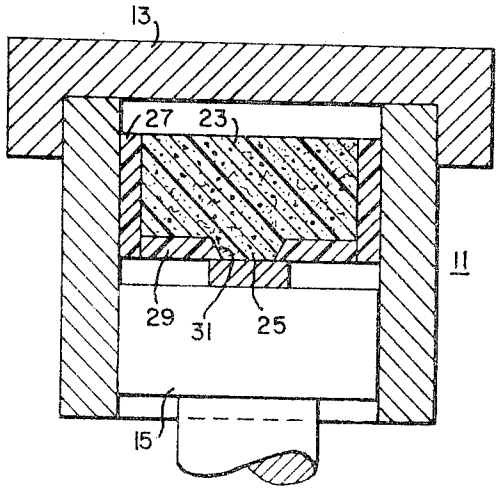

Fig.1

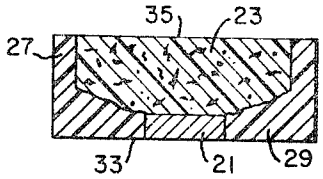

Fig.2

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METHOD OF MAKING A SPECIMEN ASSEMBLY

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4 Claims. (Cl. 264—266)

This application is a division of application Serial No. 125,442, filed July 20, 1961.

This invention relates to the etching and polishing art and has particular relationship to specimen assemblies or holders for etching with an electric discharge, or cathodic-vacuum etching as it is called, or for electrolytic etching or polishing or the like. Patent 3,097,154 granted July 9, 1963 to John E. Eck and Francis M. Cain, Jr. is incorporated in this application by reference.

The Eck-Cain application shows a cathodic-vacuum etcher in which the specimen holder disclosed in this application is used. In the operation of the Eck-Cain etcher the specimen acts as the cathode of an electric-discharge in gas (usually an inert gas) at a low pressure which may be as low as 1 micron in special situations but is usually between 20 and 300 microns. The specimen is mounted with its surface exposed to the low-pressure atmosphere. It is essential that the etching discharge be concentrated on the specimen and facilities must be provided for impressing an electrical potential between the specimen and the anode of the etcher. It is an object of this invention to provide a method of making a specimen assembly which shall expose only a small part of the surface of the specimen to the etching electric discharge and shall also provide effective means for impressing an electrical potential on the specimen. It is also an object of this invention to provide an article combined in the assembly in the practice of this method.

In accordance with the teachings of the prior art the specimen is embedded in an insulator and, when the form of the specimen permits, the electrical connection is effected to the surface of the specimen opposite to the surface to be etched by exposing this surface through the insulator. Where the form of the specimen does not permit it is necessary in accordance with the teachings of the prior art to bore a hole through the insulator and insert a connection. While etching has been successfully performed with such specimen assemblies more effective connecting means is desirable and it is an object of this invention to provide a method for making a specimen assembly with which highly effective electrical connection can be made and which shall be of such structure as to concentrate the electrical discharge on the specimen and an article, constituting a part of this assembly, which shall lend itself uniquely to the practice of this method.

In accordance with this invention in its broader aspects the specimen is embedded in a block of a moldable electrically conducting material. A typical such material may consist of a mixture of two parts of a phenolic condensation powder (Bakelite) with a mica filler and/or wood-flour filler or the like and one part aluminum powder, 60 to 100 mesh.

Such a block establishes firm electrical contact with the specimen. Electrical potential may be impressed between the specimen and the anode by a connection to the block. In accordance with the broader aspects of this invention a specimen assembly consisting of the block and specimen only is provided. Such an assembly may be made by mounting the specimen in a die encircled by a sleeve, inserting the conducting powder mixture in the sleeve, removing the sleeve and applying heat and pressure.

This assembly has disadvantages particularly where, as is usually the case, the exposed specimen surface is small and is surrounded by the surface of the conducting block. In this case the discharge is not concentrated but is diffused over the exposed surface of the specimen and the contiguous surface of the block. In addition, the sleeve adjacent this contiguous surface of the block, which encircles the assembly and which is composed of an insulator such as Bakelite, is eroded by the adjacent discharge. The products of the erosion are deposited on the exposed surface of the specimen rendering the specimen unsuitable for crystallographic studies. In the case of electrolytic etching such an assembly suffers from the disadvantage that the flow of current into the specimen is not adequately concentrated.

In accordance with an important specific aspect of this invention an assembly is made up by compressing together at an elevated temperature a premold of the moldable electrically conducting material, an insulator enclosing the premold and the specimen. To make this assembly a block of moldable conducting material is premolded with the material in the premold left uncured. For this purpose the mixture of phenolic condensation powder, mica, and/or wood flour or the like and aluminum is cold compressed at a pressure such as to produce a solid but not to cure or set. A typical pressure is about 12,000 pounds per square inch. The block is then mounted in a die with the specimen with a portion of the surface of the block encased in an uncured insulator similarly cold pressed. Preferably another small portion of the block extends through the insulator and is in contact with the specimen. The block, insulator and specimen are then compressed and cured. The result is an assembly in which the specimen is embedded in the block in firm electrical contact with the block and with its exposed surface flush with the insulator. The surface of the block which was not encased in the insulator is also exposed so that electrical connection may be made to the specimen through the block.

The block material may be the phenolic-condensation aluminum material described; the insulator may also be a phenolic condensation product. Typical pressure for compression and curing is 4200 pounds per square inch. The treating temperature is about 250° F.

The novel features considered characteristic of this invention are discussed generally above. The invention itself, both as to its organization and as to its method of operation, together with additional objects and advantages thereof will be more thoroughly understood from the following detailed description taken in connection with the accompanying drawings in which:

FIGURE 1 is a diagrammatic view in longitudinal section showing apparatus for practicing this invention with the components of the specimen assembly in a position to be formed; and

FIG. 2 is a view in longitudinal section of a specimen according to this invention.

FIG. 1 shows a press 11 in which the specimen assembly is formed. This press includes a chamber 13 of generally cylindrical form in which a ram 15 is slidable.

The parts of the assembly which are formed include a specimen 21 which may be a circular cylinder or may have conical or any other form. The parts also include a premold 23 of moldable electrically conducting material. This premold is generally circularly cylindrical but has a projection 25 extending centrally from one of its bases. The premold is preformed by molding from the conducting material but is not cured. Also included is a cylindrical shell 27 of uncured thermosetting insulating material such as a phenolic-condensation product and a plate 29 of like material. The shell 27 has an external diameter such as to slide readily in the chamber 13 and an

inner diameter such that the premold may be readily slid into the cylinder. The plate **29** has a central opening **31** which is dimensioned to conform to the projection **25**. The tip of the projection **25** extends through the opening slightly.

In the practice of this invention the specimen **21** is disposed centrally on the ram **15** and the premold **23**, and plate **29** is disposed in the shell **27** and the parts so combined are disposed on the top of the specimen **21** with the projection **25** in engagement with the specimen. The parts are then compressed by the ram and the temperature is raised to set or cure the moldable material. An assembly as shown in FIG. 2 is thus formed.

Usually the operator of an etcher or polisher will buy the assembly consisting of the uncured moldable conducting premold **23** encased in the uncured-insulating shell **27** as shown in FIG. 1. The presses **11** are of standard dimensions and are readily available. The premolds are dimensioned to fit the standard presses. The user of the composite premold can mount his specimens readily in the standard presses.

In this assembly the specimen **21** is embedded in the premold **23**. The exposed surface **33** of the specimen is flush with the insulator plate now also partly embedded in the premold. The premold is also molded to the shell **27** and only the face **35** opposite to the specimen **21** is exposed. The cathode connection to the etcher may be made to the face **35**.

While the assembly shown in FIG. 2 has marked advantages it may take different forms than that disclosed. For example, for etchers, which could be used where the operating pressure is very low (1 micron), in which the side walls of the assembly are encased by a protective insulating shell which forms part of the etcher, the shell **27** may be dispensed with. In situations in which the pressure is substantially higher than 1 micron so that the mean-free-path of the ions of ionizable gas is small the insulating shell **27** is indispensible. It is desirable but not indispensible that the exposed surface **35** shall be opposite the specimen. It is conceivable that another part of the premold surface may serve for connection to the etcher supply. The assembly may also have other than cylindrical form.

While a preferred embodiment of this invention has been disclosed herein, many modifications thereof are feasible. This invention is not to be restricted except insofar as is necessitated by the spirit of the prior art.

We claim:

1. The method of producing an assembly for subjecting a specimen to etching and polishing and the like comprising placing a hollow uncured thermosetting insulator means over a portion of the external surface of an uncured premold of thermosetting moldable electrically conducting material, a first portion of said external surface being exposed and having said insulator means adjacent thereto and a second portion of said external surface being also exposed, placing said specimen with one of its faces in engagement with said adjacent insulator means and said first portion of said surface and compressing said premold, insulator means and specimen together, while curing said premold and insulator means, until said specimen is embedded in said premold with the face of said specimen substantially flush with the adjacent surface of said insulator means and with said second portion of said external surface of said premold remaining exposed.

2. The method of producing an assembly for subjecting a specimen to etching and polishing and the like comprising placing uncured thermosetting insulator means over a first portion of the surface of an uncured premold of thermosetting moldable electrically conducting material, said premold having a projection extending from said first portion of its surface and said insulator means having an opening dimensioned to be coextensive with said projection and being placed over said portion of said surface so that said opening is coextensive with said projection, placing said specimen with one of its faces in engagement with said projection and the portion of the surface of said insulator means bounding said opening, said premold, specimen and insulator means being placed so that there is contact between said specimen and said premold through said projection, and compressing said premold, insulator means and specimen together, while curing said premold and insulator means, until said specimen is embedded in said premold with the face of said specimen substantially flush with the adjacent surface of said insulator means and with a second portion of the surface of said premold exposed.

3. The method of producing an assembly for subjecting a specimen to etching and polishing and the like comprising placing uncured thermosetting insulator means over a portion of the surface of an uncured premold of thermosetting moldable electrically conducting material with a portion of said premold extending through said insulator means, placing said specimen with one of its faces in engagement with a portion of the surface of said insulator in the region where said portion extends through said insulator so that said specimen is in contact with said portion, and compressing said premold, insulator means and specimen together, while curing said premold and insulator means, until said specimen is embedded in said premold with the face of said specimen substantially flush with the adjacent surface of said insulator means and with another portion of the surface of said premold exposed.

4. The method of producing an assembly for subjecting a specimen to etching and polishing and the like comprising partly encasing an uncured premold of thermosetting moldable electrically conducting material in a shell of uncured thermosetting insulator material, said shell in one region thereof bounding an unencased portion of said premold, placing said specimen adjacent said last-named portion and adjacent to said region of said shell, and compressing said premold, insulator material and specimen together, while curing said premold and insulator material, until said specimen is embedded in said material with the face of said specimen substantially flush with the adjacent surface of said insulator material and with another portion of the surface of said premold exposed.

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