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3,549,439

CHEMICAL LAPPING METHOD

Filed Sept. 15, 1967

2 Sheets-Sheet 2

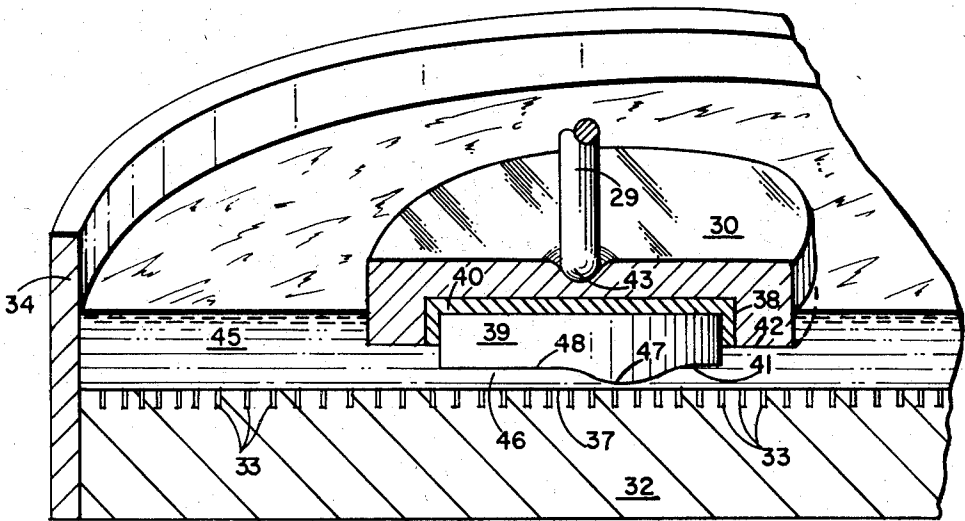


FIG. 2

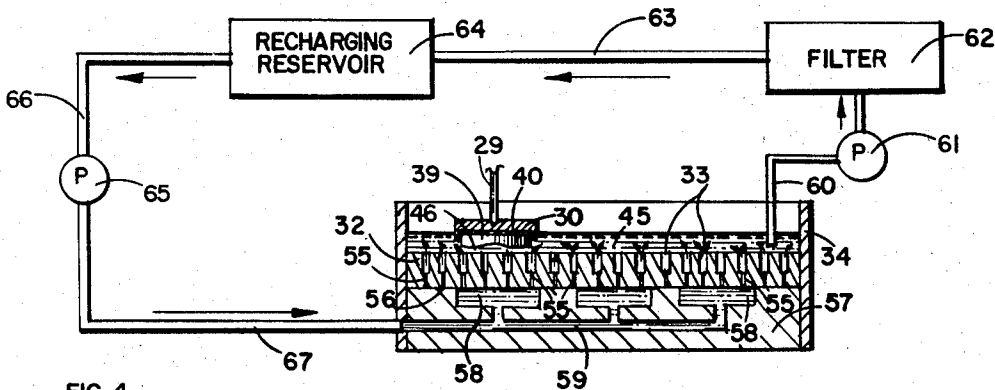


FIG. 4

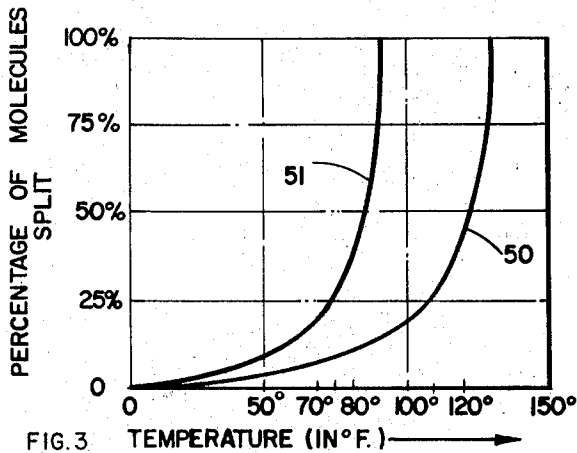


FIG. 3

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2 Sheets-Sheet 1

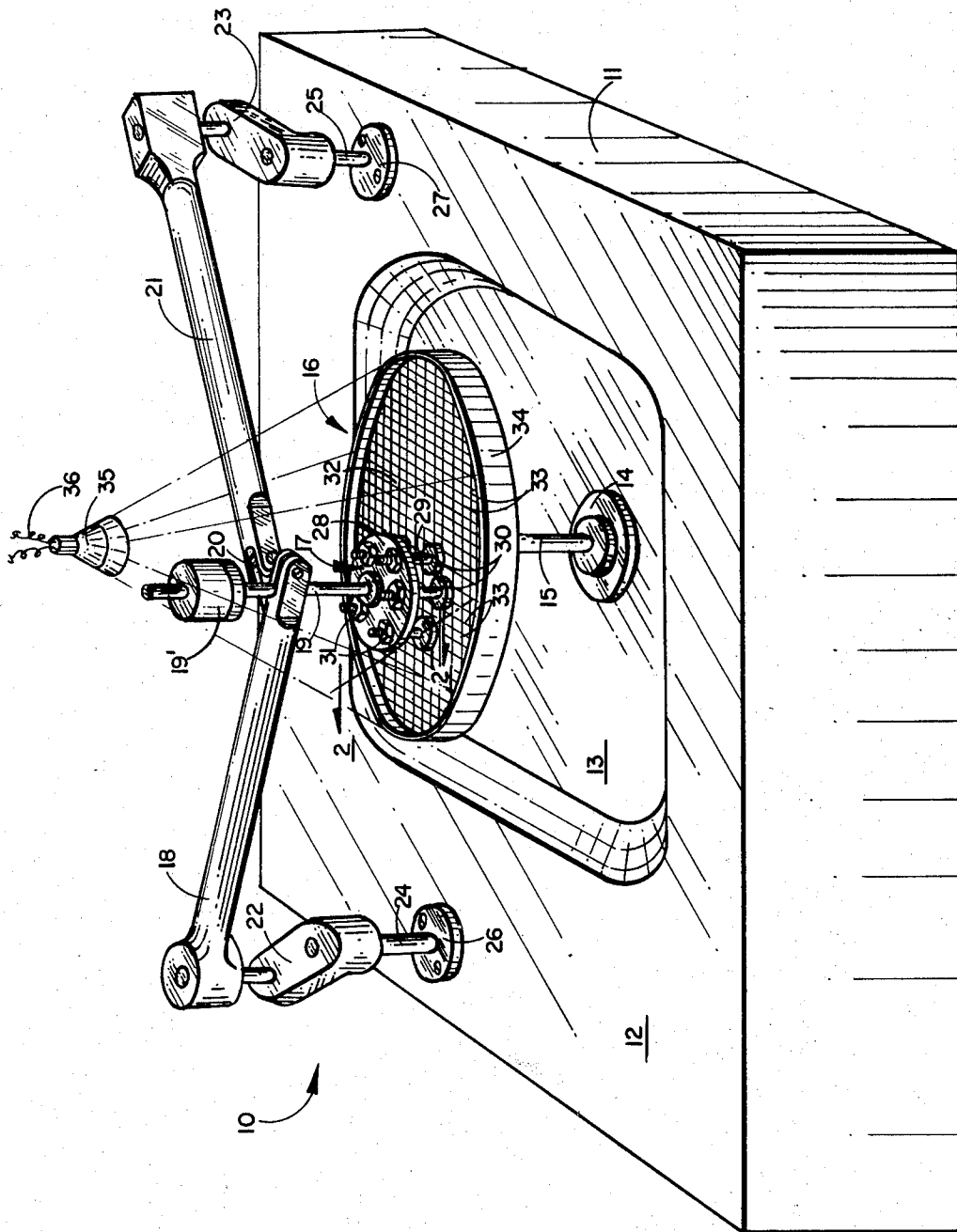


FIG. 1

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CHEMICAL LAPPING METHOD

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4 Claims

ABSTRACT OF THE DISCLOSURE

A process and apparatus for chemically lapping a specimen of quartz, ferrite, MgO, and the like. The surface of a graphite lapping plate is covered with a chemical lapping reagent comprising an organic salt which when heated splits to provide a molecule reactive with the specimen. Friction caused by relative motion of the specimen and the lapping plate causes heating of a film of the compound beneath the specimen, and the molecules thus provided preferentially lap the specimen. As this etching progresses, the specimen takes on the same curvature as the surface of the lapping plate. The lapping reagent may be maintained under positive pressure to ensure the presence of a continuous film of lapping reagent between the specimen and the graphite plate.

BACKGROUND OF THE INVENTION

(1) Field of the invention

The present invention relates to a chemical lapping method and apparatus. More particularly, the invention relates to a lapping process utilizing a chemical lapping reagent which at ambient temperature is nonreactive with specimen being lapped. When heated by friction resulting from relative motion of the specimen and the lapping plate, the lapping reagent splits to provide a molecule which etches the specimen.

(2) Description of the prior art

In the past, two basic processes have been used to lap quartz, MgO, ferrite and the like. Most commonly, a mechanical abrasive such as a diamond paste was used in conjunction with a rotating lapping table. The specimen to be lapped, mounted in an appropriate holder, was held against the rotating table and ground or lapped by the abrasive until the specimen surface conformed to the curvature of the table. While this technique achieved a specimen of desired curvature, the process had the disadvantage that the specimen surface was marred by scratches caused by the abrasive lapping compound. These scratches, although quite small (corresponding generally to the dimension of the abrasive particles used), were considerably larger than molecular size.

Alternatively, chemical etching has been used in the past to achieve a very smooth specimen surface. Typically, the specimen was immersed in a chemical etchant which reacted more or less uniformly with the entire surface of the specimen, providing a very smooth surface. Of course, if the specimen was not originally of the desired curvature, the etching did not provide this curvature. The chemical etching had the additional disadvantage that the etching was difficult to control and typically excess etching occurred at regions of the specimen which were scratched or had other discontinuities. Such excess etching occasionally even ruined the controlled surface curvature of the specimen.

One approach of the prior art toward overcoming these obstacles was to cover the surface of a lapping plate with a cloth soaked in an etchant. The specimen then was rubbed over the soaked cloth to accomplish controlled etch-

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ing of the specimen. This technique had a number of shortcomings. First, it was difficult to control the flatness of the cloth, and typically during operation the cloth would bunch up, distorting the curvature achieved. Moreover, the acid concentration at various portions of the cloth would become greater or less than at other locations. Consequently, uncontrolled etching of the specimen occurred, and often the specimen surface curvature achieved did not conform to the reference curvature of the lapping plate. Moreover, the etchant did not work-react preferentially on protruding regions of the specimen, but etched substantially uniformly over the entire specimen surface. Often this accentuated rather than reduced discrepancies in the smoothness of the specimen.

The present technique provides a chemical lapping technique and apparatus by means of which the advantages of both the chemical and mechanical lapping techniques may be achieved with none of the attendant disadvantages of either technique.

SUMMARY OF THE INVENTION

In accordance with the present invention, there is set forth a process and apparatus for chemically lapping a specimen. The apparatus comprises a rotatable lapping table atop which a specimen to be lapped is mounted in a holder. Generally ellipsoidal motion of the specimen holder is provided by a pair of connecting rods driven by rotating cranks.

In a preferred embodiment, the lapping table of the present invention comprises a plate of atomic reactor grade graphite having grooves in its upper surface. A retaining ring surrounds the lapping plate. The plate is covered with a chemical lapping reagent having the characteristic that at an ambient temperature the reagent is nonreactive with the specimen being lapped. At an elevated temperature, the lapping reagent splits to provide a molecule which is reactive with the specimen material. Sufficient heat to accomplish molecular splitting is realized by the friction resulting from relative motion of the specimen and the rotating lapping table. Chemical lapping occurs preferentially on those regions of the specimen which depart most from the desired curvature.

Chemical lapping in accordance with the invention provides a specimen surface which is chemically etched and hence free of scratches characteristic of an abrasive lap. Moreover, the specimen which is chemically lapped takes on a curvature corresponding to that of the reference curvature of the lapping plate.

Thus it is an object of the present invention to provide a chemical lapping process and apparatus.

Yet another object of the invention is to provide a process whereby a specimen is lapped by chemical reaction with a molecule split from a lapping compound as a result of friction generated heat.

A further object of the present invention is to provide a chemical lapping apparatus comprising a rotatable graphite lapping plate supporting a chemical compound and a specimen holder. Friction resulting from relative motion of the specimen and the plate causes molecular splitting of the compound producing a product which preferentially laps the specimen.

These and other objects and features of the present invention will become clear in conjunction with the following figures and description of the preferred embodiments which are illustrative of the invention.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a perspective view of a chemical lapping apparatus in accordance with the present invention. Evident in the figure is the rotatable, grooved lapping plate surrounded by a ridge for retaining a chemical lapping reagent atop the plate.

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FIG. 2 is an enlarged sectional view of a portion of the inventive chemical lapping apparatus as seen generally along the lines 2—2 of FIG. 1. The relative locations of the lapping plate, the specimen being lapped, and the chemical lapping reagent are evident in FIG. 2.

FIG. 3 shows a graph of the percentage of molecules split as a function of temperature for various typical chemical lapping reagents useful in conjunction with the present invention.

FIG. 4 is a schematic diagram of an alternative embodiment of the inventive chemical lapping apparatus, including means for maintaining the lapping reagent under positive pressure.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

An apparatus useful for practicing the inventive chemical lapping technique is illustrated in FIG. 1. As shown in FIG. 1, chemical lapping apparatus 10 comprises support member 11 having a planar upper table 12, the central depressed region of which forms sink 13. Extending upward through bushing 14 at the center of sink 13 is shaft 15, to which shaft is attached rotatable lapping table 16. Shaft 15 is driven by a motor (not visible in FIG. 1) beneath sink 13 so as to rotate lapping table 16 at a substantially constant rate.

Situated above lapping table 16 is specimen holder 17, which, as may be seen in FIG. 1, is attached to connecting rod 18 by means of shaft 19. Also attached to connecting rod 18, by means of flexible joint 20, is connecting rod 21. Motion is imparted to connecting rods 18 and 21 by cranks 22 and 23, respectively. Cranks 22 and 23 themselves are driven by motors (not shown) via shafts 24 and 25 which extend through table 12 via bushings 26 and 27.

It will be appreciated that as lapping table 16 rotates, shafts 24 and 25 also rotate, typically at rates different from that of table 16. Due to the offset action of cranks 22 and 23, and because connecting rod 21 is attached to connecting rod 18 somewhat offset from the end thereof, shaft 19 and hence specimen holder 17 will perform a somewhat ellipsoidal passage over the surface of lapping table 16. This motion insures that the specimen being chemically lapped will be exposed to the surface of table 16 at a constantly changing radial location.

Still referring to FIG. 1, it may be seen that specimen holder 17 itself comprises disc shaped member 28 which is attached to shaft 19 in such a manner that member 28 is free to rotate about shaft 19. Extending downward from disc shaped member 28 are a plurality of shafts 29 each terminating in a specimen holder 30. Shafts 29 are attached to disc shaped member 28 by means of connectors 31; shafts 29 may be either rigidly connected to member 28 or may be adapted to move vertically thereof. If adapted to move vertically, shaft 29 might also include a biasing spring (not shown in FIG. 1) to bias specimen holder 30 against the surface of lapping table 16.

Weight 19' is mounted on shaft 19 and provides a downward force on specimen holder 17, and hence on the specimens mounted in holders 30. As will be discussed in detail hereinbelow, the function of weight 19' is to insure appropriate friction between table 16 and the specimens during lapping.

In a preferred embodiment, lapping table 16 itself comprises lapping plate 32, the upper surface (designated 37 in FIG. 2) of which exhibits a flatness or curvature corresponding to that desired for the specimen being chemically lapped. For example, if a flat specimen surface is desired, the surface of lapping plate 32 itself should be flat. Similarly, if spherical shaped specimens are to be produced, lapping plate 32 itself should exhibit a spherical curvature.

In a preferred embodiment, lapping plate 32 is made of atomic reactor grade graphite. Further, lapping plate 32 (see FIG. 1) contains a criss-cross pattern of grooves 33 in its upper surface. As will be described more fully

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hereinbelow, grooves 33 insure that the liquid chemical lapping reagent (not shown in FIG. 1) will flow beneath the specimens being lapped. Preferably, the space between adjacent grooves 33 is small compared with the dimension of the specimen being lapped.

Surrounding graphite lapping plate 32 (see FIG. 1), is retaining ridge 34. Ridge 34 extends above the upper surface of lapping plate 32 a sufficient distance to insure that a liquid chemical lapping reagent, present on top of lapping plate 32 during normal operation, will not overflow into sink 13.

Lamp 35, typically of the infrared variety, may be suspended atop lapping apparatus 10 and used to provide heat to the liquid chemical lapping reagent situated atop lapping plate 32. Electricity for lamp 35 is provided via wires 36.

Additional details of lapping table 16 and specimen holder 30 are shown in the sectional view of FIG. 2, taken along line 2—2 of FIG. 1. Referring now to FIG. 2, it may be seen that specimen holder 30 contains a recessed region 38 in which specimen 39 (the specimen to be chemically lapped in accordance with the present invention) is mounted by means of wax 40. Wax 40 typically may be beeswax, sealing wax, or Dekotinski wax, such as is commonly used to hold optical components while they are being lapped. It is desirable that lower surface 41 (of specimen 39) extend somewhat below bottom surface 42 of specimen holder 30.

As shown in FIG. 2, the center of specimen holder 30 contains a depressed region 43 into which seats the lower end of shaft 29. This arrangement permits specimen holder 30 to rotate freely with respect to shaft 29.

Still referring to FIG. 2, note that graphite lapping plate 32 and ridge 34 form a reservoir for chemical lapping reagent 45. Lapping reagent 45, which preferably is liquid, flows under specimen 39 forming a film 46 between lower surface 41 (of specimen 39) and top surface 37 of graphite plate 32. The presence of film 46 is insured by grooves 33 which permit liquid chemical lapping reagent 45 to flow therethrough to positions underneath specimen 39. It is for this reason that the preferred spacing of grooves 33 is small compared with the width or radius of specimen 39.

Chemical lapping reagent 45 characteristically is a liquid or a paste which at ambient temperature is non-reactive with the material of specimen 39. However, chemically lapping reagent 45 has the further characteristic that when heated above a certain temperature, it splits molecularly to provide a molecule which itself is reactive with specimen 39.

The specific chemical composition of lapping reagent 45 will depend on the material which is being lapped. For example, if specimen 39 comprises MgO, lapping reagent 45 may comprise an alkyl aryl polyoxyether phosphate modified to a phosphate salt using an alkyl phosphate or pyrophosphate. An example of such a reagent is EMCOL T-36 manufactured by the Whitco Chemical Co., Los Angeles, Calif., as modified with Santosizer 141 organic phosphate ester. The resultant organic compound, when heated, splits to provide molecules of $[\text{HPO}_4]^-$. In turn, the $[\text{HPO}_4]^-$ reacts with the MgO of specimen 39 to produce magnesium phosphate. Of course, this reaction gradually etches away the surface of specimen 39.

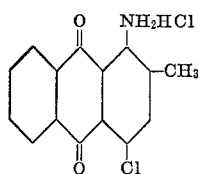
The chemical splitting of the modified phosphate salt is a function of temperature; this relationship is shown graphically by curve 50 of FIG. 3. Referring to FIG. 3, it is seen that for a typical chemical lapping reagent 45 (e.g., alkyl aryl polyoxyether phosphate) the percentage of molecules split (e.g., the percentage of phosphate salt molecules split to yield molecules of $[\text{HPO}_4]^-$) increases with the temperature of lapping reagent 45. For example, at 110° F., less than 20% of the molecules of a typical reagent may be split, whereas at 120° F. considerably more than 40 percent of the molecules may be split.

For lapping specimens 39 of material other than MgO, other lapping reagents 45 may be used. For example, if

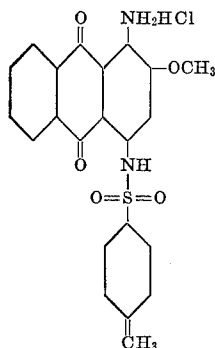
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quartz is to be lapped, lapping reagents 45 may comprise chromium tetrafluoride in an oil emulsion or alternatively, chromium tetrafluoride in a solution of glycerine or ethylene glycol. When heated, molecular splitting occurs providing fluorine atoms which are reactive with quartz.

Still another type of material which may be chemically lapped in accordance with the present invention is ferrite. For example, manganese doped ferrite such as $MnFe_2O_4$ may be etched using lapping reagent 45 comprising 1-amino-4-chloro-2-methylantraquinone hydrochloride salt having the following formula:



Alternatively, lapping reagent 45 may comprise N-(4-amino-3-methoxyanthraquinon-1-yl)-p-toluene sulfonamide hydrochloride having the formula:



In each case, an azoline type amide may be used to provide a nonaqueous solution. When heated, either of the above reagents split releasing molecules of HCl which react with the ferrite to form ferric chloride and water.

Clearly, chemical lapping reagents 45 in accordance with the present invention are not limited to the examples just stated. In general, lapping reagent 45 need only comprise an organic salt in a nonaqueous solution, which salt will split when heated to provide a molecule which is reactive with the material of specimen 39. Note that the percentage of molecular splitting as a function of temperature is not critical; this relationship may be that exemplified by curve 50 of FIG. 3 (which requires a temperature elevated somewhat above normal ambient to obtain considerable molecular splitting) or that exemplified by curve 51 of FIG. 3. Note that reagents 45 characterized by curve 51 show little molecular splitting at 70° F., but exhibit considerable molecular splitting at temperatures just a few degrees higher.

It should be evident from the foregoing that under ambient conditions lapping reagent 45 (see FIG. 2) will not react with the material of specimen 39. Thus, in contradistinction to the prior art, when specimen 39 is immersed in lapping reagent 45 essentially no etching of specimen 39 occurs. However, when specimen 39 (as mounted in specimen holder 30) experiences relative motion with respect to surface 37 of lapping plate 32 (see FIG. 2), film 46 of lapping reagent 45 is heated by the resultant friction between surfaces 37 and 47. This friction generated is sufficient to produce in film 46 of lapping reagent 45 sufficient molecular splitting to cause considerable reaction with specimen 39.

By way of example, if lapping reagent 45 exhibits molecular splitting as a function of temperature typified by curve 50 of FIG. 3, lapping compound 45 situated atop plate 32 may be heated to a temperature of about 110° F. by means of lamp 35 (see FIG. 1). Then, when table 16 is rotated and specimen holder 20 moved radially (by means of connecting rods 18 and 21), the resultant friction

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between specimen 39 and surface 37 of lapping plate 32 is sufficient to cause film 46 of lapping reagent 45 to be raised in temperature to about 120° F. As evident from curve 50 of FIG. 3, at 120° F. considerable molecular splitting occurs in reagent 45. Thus film 46 will contain a sufficient concentration of molecules reactive with specimen 39 to accomplish etching thereof.

Note that the lapping of specimen 39 will be preferential. That is, regions 47 which protrude furthest from specimen 39 will experience more friction with respect to surface 37 than will other regions (such as region 48). Consequently lapping reagent 45 directly beneath region 47 will have present a higher percentage of molecules split, and hence a higher concentration of molecules which can react with specimen 39, than will lapping reagent 45 present under region 48 of the same specimen. Thus, the regions of the specimen which differ the most from the desired curvature (i.e., the curvature of surface 37 of graphite plate 32) will be etched fastest. Clearly, as the chemical lapping progresses, surface 41 of specimen 39 will take on the desired curvature of surface 37 of plate 32.

Note that if lapping compound 45 is one whose percentage of molecular splitting as a function of temperature is typified by curve 45, then sufficient heat is generated by the relative motion of specimen 39 and lapping plate 32 to accomplish localized etching. Note further that the amount of friction experienced between surfaces 41 and 37 (of specimen 39 and plate 32 respectively) may be controlled by appropriate selection of the size of weight 19' (see FIG. 1). The heavier the weight, the greater the friction and hence the higher percentage of molecules split.

It should be appreciated from the foregoing that the inventive chemical lapping technique combines the advantages of both chemical etching and mechanical lapping while suffering the shortcomings of neither. For example, when a prior art chemical etchant was used, mere immersion of the specimen in the etchant would initiate etching. Thus, while a smooth surface was obtained, no control was possible over the flatness or curvature of the resultant surface. On the other hand, mechanical lapping permitted excellent control of the curvature of the specimen, but could not achieve as high a polish of the specimen as was possible with the chemical etchant.

In accordance with the present chemical lapping technique, no scratches typical of a mechanical abrasive are formed in the specimen. Nevertheless, since the degree of reactivity of the lapping reagent with the specimen material is dependent on the amount of friction developed during relative motion of the specimen and the lapping plate, the result is obtained that the specimen takes on the curvature of the plate. Moreover, any scratches present in the top surface of the lapping plate are averaged out as the specimen is moved over its surface, so that the smoothness of the graphite plate is not a factor limiting the achievable specimen smoothness.

To accomplish most efficient chemical lapping of specimen 39 it is necessary that a film 46 be present under the entire lower surface area of the specimen. As specimen 39 conforms more and more to the curvature of surface 37 of lapping plate 32, the average distance therebetween may decrease, with the result that lapping compound 45 may not flow freely beneath sample 39. The presence of grooves 33 in lapping plate 32 of course ameliorates this situation, since lapping reagent 45 can flow underneath specimen 39 via these grooves. However, in some instances, it may be desirable to place lapping compound 45 under a positive pressure to ensure a continuous film 46 of the lapping compound beneath specimen 39. An apparatus to accomplish this is shown in FIG. 4.

Referring now to FIG. 4, there is shown an alternative embodiment of the inventive chemical lapping apparatus, correspond to like numbered elements in FIGS. 1 and 2. Evident in FIG. 4 is lapping plate 32 having grooves 33 therein and surrounded by retaining wall 34. Also evident is specimen holder 30 and typical specimen 39 mounted

in specimen holder 30 by means of wax 40. Note however that in the embodiment of FIG. 4 lapping plate 32 contains a plurality of holes 55 extending downward from grooves 33 to lower surface 56 of lapping plate 32. Typically, holes 55 may be located at each of the intersections of crisscross grooves 33.

Still referring to FIG. 4, note that lapping table 32 is mounted on support member 57. In the embodiment illustrated, support member 57 contains several hollow recessed regions 58 adjacent its interface 56 with the lower surface of lapping table 32; regions 58 each are connected to bore 59.

Also evident in FIG. 4 as extending into lapping reagent 45 atop plate 32 is tube 60, which tube is connected to pump 61. Pump 61 functions to pick up liquid lapping reagent 45 via tube 60 and feed reagent 45 to filter 62. Filter 62 itself may comprise a settling tank or other type of filter and serves two functions. First, filter 62 may filter out the reaction product of the lapping compound and the material of specimen 39, and second, filter 62 may filter out spurious particles of dust, grit, or other material which may accumulate in lapping reagent 45.

Filter lapping reagent 45 from filter 62 (see FIG. 4) is fed via tube 63 to recharging reservoir 64. Reservoir 64 serves the purpose of maintaining chemical lapping reagent 45 at the desired concentration. It is evident that as lapping reagent 45 is molecularly split and the resultant molecules react with specimen 39, the concentration of unsplit molecules in reagent 45 is reduced. Within recharging reservoir 64 additional chemicals are added to lapping reagent 45 to bring its reactivity (i.e., its concentration of unsplit molecules) up to a desired value. Thus recharged, chemical lapping reagent 45 is pumped by pump 65 from reservoir 64 via tubes 66 and 67 into bore 59.

Due to the action of pump 65, chemical lapping reagent 45 passing through bore 59 into chambers 58 is under a positive pressure. This positive pressure forces lapping reagent 45 through holes 55 in lapping plate 32. This insures that a film 46 of lapping reagent 45 will be present underneath the lower surface of specimen 39 at all times.

Although the invention has been described and illustrated in detail, it is to be clearly understood that the same is by way of illustration and example only, and is not to be taken by way of limitation, the spirit and scope of the invention being limited only by the terms of the appended claims.

We claim:

1. A chemical lapping process using an optically flat and inelastic lapping plate having a pattern of grooves in the surface thereof, said process comprising the steps of:

injecting a chemical lapping reagent on the surface of said lapping plate and in said grooves for maintaining said reagent between the surface of said lapping plate and a specimen being lapped, said reagent being non-reactive with said specimen until the relative temperatures of the reagent, lapping plate, and specimen are above a certain temperature determined as a function of the type of reagent selected and the type of specimen being lapped,

moving said specimen over said surface to produce friction therebetween, said friction causing said relative temperatures to increase for enabling said specimen to be chemically lapped until the specimen assumes the reference curvature of said lapping plate.

2. A process for chemically lapping a specimen to conform to the surface curvature of an inelastic lapping plate, said process comprising the steps of:

injecting a reagent atop said lapping plate through apertures provided in said lapping plate whereby the reagent is maintained on the surface of said lapping plate under a specimen throughout the lapping process, said reagent being substantially non-reactive with said specimen below a first temperature, a substantial percentage of said reagent being split molecularly above said first temperature to produce molecules which are chemically reactive with said specimen, and

moving said specimen with respect to said lapping plate producing friction therebetween, said friction heating said reagent between said specimen and said plate to above said first temperature, the molecules produced thereby chemically lapping said specimen.

3. The process defined in claim 2 wherein said inelastic lapping plate comprises atomic reactor grade graphite for providing an optically flat surface.

4. The process defined in claim 3 wherein the surface of said lapping plate is grooved, said holes terminating under certain of said grooves, said grooves facilitating flow of said reagent between said specimen and said plate.

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JACOB H. STEINBERG, Primary Examiner

U.S. Cl. X.R.

156—19, 345, 6; 252—79.3, 79.4

UNITED STATES PATENT OFFICE
Certificate of Correction

Patent No. 3,549,439

December 22, 1970

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It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

In the Letters Patent only, the second sheet of drawings should be included.

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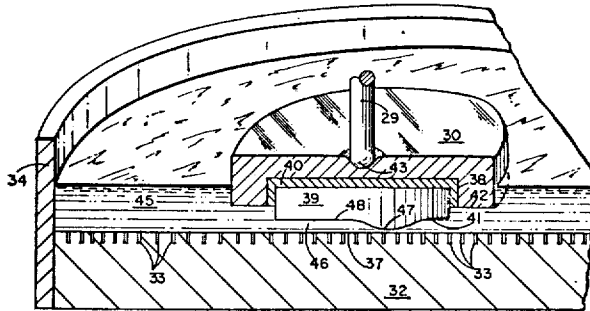


FIG. 2

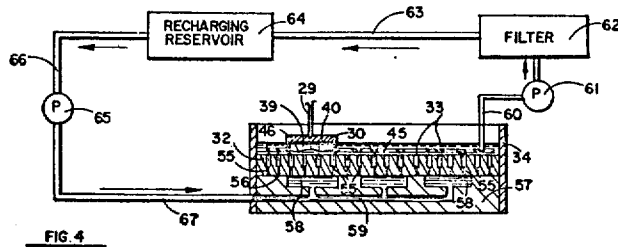


FIG. 4

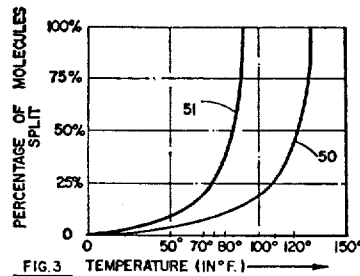


FIG. 3

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Signed and sealed this 17th day of August 1971.

[SEAL]

Attest:

EDWARD M. FLETCHER, JR.,

WILLIAM E. SCHUYLER, JR.,