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POLISHING MEANS AND METHOD

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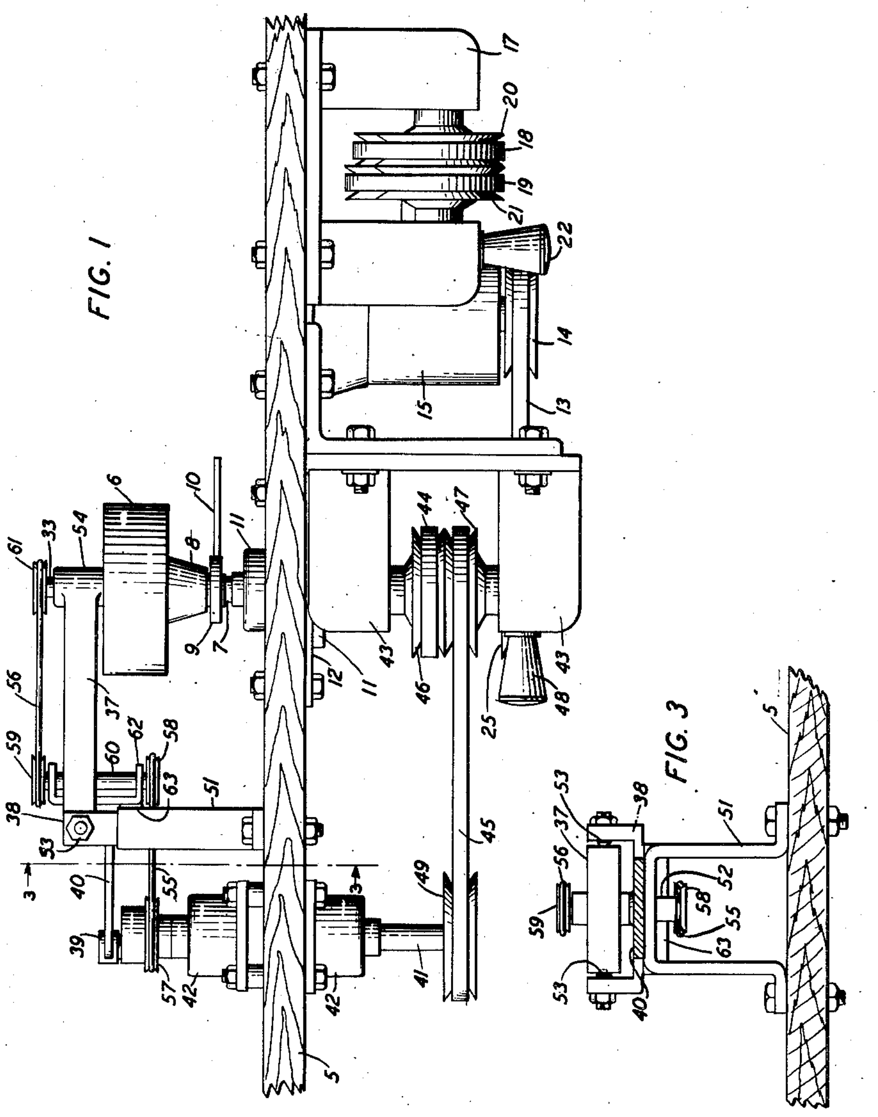

FIG. 1

FIG. 3

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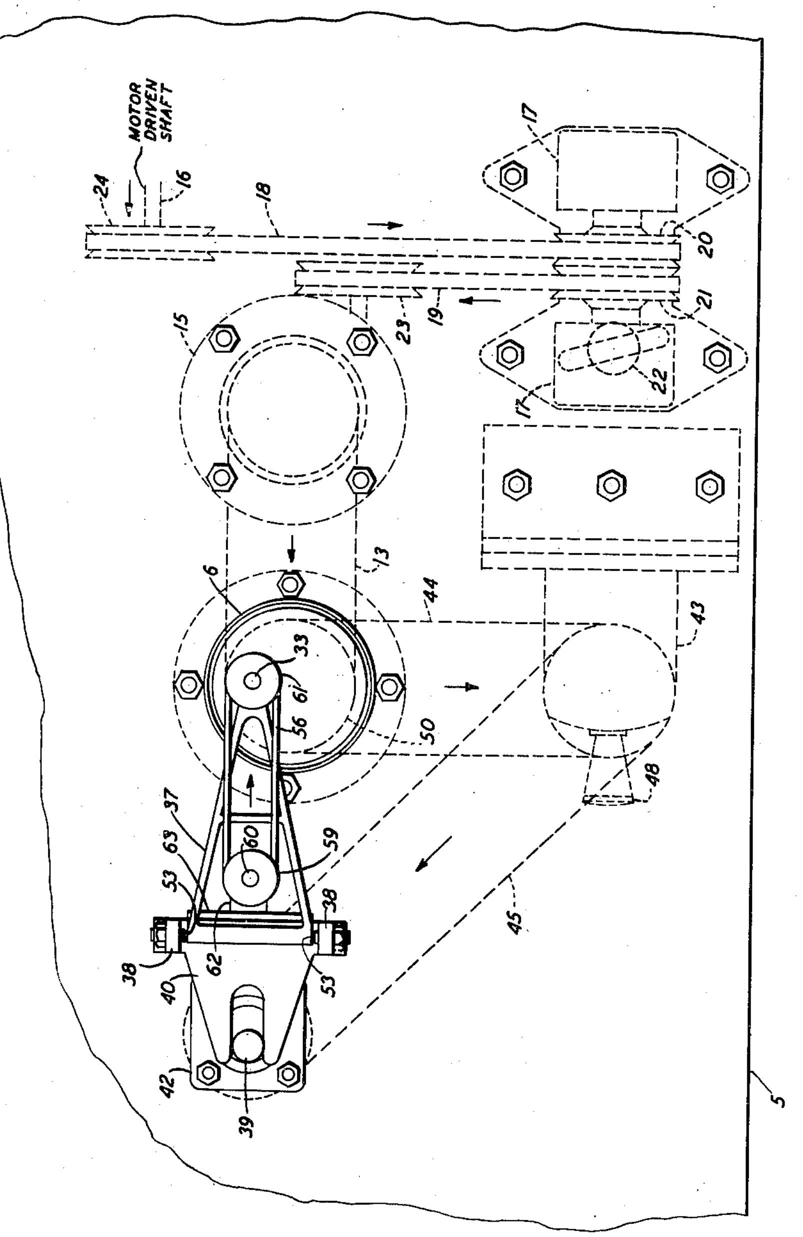

FIG. 2

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FIG. 4

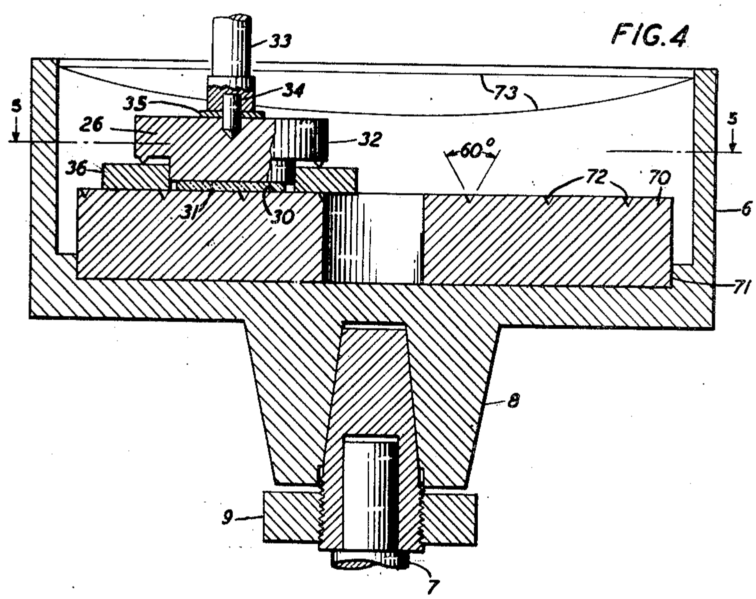
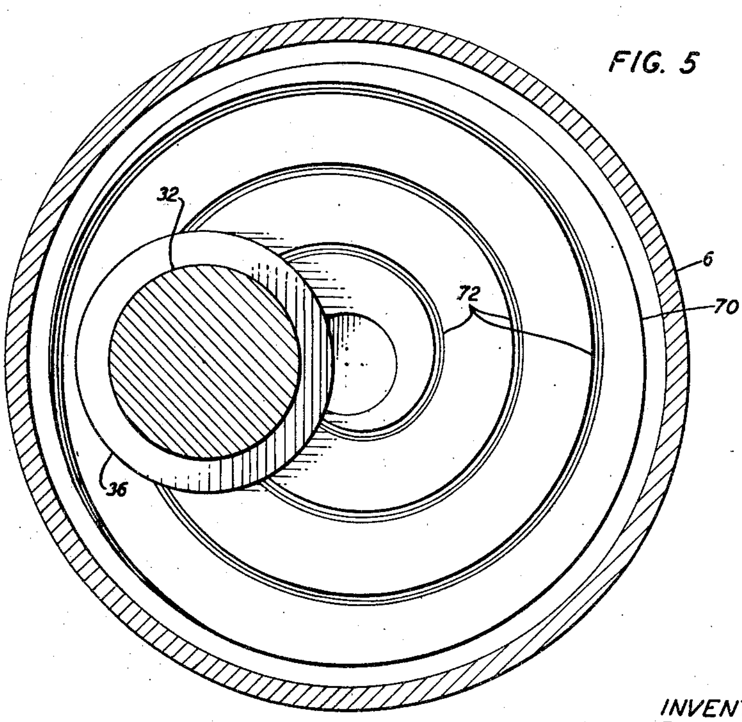


FIG. 5



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POLISHING MEANS AND METHOD

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13 Claims. (Cl. 51—124)

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This invention relates to method and means for polishing brittle crystalline material.

An object of the invention is to provide an improved device for polishing brittle crystalline material.

Another object is to provide an improved method of producing substantially optically flat surfaces on brittle crystalline material.

A further object is to provide an improved polishing technique for producing flat surfaces on pieces of highly pure solidified fused silicon.

In an example of practice an approximately flat surface of a slab of silicon cut from an ingot of solidified fused silicon of high purity is polished to produce a substantially optically flat surface. The method employed comprises producing relative movements of rotation and translation between a tin lap and the surface of the slab of silicon over a nonrepetitive path while the lap and slab are both immersed in water carrying an abrasive such as sapphirine and dioctyl sodium sulfosuccinate which is a water soluble chemically inactive detergent. The surface of the tin lap is provided with a spiral groove, the function of which is to cause a continual flow of abrasive charged water from the center of the lap toward the periphery to continually carry away the particles of silicon removed from the surface of the slab and bring in fresh abrasive. In carrying out the method a rotatable upright cup mounted on a vertical shaft is provided with a tin lap having a flat ground surface which has a spiral groove concentric with the shaft. The tin lap is centrally positioned in the bottom of the cup with the ground surface facing upward. A metal disc with the slab of silicon cemented to one face thereof rests on the grooved surface of the tin lap with the exposed surface of the slab in contact with the lap. By means of a second rotatable shaft, the lower end of which rests in a recess in the metal disc, the silicon slab is given a reciprocal motion of translation across the lap and a motion of rotation about a vertical axis. The cup is filled with a liquid to a depth sufficient to immerse both the silicon slab and the lap while the cup and lap are rotating during the polishing operation. The liquid bath comprises distilled water containing levigated sapphirine, 1/100 per cent dioctyl sodium sulfosuccinate solution and 1/5 per cent potassium bichromate. The pressure for polishing is about 5 pounds per square centimeter. The polishing is accomplished by a true abrasive action which is effective because the sapphirine particles are

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extremely small and of the proper hardness to produce on silicon a highly polished substantially optically flat surface. The combination of the dioctyl sodium sulfosuccinate with the very fine polishing material reduces the surface tension of the water and increases the effectiveness of the abrasive.

An advantage of this invention is that a substantially optically flat surface is produced on the slab instead of a lemon peel effect which is produced by a buffing method of polishing. Another advantage is that the surface being polished faces downward which facilitates removal of the waste particles of silicon disengaged from the surface during the polishing operation. By the method and means of this invention silicon slabs can be polished to a flatness of at least one-quarter wavelength of green light which is considerably better than can be obtained by any prior method known to applicant.

A more detailed description of this invention will now be given having reference to the accompanying drawings.

Fig. 1 is a side view of a polishing machine embodying this invention.

Fig. 2 is a plan view of the machine of Fig. 1.

Fig. 3 is a fragmentary view in section of the machine of Fig. 1 along the line 3—3 looking in the direction of the arrows.

Fig. 4 is a vertical section showing the liquid containing rotatable cup with the grooved lap and the iron disc with a silicon slab cemented thereto and resting on the grooved lap.

Fig. 5 is a horizontal section of the device of Fig. 4 taken along the line 5—5 looking in the direction of the arrows.

The same reference characteristics are used to indicate identical elements in the several figures of the drawings.

Referring now to the drawings, particularly to Fig. 1, a specific embodiment of this invention is shown mounted on a wooden table 5, a fragment only of which is shown. A rotatable cup 6 is supported on the upper end of a shaft 7 which carries a rigidly attached tapered cap. The bottom portion 8 of the cup 6 is provided with a tapered recess within which the tapered cap on the shaft 7 fits snugly. A threaded collar 9 on a threaded cylindrical portion of the tapered cap on the shaft 7 facilitates removal of the cup 6, the cup 6 being loosened by screwing the collar 9 upward by means of a handle 10. This arrangement for mounting cup 6 on shaft 7 is shown in more detail in Fig. 4.

The shaft 7 is supported in a housed bearing

11 secured to a steel plate 12 which plate is bolted to the under surface of the table 5. A pulley 25 on the lower end of shaft 7 is driven by a belt 13 on the output pulley 14 of a speed-reducing mechanism 15. Power for the mechanism 15 is furnished by a motor-driven shaft 16 (see Fig. 2) through an all-speed transmission mechanism 17 and belts 18 and 19. Mechanism 17 comprises an input pulley 20, an output pulley 21 and a speed control handle 22. The speed-reducing mechanism 15 is driven by belt 19 on input pulley 23. Belt 18 is driven by pulley 24 on power shaft 16.

Movements of oscillation and rotation are imparted to the material to be polished by the mechanism now to be described. Referring first to Fig. 4, a slab 30 of material to be polished is cemented to the face 31 of a metal disc 32, such as steel or brass. The disc 32 is adapted to be moved horizontally by shaft 33 through the intermediary of pin 34 which is inserted into a hole in the lower end of shaft 33 and rigidly secured to the shaft 33 by a set screw. The lower end of pin 34 projects into a hole in disc 32. A washer 35 surrounds the pin 34 between the shaft 33 and the disc 32. A relatively large steel washer 36 surrounds a cylindrical projection 26 of the disc 32 with a turning fit.

Referring again to Fig. 1 the shaft 33 is carried by an arm 37 pivoted between the legs of a U-shape member 38 (see also Fig. 3) which arm is adapted to oscillate about a vertical axis and is driven by an eccentric cam 39 acting on a cam follower 40. The eccentric cam 39 is secured to the upper end of shaft 41 which is supported in a housed bearing 42 bolted to the table 5. The shaft 41 is driven from a pulley on the lower end of shaft 7 through an all-speed transmission mechanism 43 and belts 44 and 45. The mechanism 43 comprises an input pulley 46, an output pulley 47 and a speed control handle 48. The shaft 41 is driven by belt 45 on pulley 49. Input pulley 46 is driven by belt 44 on pulley 50 (see Fig. 2) on the lower end of shaft 7.

In addition to the elements previously mentioned, the mechanism for oscillating the material 30 to be polished comprises a U-shaped support 51, the legs of which are bolted to the table 5. The U-shaped member 38 rests on the upper surface of the cross member of U-shaped member 51 and is adapted to rotate about a vertical axis. A downwardly extending steel rod having a drive fit in the cross member of U-shaped member 38 fits into a downwardly extending steel bushing 52 having a drive fit in the cross member of U-shaped member 51 and being welded to the cross member. This bushing 52 serves as a bearing for the downwardly extending rod of U-shaped member 38 to support U-shaped member 38 for oscillation about a vertical axis on top of U-shaped member 51. Oscillating arm 37 is supported between the upstanding legs of U-shaped member 38 by pivots 53. Shaft 33 is supported in a bearing 54 carried by arm 37 at the end opposite to the pivots 53. As cam 39 rotates cam follower 40 is oscillated about the axis of bushing 52 to oscillate the U-shaped member 38 and so to oscillate the arm 37 carrying the shaft 33 and the material 30 to be polished.

The rotation of shaft 41 also rotates shaft 33 through the intermediary of belts 55 and 56. Belt 55 is driven by pulley 57 secured to the shaft 41 just above bearing 42 and drives pulley 58. Pulley 58 drives pulley 59 through the intermediary of shaft 60, to which shaft both pulleys 58 and 59 are rigidly secured. Belt 56 is driven by pulley 59 and drives pulley 61 which is rigidly secured to the top end of shaft 33. Shaft 60 is supported in bearings in the legs of U-shaped member 62. The lower part of the cross member of the U-shaped member 62 is welded to a cross bar 63, the ends of which cross bar are welded to the upper portion of the legs and the cross member of U-shaped member 51 to support shaft 60 with its axis vertical.

Referring now to Figs. 4 and 5, the arrangement of the tin lap 70 within the rotatable cup 6 is shown in more detail. The lap 70 consists of substantially pure tin in the shape of a broad faced washer, which washer fits snugly into a finished recess 71 in the bottom of the cup 6. One face of this washer or lap 70 which is the upper face after the lap is assembled within the cup 6, is provided with a spiral groove 72 of many turns starting at the hole in the center and ending at the periphery of the washer 70. The groove 72 is V-shaped 0.005 inch deep with a 60-degree angle between the sides and has a pitch of $\frac{3}{64}$ inch. A few only of the many turns of groove 72 are shown in Figs. 4 and 5.

The power from the driving motor is transmitted to the various driven elements of the device as described hereinbefore and in the direction indicated by the arrows in Fig. 2.

The relationship of the slab 30 to be polished and the lap 70 is shown in Fig. 4. The polishing is done under water carrying the abrasive. When the device is operating, the cup 6 and lap 70 are rotating about the axis of shaft 7 and the surface of the liquid in the cup 6 assumes the approximate shape shown by the lines 73. In a modified arrangement the liquid is pushed back toward the center by a piece of $\frac{1}{32}$ inch lead sheet fastened to a support on the table and overhanging into the outer periphery of the liquid. This permits less liquid to be used and the polishing disc can be run faster. The oscillating arm 37 is in the position shown in Fig. 1. The opening in the arm 37 through which the stationary U-shaped member 62 and shaft 60 protrude as shown in Fig. 2, is large enough to permit the arm 37 to oscillate so that the slab 30 is moved substantially radially across one side of the lap 70. The center of the pulley 59 is close enough to the axis of oscillation of the arm 37 so that the tension on belt 56 is not changed materially for different positions of the arm 37 during each oscillation. In order to remove the slab from the machine the arm 37 is raised by rotation about the pivot 53. This can be done without removing the belt 56 which is merely loosened as the arm 37 is raised. The speed of rotation of the lap 70 is adjusted by means of the all-speed transmission mechanism 17 and the relative speeds of rotation of the lap 70 and the shaft 33 is controlled by the all-speed transmission mechanism 43.

This polishing device has been found to be particularly useful in polishing slabs of solidified fused silicon of high purity. Such material is produced by fusing silicon powder of high purity in a silica crucible in an electric furnace and allowing the fused silicon to cool at a controlled rate. Such material has the scratch hardness of crystalline quartz but is not nearly as strong. It cannot be machined to approximate shape like malleable metals. It has a very small elastic range and therefore will stand very little bending force without breaking. The usual buffing methods of applying a final polish result only in lemon peel effects. The method of this invention and the use of the above described machine have given good results in producing an optically flat surface on slabs of such material.

For a detailed description of a method of producing material which can be polished to advantage by the method and means of this invention reference is made to Patent 2,402,582 of J. H. Scaff issued June 25, 1946 on application Serial No. 386,835 filed April 4, 1941.

The process of polishing a slab of silicon will now be described.

A slab of silicon is cut from a suitably sized and homogeneous piece of silicon. A Norton metal bonded slicing wheel is most suitable for this purpose. The peripheral speed of the slicing wheel may be conveniently about 120 feet per minute and should be so arranged as to be constantly washed in a sludge accumulator containing otherwise clean water or in a small stream of clean water. Such slab is conveniently about 0.028 inch thick. This slab may then be flattened on both sides by lapping it on a flat cast iron lap using American Optical Company abrasive M 302. The flattened faces are approximately parallel and the slab is now about 0.019 inch thick. One flattened surface may then be cemented to the end of a round metal form with lapidarist's cement (unpolymerized shellac). The silicon slab can now be rounded by cutting with the diamond slicing wheel, or if more accuracy is required, the final roundness may be gotten by grinding in a lathe with a continuously moistened resinoid-bonded diamond wheel having a mesh of about 400 or finer to insure the absence of deep chipping along the edges. The slab may now be removed from the rounding form and mounted on the final polishing jig.

An optically flat product is readily obtained by closely following the now-to-be described procedure. A cast iron lap is prepared by cutting a flat surface on a good lathe. The surface should not be free from cutting marks left by the cutting tool. Such cutting marks indicate ridges which permit the abrasive to cut under the whole surface of the slab. When the slab has been brought to a flat matt finish with M 302 abrasive mixed with distilled water, about $\frac{1}{100}$ per cent dioctyl sodium sulfosuccinate solution and about $\frac{1}{5}$ per cent potassium bichromate it is ready for the polishing procedure. After carefully removing all abrasive from the sample and the jig by brushing with soap and under running water, the slab is placed in the first polishing cup. This cup may contain a pure tin lap whose surface has been machined smooth and 60 degree grooves 0.005 inch deep cut into the surface at a pitch of $\frac{3}{64}$ inch. A small quantity of 400 mesh boron carbide is contained in the liquid which covers the lap. The liquid is made up of distilled water containing about $\frac{1}{100}$ per cent dioctyl sodium sulfosuccinate solution and about $\frac{1}{5}$ per cent potassium bichromate. A fine diamond charged tin lap works faster for the first cup than the 400 mesh boron carbide. The pressure is about ten pounds. The machine is now operated until all the pits from the previous matt finish are removed. Depending on the size of the silicon slab, the time required for this may vary from about 3 to 30 minutes.

This procedure is repeated with levigated 800 mesh boron carbide until the coarse scratches left by the previous operation are removed.

The final polish is obtained in a similar way using sapphirine as the cutting agent. The pressure is about fifteen pounds per square centimeter. If the whole operation is carried out with extreme cleanliness and care, the sample will be free from scratches as seen under a magnification of 150 diameters. The sample will be uniformly flat within $\frac{1}{4}$ wavelength of green light as measured with Fresnal interference fringes.

A good polish can also be obtained by the use of diamantine (or its equivalent) polishing powder in place of sapphirine.

A most important feature of this polishing technique is the machine for producing the desired mechanical movements. The machine rotates the cutting laps. Due to frictional differences depending upon the distance from the center of the lap, a rotating torque is developed in the sample. In some cases this torque is sufficient to spin the sample being polished in other cases it is not, therefore the rotation is aided by a power drive. In addition, a reciprocating motion is given the sample so that the lap does not become worn and develop uneven grooves. This reciprocating motion is controlled by a stroke speed adjustment. The speeds are so arranged that the sample has no fixed multiple relation to the rotation of the lap so as to avoid the formation of a pattern on the lap, which would be harmful.

The polishing process is carried out under water and is strictly a cutting process as compared to a buffing process. The pieces of silicon which are removed in the final process are so fine that a chemical action with water seems to occur. This is indicated by the formation of a surface layer as indicated by electrical tests. This surface layer gives every indication of being ortho-silicic acid produced by the action of the finely divided silicon upon the water. The ortho-silicic acid surface will absorb and lose water until it retains less than 17 per cent water when it becomes meta-silicic acid which finally dries to a permanent hard surface. For optical purposes this thin surface of ortho-silicic acid does no harm but if it is found necessary to remove it, this can readily be done by immersing the slab in freshly prepared concentrated nitric acid held in a parawax container with one per cent of 47 per cent hydrofluoric acid added. An immersion of one second immediately followed by a wash in running distilled water removes the ortho-silicic acid without affecting the silicon surface. Sometimes the meta-silicic acid surfaces require as much as 10-second treatment if they have been thoroughly dehydrated.

An excellent polish can also be obtained by the use of pure lead laps. Lead used for this purpose should be free from silver and other hard metals, although tin in various proportions does little harm. If lead laps are used, a few drops of water glass must be substituted in place of potassium bichromate which latter chemical attacks the lead in time.

The purpose of the dioctyl sodium sulfosuccinate is to reduce the liquid surface tension which allows the fine abrasive to cut more readily.

The potassium bichromate is added to arrest corosion of the metal parts within the lapping cups. It is especially effective in the prevention of rusting of cast iron. The chromate is not ordinarily used with lead laps because of the chemical action on the lead.

The organic compounds mentioned hereinbefore belong to the class of materials designated by the terms surface active agents or wetting agents. Materials of this class may be used as the wetting agent in the practice of this invention providing that they are chemically inactive with respect to the materials with which they come in contact during the polishing process. Examples of such materials are:

Dioctyl sodium sulfosuccinate
Dihexyl sodium sulfosuccinate
Diamyl sodium sulfosuccinate
Dibutyl sodium sulfosuccinate
Isopropyl naphthalene sodium sulfonate

The commercial product of American Cyanamid and Chemical Corporation which contains 25 per cent of pure dioctyl sodium sulfosuccinate, a mutual solvent consisting of one of the compounds selected from the group of compounds consisting of alcohol, acetone, ethylene glycol monoethyl ether and diethylene glycol monobutyl ether, and water to make up the remaining 75 per cent is particularly easy to use in the practice of this invention. The specific percentages of dioctyl sodium sulfosuccinate mentioned in this application refer to the above described commercial product.

Water glass which is mentioned hereinbefore as a substitute for potassium bichromate when a lead lap is used, also functions as a wetting agent for the practice of this invention.

The tin lap may be charged with sapphirine to good advantage. To effect such charging the grooved surface of the lap is coated with a paste of sapphirine and water and the coated surface is rolled with a metal roller so that particles of the sapphirine are embedded in the surface of the lap. The charged lap is used for polishing in the same manner as an uncharged lap in the manner described hereinbefore.

A convenient size of silicon slab is about one and one-eighth inches square polished on a lap four and five-eighth inches in diameter.

The same polishing technique has been used successfully on germanium, zincite, hardened steel and a number of other solids to produce an optical finish.

The invention as defined in the appended claims may find embodiment in structures and methods differing widely from the specific structures and methods described in this specification. Such embodiments come within the purview of the claims.

What is claimed is:

1. The method of polishing a crystalline material of the nature of highly pure crystalline silicon with respect to the action of abrasives thereon comprising moving a surface of said material to be polished in contact with the surface of tin lap in a cyclically nonrepetitive path, immersing the material during movement in a liquid bath of distilled water containing a finely divided abrasive and dioctyl sodium sulfosuccinate, and rotating a container within which said lap is fitted snugly, said container containing said liquid during the movement of said material for polishing said surface.

2. The method of polishing solidified fused crystalline silicon which comprises immersing a surface of a tin lap and a silicon surface to be polished in distilled water carrying finely divided sapphirine and surface active agent which is chemically inactive with respect to the material in contact with said water, rotating the tin lap, and moving the surface of the silicon body to be polished in contact with the surface of and repeatedly transversely of the rotating tin lap surface while both are immersed in said water.

3. The method of polishing a crystalline material of the nature of highly pure crystalline silicon with respect to the action of abrasives thereon comprising moving a surface of said material to be polished in contact with the surface of a tin lap in a cyclically nonrepetitive path with the surface of the material being polished facing downward, immersing the material during movement in a liquid bath of distilled water containing finely divided sapphirine and isopropyl naphthalene sodium sulfonate, and rotating a container containing said lap and said liquid during the movement of said material for polishing said surface.

4. The method of polishing a crystalline material of the nature of highly pure crystalline silicon with respect to the action of abrasives thereon comprising moving a surface of said material to be polished in contact with the surface of a soft metallic lap in a cyclically nonrepetitive path with the surface of the material being polished facing downward, immersing the material during movement in a liquid bath of distilled water containing a finely divided abrasive, dialkyl sodium sulfosuccinate and potassium bichromate, and rotating a container within which said lap is fitted snugly, said container containing said liquid during the movement of said material for polishing said surface.

5. The method of polishing solidified fused crystalline silicon which comprises immersing the surface of said silicon to be polished in distilled water carrying finely divided sapphirine and dialkyl sodium sulfosuccinate and in contact with a metallic surface having hardness of the order of that of tin, and maintaining the contacting surfaces immersed while giving them relative movement to effect the polishing action.

6. The method of polishing a crystalline material of the nature of highly pure solid crystalline silicon with respect to the action of abrasives thereon comprising moving a surface of said material to be polished in contact with the surface of a soft metallic lap in a cyclically non-repetitive path, immersing the material during movement in a liquid bath of distilled water containing a finely divided abrasive and a dialkyl sodium sulfosuccinate, and rotating a container within which said lap is fitted snugly, said container containing said liquid during the movement of said material for polishing said surface.

7. The method of polishing a crystalline material of the nature of highly pure solid crystalline silicon with respect to the action of abrasives thereon comprising moving a surface of said material to be polished in contact with the surface of a soft metallic lap in a cyclically non-repetitive path, immersing the material during movement in a liquid bath of distilled water containing a finely divided abrasive and dioctyl sodium sulfosuccinate, and rotating a container within which said lap is fitted snugly, said container containing said liquid during the movement of said material for polishing said surface.

8. The method of polishing a crystalline material of the nature of highly pure solid crystalline silicon with respect to the action of abrasives thereon comprising moving a surface of said material to be polished in contact with the

surface of a soft metallic lap in a cyclically non-repetitive path, immersing the material during movement in a liquid bath of distilled water containing a finely divided abrasive and isopropyl naphthalene sodium sulfonate, and rotating a container within which said lap is fitted snugly, said container containing said liquid during the movement of said material for polishing said surface.

9. The method of polishing a crystalline material of the nature of highly pure solid crystalline silicon with respect to the action of abrasives thereon comprising moving a surface of said material to be polished in contact with the surface of a soft metallic lap in a cyclically non-repetitive path, immersing the material during movement in a liquid bath of distilled water containing a finely divided abrasive and water glass, and rotating a container within which said lap is fitted snugly, said container containing said liquid during the movement of said material for polishing said surface.

10. A machine for polishing highly pure crystalline silicon comprising an upright cup-shaped member adapted to be rotated about its axis in a vertical position, a cylindrical tin lap lying flat on the bottom of said member adapted to be rotated thereby, a liquid within said cup in contact with and covering said lap, said liquid comprising distilled water, levigated sapphirine, a small percentage of dioctyl sodium sulfosuccinate and potassium bichromate immersing said lap during rotation, a crystal holder positioned in opposing juxtaposition to the lap and adapted to hold a slab of highly pure crystalline silicon, means coupled to one of said above-mentioned opposed elements for urging them toward one another, and means secured to said holder and coupled with said cup-shaped member for moving said holder repeatedly transversely of the direction of movement of said lap as said lap is rotated.

11. A machine for polishing highly pure crystalline silicon comprising an upright cup-shaped member adapted to be rotated about its axis in a vertical position, a cylindrical tin lap lying flat on the bottom of said member adapted to be rotated thereby, a liquid within said cup in contact with and coverning said lap, said liquid comprising distilled water, levigated sapphirine, a small percentage of isopropyl naphthalene sodium sulfonate and potassium bichromate immersing said lap during rotation, a crystal holder positioned in opposing juxtaposition to the lap and adapted to hold a slab of highly pure crystalline silicon, means coupled to one of said above-mentioned opposed elements for urging them toward one another, and means secured to said holder and coupled with said cup-shaped member for moving said holder repeatedly transversely of the direction of movement of said lap as said lap is rotated.

12. A machine for polishing highly pure crystalline silicon comprising an upright cup-shaped member adapted to be rotated about its axis in a vertical position, a cylindrical tin lap lying flat on the bottom of said member adapted to be rotated thereby, a liquid within said cup in contact with and covering said lap, said liquid comprising distilled water, levigated sapphirine, a small percentage of dialkyl sodium

sulfosuccinate and potassium bichromate immersing said lap during rotation, a crystal holder positioned in opposing juxtaposition to the lap and adapted to hold a slab of highly pure crystalline silicon, means in operative engagement with one of said above-mentioned opposed elements for urging them toward one another, and means secured to said holder and coupled with said cup-shaped member for moving said holder repeatedly transversely of the direction of movement of said lap as said lap is rotated.

13. A machine for polishing highly pure crystalline silicon comprising an upright cup-shaped member adapted to be rotated about its axis in a vertical position, a cylindrical substantially pure lead lap lying flat on the bottom of said member adapted to be rotated thereby, a liquid within said cup in contact with and covering said lap, said liquid comprising distilled water, levigated sapphirine, a small percentage of dioctyl sodium sulfosuccinate and water glass immersing said lap during rotation, a crystal holder positioned in opposing juxtaposition to the lap and adapted to hold a slab of highly pure crystalline silicon, means in operative engagement with one of said above-mentioned opposed elements for urging them toward one another, and means secured to said holder and coupled with said cup-shaped member for moving said holder repeatedly transversely of the direction of movement of said lap as said lap is rotated.

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