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CRYSTALS WITH SMALL APERTURES

Fordyce H. Horn, Schenectady, N. Y., assignor to General Electric Company, a corporation of New York

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This invention relates to crystals with small holes. More particularly, it relates to the preparation of crystals having holes of the order of less than 1 micron diameter, ranging up to a diameter of several hundred microns.

It is an object of this invention to provide a method whereby crystals grown in accordance with a particular pattern may have a hole incorporated therein.

It is another object of the invention to produce a crystal having a small hole suitable for wire-drawing and for serving as an aperture for pinhole beams in X-ray or electron diffraction techniques.

It is another object of the invention to provide a small hole in a metal film.

In the Discussions of the Faraday Society for 1949, volume #5 on page 48 comprises an article by F. C. Frank, wherein he discloses a mechanism, referred to as a "screw dislocation," whereby the growth of certain crystals is effected.

As is well known, crystal growth takes place only from a super-saturated solution. In most cases the rate of crystal growth depends upon the degree of supersaturation. There is a set of conditions which will permit of crystallization from solution at a rapid rate even though the solution is only very slightly supersaturated. The above cited article by F. C. Frank describes one mechanism whereby the rapid growth of crystals from a solution having only a slight degree of supersaturation may be accomplished.

According to the Frank publication, spiral growth of a crystal is initiated by a screw dislocation. In accordance with this hypothesis a crystal begins to grow from a starting nucleus in a solution having a slight degree of supersaturation. If the crystal growth is of the type subject to screw dislocation, the internal stresses produce a bend running outward from a point near the center of the crystal. This bend relieves the internal stresses by distorting the crystal plane slightly to give it a spiral configuration. Further growth of the crystal then does not depend upon the nucleation of a fresh surface. The spiral provides continuous steps on which the growth units can deposit, and every time a growth unit deposits, it automatically produces another similar step. Thus, a spiral built from a screw dislocation automatically perpetuates conditions suitable for further growth. The axis of the screw is perpendicular to the planes of growth, and unless the process is interrupted, the axis will extend through the crystal from top to bottom surfaces.

The screw axis is at the center of the spiral. In accordance with one aspect of my invention, I make use of my discovery that the region immediately surrounding the spiral axis differs from the bulk of the crystal in that it is a region of poorly oriented building units which are more subject to chemical attack than the remainder of the crystal. I subject a screw dislocated crystal to chemical attack to produce a hole through the spiral axis. I control the size of this hole for any particular thickness of crystal by varying the time of application and character of the chemical attack.

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Spiral growth has been observed from a screw dislocation in such diverse substances as aluminum boride, silicon carbide, cadmium iodide, long-chain paraffins, and beryl.

The phenomenon of spiral crystal growth initiated by screw dislocation is not limited to any particular class of substances or to any particular type of crystal lattice. The above enumerated list of materials contains organic as well as inorganic substances. While most of the above substances crystallize on a hexagonal lattice, this is not a necessary requirement. However, the hexagonal system is a preferred type of crystal, as it has proved to be very practical.

Spiral crystalline growth from a screw dislocation is not limited to simple depositions. Silicon carbide crystals produced in a high temperature electric furnace show the characteristic spiral growth when viewed through a microscope. It frequently happens that several screw dislocations will occur on the same crystal. The spiral growths initiated by these several screw dislocations will merge into a single growth. When a crystal having several screw dislocations is subsequently subjected to dissolution action, a hole will be produced at each of the spiral axes.

When a crystal has built up to the desired plate thickness, it is subjected to a treatment which will produce a hole along the spiral axis. The treatment may consist of a solvent wash or it may consist of heating or reducing the concentration of the solution to a point where it is not quite saturated. Any treatment which will tend to dissolve the crystal fairly rapidly is satisfactory. If dissolution is too rapid or too slow the entire crystal will dissolve without formation of a hole. When the crystal is subjected to a dissolution treatment, the poorly oriented building units near the spiral axis are attacked first, with the result that holes are produced through the spiral axis. The size of these holes may be controlled by varying the time of the dissolution treatment to which the crystal is subjected.

In preparing aluminum boride (AlB_2) crystals in accordance with my invention, I place Al and B powders (with ratio from about 12Al:B to 2Al:B) in a crucible and heat to 1000° C. for about 6 hours. This process produces aluminum boride crystals formed in the aluminum which is present in excess. The aluminum boride crystals are recovered from the aluminum by treating the mixture with hydrochloric acid of about 1:4 to 1:10 dilution. This treatment dissolves the aluminum and also a portion of the aluminum boride along the spiral axis. Most of the crystals produced by this process are in the form of hexagonal plates with a single hole located near the center of the hexagon.

Aluminum boride crystals produced as above may readily be coated with various metals by the well-known vacuum evaporation process. Certain metals may also be applied by deposition out of a solution. For example, the method whereby mirrors are formed by plating silver on glass out of a sugar solution may be employed to place a metallic coating on aluminum boride crystals prepared as above. Metal coatings applied by the methods described above may be separated from the crystal thus leaving a thin metal film having a small hole therein.

Cadmium iodide is an example of a water soluble substance which is subject to crystal growth by screw dislocation. In order to apply my process to produce small holes in cadmium iodide crystals, it is only necessary to prepare a slightly supersaturated aqueous solution of cadmium iodide. When the crystals have attained the desired size, holes are produced most expeditiously either by heating the solution in order to reduce the concentration to slightly below the saturation point, or by adding sufficient water to accomplish the same result. The time

of the dissolution treatment is correlated to the size hole desired.

Silicon carbide is heated in fused sodium carbonate or a mixture of fused sodium carbonate and fused potassium carbonate at 1000° C. for 2 to 6 hours in order to dissolve the spiral axes to produce holes of about 1 micron diameter.

While I have given specific directions for the preparation of aluminum boride, cadmium iodide, and silicon carbide crystals having holes therein in accordance with my invention, it is obvious that any crystal which has been formed by spiral growth around a screw dislocation may have holes formed therein in accordance with my invention. Since one skilled in the art can readily determine the best mode of applying my invention to any crystal formed by spiral growth around a screw dislocation, I will not present additional specific examples. It will be understood that numerous modifications may be made by those skilled in the art without actually departing from the invention. Therefore I aim in the appended claims to cover all such equivalent variations as come within the true spirit and scope of the foregoing disclosure.

What I claim as new and desire to secure through Letters Patent of the United States is:

1. The method of forming a small symmetrical axial aperture in a crystal of silicon carbide having therein a screw dislocation which method comprises immersing said crystal in a bath of a fused alkali carbonate to dissolve away that portion of the crystal along the axis of the dislocation.

2. The method of forming a small symmetrical axial aperture in a crystal of aluminum boride having therein a screw dislocation which method comprises heating a mixture of aluminum and boron in weight ratios of approximately 2 to 12 parts aluminum to 1 part boron at a temperature of approximately 1000° C. for approximately 6 hours, cooling the mixture to form a crystalline mass, treating the crystalline mass with approximately 1-4 to 1-10 dilute hydrochloric acid to produce hexagonal aluminum boride crystals with small axial apertures therein.

3. The method of forming a small symmetrical axial aperture in a crystal having therein a screw dislocation and having the characteristic that the portion along the axis of dislocation is preferentially subject to dissolution by a solvent of said crystal which method comprises subjecting said crystal to a solvent therefor and dissolving away that portion of the crystal along said axis of dislocation.

4. The method of forming a small symmetrical axial aperture in a single crystal having therein a single screw dislocation and having the characteristic that the portion along the axis of dislocation is preferentially subject to corrosion by a corrosive agent of said crystal which method comprises subjecting said crystal to a corrosive agent therefor to corrode away that portion of the crystal along said axis of dislocation.

5. The method of forming a small symmetrical axial metal sheathed aperture in a crystal having therein a screw dislocation and having the characteristic that the portion along the axis of dislocation is preferentially subject to corrosion by a corrosive agent of said crystal which method comprises subjecting said crystal to a corrosive agent therefor to corrode away that portion of the crystal along said axis of dislocation, and plating a metallic coating on said crystal.

6. The method of claim 5 wherein plating is accomplished by an evaporation process.

7. The method of forming a small symmetrical axial aperture in a crystal having therein a hexagonal screw dislocation and having the characteristic that the portion along the dislocation axis is preferentially subject to corrosion by a corrosive agent of said crystal which method comprises subjecting said crystal to a corrosive agent therefor to corrode away that portion of the crystal adjacent said dislocation axis.

8. An article of manufacture comprising a single crystal including a screw dislocation therein, in which the portion along the axis of dislocation is preferentially subject to dissolution by chemical action, and having a small axial symmetrical aperture the order of one to several hundred microns in diameter along said axis of dislocation, said aperture being formed by chemical dissolution.

9. An article of manufacture comprising a single crystal including a screw dislocation therein, in which the portion along the axis of dislocation is preferentially subject to dissolution by chemical action, and having a small axial symmetrical aperture the order of one to several hundred microns in diameter along said axis of dislocation, said aperture being formed by chemical dissolution, and a protective metallic coating on said crystal in the vicinity of said aperture.

10. The article of manufacture of claim 8 wherein the single crystal comprises silicon carbide.

11. The article of manufacture of claim 8 wherein the single crystal comprises aluminum boride.

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