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PROCESS FOR THE MANUFACTURE OF ARTIFICIAL CRYSTALS

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Fig. 1

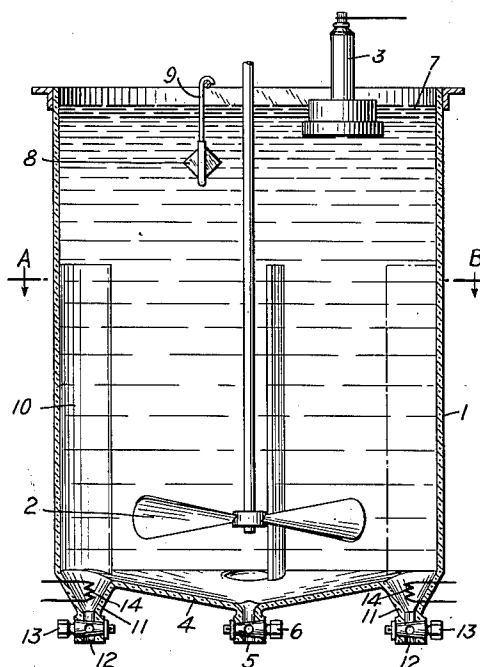
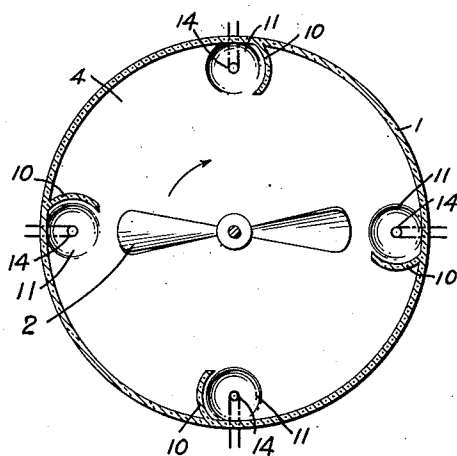


Fig. 2



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PROCESS FOR THE MANUFACTURE OF
ARTIFICIAL CRYSTALS

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6 Claims. (Cl. 23—301)

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The present invention relates to the preparation of artificial crystals, such as piezoelectric crystals, from a supersaturated solution of the salt of the crystal desired.

When crystals are caused to crystallise in a solution which is supersaturated, great difficulty is encountered due to the presence in the solution of dust and suspended particles. Such particles form centres of crystallisation on which are deposited microscopic crystals. The majority of these fall to the bottom of the receptacle quite slowly and rob the solution of strength, in such a way that the large crystals that one desires to obtain are seldom obtained. Furthermore some of the microcrystalline particles in suspension in the solution become fixed on the surfaces of the large crystals in positions that do not correspond to the general disposition of ions in the principal crystalline lattice. This results in local flaws which cause inclusions of supersaturated solution in the crystal.

It is known that the seriousness of these factors can be reduced by various procedures, such as filtering, centrifuging, etc. These methods diminish the number of dangerous parasitic particles but do not remove them entirely.

An object of the present invention is to provide means for obtaining supersaturated solutions for the preparation of artificial crystals not containing impurities. A further object is to provide means whereby parasitic crystal nuclei which may form in the solution during the growth of the desired crystals may be prevented from adhering to the desired crystal and interfering with its regular growth.

According to one feature of the invention, the supersaturated solution is submitted to amplitude vibrations of frequencies such that the parasitic crystals described above are produced very rapidly; then the solution is freed from the crystals thus formed. For this purpose it may be desirable to employ ultrasonic frequencies.

According to another feature of the invention, the solution is stirred during the application of the ultrasonic waves, in order to ensure that the parasitic particles pass through the ultrasonic beam.

A description of a process employing the principles of this invention is given below which should be read in conjunction with the accompanying drawings, in which:

Figure 1 is a vertical section of an apparatus employing the principles of this invention;

Figure 2 is a schematic section of an apparatus taken through the plane A—B of Figure 1.

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In a container 1, preferably transparent, maintained at any desired temperature by a regulated heating system (not shown) a saturated solution of a salt is prepared at a given temperature. To simplify the description, we will suppose that it is desired to obtain piezoelectric crystals of monophosphate of ammonia; it will be quite evident that the procedure is as well applied to crystals of other salts, providing the appropriate temperatures and concentrations are employed. In 100 litres of water heated for example, to a temperature of 50° C., are dissolved 69.5 kg. of monophosphate of ammonia; there is thus obtained a saturated solution at 50°; the mixture is stirred, for example by means of a propeller 2, to obtain throughout the solution a homogenous temperature.

The temperature of the solution is then reduced to 45° C., obtaining thus a supersaturated solution and at the same time the solution is submitted to vibrations preferably by means of a beam of ultrasonic waves produced by generator 3. This beam has the property of provoking an instantaneous formation of microscopic crystals, these crystals forming on the particles in suspension. These particles are then, therefore, rapidly encased in small crystals which cause them to become heavy and to fall to the bottom of the receptacle. Owing to the operation of propeller 2, the solution is made to pass through the ultrasonic beam.

When the density of the solution in this example has become that of a solution saturated at 45° C., the production of the ultrasonic waves, and the operation of propeller 2 are stopped allowing the crystals to fall to the bottom of the receptacle, which has preferably a bottom 4 of conical form, supplied with a tube 5 and a stopcock 6. Care has been taken to apply to the surface of the solution before the action of the ultrasonic beam a film 7 of a neutral liquid, such as oil of Vaseline which prevents the introduction of dust particles into the solution.

The crystals which are deposited at the bottom of container 1, are siphoned off by means of stopcock 6, and it is verified by causing a luminous beam to travel through the solution that the solution is optically transparent.

After having eliminated all the crystals and all their germs, which have been combined with the dust particles in the suspension of the solution, the solution is re-heated several degrees to redissolve any nuclei which remain in the container; then is allowed to cool to 40° C. for example, which furnishes a supersaturated solution

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which is free from impurities. There are then plunged into the solution one or more crystalline nuclei which it is desired to cause to increase, suspended for example by means of supports 9, continuing to stir the solution by means of propeller 2, until the nuclei have grown into crystals of the desired dimension.

Other crystalline particles can accidentally reform in the solution, and may be deposited on the crystal under formation. The suspension of these undesirable nuclei can be avoided by disposing, for example, along the vertical walls of container 1, plates 10 which may be vertical. Because of the stirring effect of the propeller 2, these nuclei arrive in contact with the said plates where they are stopped and then fall to the bottom of the container. At the bottom of these plates may be placed small funnels 11, provided with evacuation tubes 12 closed by stopcocks 13, in order to evacuate these undesired particles from the solution. Equally heating elements 14 may be provided in the funnel 11, which elements may be, for example, electrical resistances, which locally elevate the temperature of the solution, and create a redissolution of the crystals thus deposited. This last device also avoids useless loss of the strength of the solution and permits one to obtain larger crystals in a given volume of solution under given temperature conditions.

Although the invention has been described with respect to one particular embodiment and for a particular kind of crystal, it is clear that it is not limited to this example, and that there may be many other forms of apparatus and elements employing the principles of the present invention, and that these may be applied to crystals of a different nature without departing from the spirit of the invention.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:

1. A process for preparation of artificial crystals comprising the steps of obtaining a supersaturated solution of a salt in a solvent, applying to said supersaturated solution mechanical vibrations, eliminating any deposit of crystal nuclei formed on impurities in said solution by said mechanical vibrations, then introducing into said solution crystal nuclei to constitute the centers of the desired crystals, and forming said crystals on said nuclei.

2. A process for the preparation of artificial crystals such as piezo-electric crystals, by crystallization from a supersaturated solution, comprising the dissolving of a salt in a solvent at a predetermined temperature, the protection of the surface of the resulting solution by a neutral

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liquid with which it is non-miscible, mechanical stirring of said solution, a cooling of the solution produced to obtain a supersaturated solution, the application to the said supersaturated solution of mechanical vibrations to rapidly form crystalline nuclei on impurities present in said solution, the elimination from said solution of said parasitic crystalline nuclei, and the introduction into the said solution of one or more crystalline nuclei to constitute the centers of the desired crystals, whereby the desired crystals may be grown in said supersaturated solution substantially free from the interference of parasitic crystalline nuclei.

3. The process according to claim 2, and further comprising the steps of re-heating said solution to dissolve any parasitic crystalline nuclei which accidentally remain in suspension after said solution is subjected to said mechanical vibrations and re-cooling said solution to again obtain a supersaturated solution.

4. A process according to claim 2, comprising local heating to redissolve any parasitic crystal nuclei produced throughout the said supersaturated solution during the growth of the crystal or crystals which it is desired to obtain.

5. The process according to claim 2 in which said mechanical vibrations are of ultrasonic frequencies.

6. The process according to claim 2, and further comprising the elimination of newly formed parasitic crystalline nuclei from said supersaturated solution during the growth of the desired crystals, by the continuous agitation of said solution to force said parasitic nuclei to suitable evacuation points.

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