May 20, 1947. H. C. KREMERS ET AL 2,420,955

METHOD OF MAKING OPTICAL BODIES

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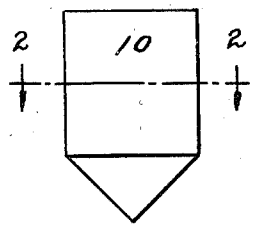


Fig. 1

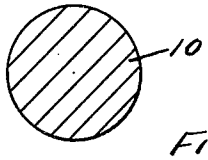


Fig. 2

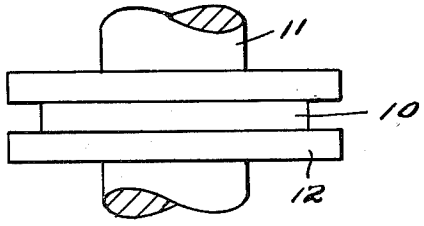


Fig. 3

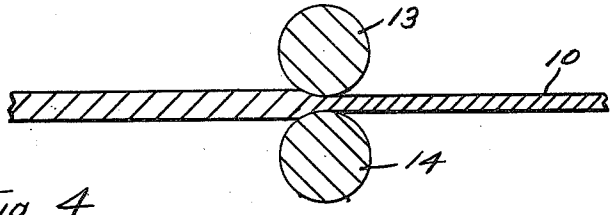


Fig. 4

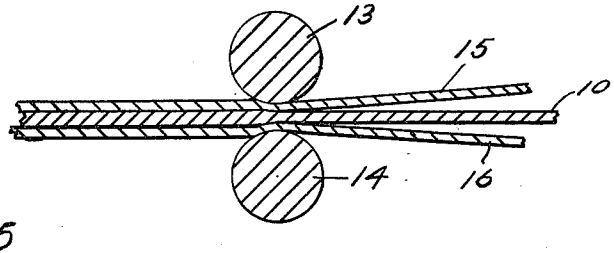


Fig. 5

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METHOD OF MAKING OPTICAL BODIES

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6 Claims. (Cl. 18—47.5)

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This invention relates to the production of optical material, such as windows, prisms, sheets, and the like, from silver halides and mercurous chloride.

It is known that these compounds have the property of forming crystals with sufficient plasticity to permit the casting of ingots which may then be shaped in various ways, such as by rolling into sheets, or pressing into other shapes. The usual procedure for producing silver chloride optical sheets is to melt the salt in a suitable container, often a Pyrex beaker, glazed porcelain crucible or quartz crucible, or a silver pot, and then cast the molten salt into a suitable mold or on a chilled plate. The resulting ingots or plates can then be rolled or pressed into sheets or special shapes, such as rods, prisms and the like. Procedures of this type are described in the literature. See Fugassi and McKinney, RSI 13, 335 (1942), O. Reinkober, Handbuch and Jahrbuch der Chemischen Physik 9, 39, (1934), J. Maslakowez, Ztschr. t. Phys. 51, 696 (1928).

In the art as practiced up-to-date, the ingot of silver chloride usually contains cloudy portions and pores containing dross which have to be removed. This can be avoided by our novel procedure as will be pointed out.

During the rolling of silver chloride ingots into sheet form, frequent annealing of the sheet is necessary to prevent excessive brittleness and subsequent shattering of the sheet. Our studies of these sheets, especially under polarized light, have led us to the conclusion that the material is a mass of plastic crystals which are deformed by the rolling, and while they remain adherent after being thus deformed, there is a difference in the structure of the material at the crystal boundaries which we are able detect under polarized light. It appears that there is some tendency of the crystals to reorient themselves, giving rise to brittleness on aging.

We have now discovered that the above indicated difficulties can be in large measure overcome by producing the ingots in the form of large single crystals. Alternatively, we may produce ingots consisting of relatively few large crystals as compared to previous practice where the crystals were small. We prefer that each ingot be a single crystal, but the benefits of the invention may be realized in considerable degree if the crystals are large as compared with previous practice, for example, each crystal having a volume of not less than one cubic centimeter. In previous practice, the crystals have had a volume of the order of 0.01 cubic centimeter. We

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find that by using these large crystals, preferably single crystals, we are able to produce optical sheets with less annealing between successive reductions in the rolling process and such sheets exhibit less age hardening and remain pliable for long periods of time.

In the drawings, Fig. 1 is an elevation of an ingot of silver chloride according to the invention; Fig. 2 is a section on the line 2—2 of Fig. 1; Fig. 3 is a fragmentary elevation showing the ingot of Fig. 1 passing into the form of a slab between the dies of a hydraulic press; Fig. 4 is a diagrammatic illustration of reduction of thickness of the slab of Fig. 3 by passing the same between rollers and Fig. 5 is a diagram illustrating the final rolling of the optical sheet between highly polished stainless steel sheets for the purpose of effecting the final reduction in thickness and producing an optical surface.

In preparing silver chloride ingots composed of single crystals or a few large crystals, we first melt silver chloride, preferably of the highest purity obtainable, and hold it at 550° C. for several hours. During this period, considerable fining takes place and the color is much improved. The so-treated silver chloride is then rapidly filtered through Pyrex glass wool and the filtered melt is run into a special crucible in which the single crystal is to be produced. The crucible may be an ordinary Pyrex beaker, or it may be composed of porcelain or silica. The beaker preferably has a spherical bottom.

The cooling of the melt in such a way as to cause a single crystal to form (sometimes a plurality of large crystals) may be carried out in accordance with methods which have been previously employed in growing large crystals of other materials, for example, sodium chloride. One method of growing such crystals is described in U. S. Patent 1,793,672 to P. W. Bridgman, and a similar method is disclosed in U. S. Patent 2,149,076 to D. C. Stockbarger. These methods essentially consist in moving the crucible which contains the melt very slowly from a chamber at a temperature above the melting point of the material into a chamber at a temperature below the melting point of the material, the temperature gradient between the two chambers being very sharp.

We have made single crystals of silver chloride up to 10 pounds weight in a period of about 72 hours. When the growth of the crystal is complete, the crystal is removed from the crucible by melting the surface which is in contact with the crucible without melting the entire crystal.

A satisfactory process of melting out is described by Kremers in Industrial & Engineering Chemistry 32, 1478 (1940). We have found that the single crystals of silver chloride so prepared are unique in that they do not need to be annealed prior to processing. For example, we have removed a five pound single crystal of silver chloride from the crucible, and by means of a hydraulic press reduced it to a slab ½ to ¾" thick and then rolled it down to ½ mm. in thickness without any annealing. A plastic optical sheet of silver chloride several feet in length may thus be obtained from the original single crystal and will be free from crystal boundaries throughout. Sheet produced from a single crystal can even be cold worked all the way down from the ½" to ¾" slab, above referred to, to sheet ½ mm. or less in thickness. Intermediate aging between pressing into the slab and rolling or between successive rolling operations does not produce age hardening or brittleness on standing. Optical material so produced is also free from cloudiness and dross.

In rolling these plastic optical materials into sheet form, it is desirable to place the sheets between thin, highly polished sheets of hard metal, such as stainless steel, so as to produce an optical surface thereon. An optical surface may be produced by the usual means of cutting and polishing, but in the case of thin sheet material the method outlined is much easier than polishing the thin sheet.

While we prefer to make use of single crystals as against using a mass composed of a plurality of large crystals, it sometimes occurs, contrary to the wishes of the operator, that an ingot will come out multiple, that is, two or three or even a half dozen crystals will form instead of one. We are able by the use of such material to realize the benefits of the invention in large measure although the single crystals are superior.

The preferred process for producing optical sheet according to the invention is illustrated in the accompanying drawing. The highly purified silver chloride is introduced into a crucible which may be of the shape indicated in Figs. 1 and 2. Upon proper cooling of the melt, an ingot 10 is produced and under most favorable circumstances will result in a single crystal. Sometimes the crystallization will not be fully successful and the ingot 10 will contain a few crystal boundaries, that is, several crystals will be produced instead of a single one. The ingot 10 may be pressed into a slab by means of a hydraulic press provided with suitable dies 11 and 12. The resulting slab may then be passed between rollers 13 and 14 until reduced to approximately the final thickness desired. The last pass or the last several passes between the rollers 13 and 14 may be accompanied by placing highly polished stainless steel sheets on each side of the silver chloride sheet 10.

This will result in a very smooth optical surface having the appearance of having been highly polished.

The resulting optical sheet, like the optical bodies referred to in the literature cited above, possesses high transparency to infra-red rays but is free from the optical imperfections growing out of the presence of crystal boundaries, or if the original ingot was composed of several crystals, it is relatively free from such imperfections.

Having thus described our invention, what we claim is:

1. A process of making an optical body comprising making an ingot of volume in excess of one cubic centimeter consisting of a single crystal of silver chloride and then forming an optical body of the desired shape by deforming such ingot.

2. A method of making an optical body comprising forming an ingot of silver chloride consisting of crystals of volume in excess of one cubic centimeter each and then forming an optical body of the desired shape by deforming such ingot.

3. A process of making optical silver chloride sheet comprising deforming a silver chloride ingot composed of crystals having a volume in excess of one cubic centimeter each, a portion at least of the deformation being accomplished by passing the silver chloride between rollers a plurality of times and materially reducing the thickness each time.

4. A process of making optical silver chloride sheet comprising deforming a silver chloride ingot composed of a single crystal of volume in excess of one cubic centimeter, a portion at least of the deformation being accomplished by passing the silver chloride between rollers a plurality of times and materially reducing the thickness each time.

5. A process of making optical silver chloride sheet comprising forming an ingot of silver chloride consisting of a single crystal of volume in excess of one cubic centimeter, pressing the resulting ingot into a slab and then rolling the resulting slab into a sheet of the desired thickness.

6. A process of making an optical body comprising making an ingot of volume in excess of one cubic centimeter and of crystal size in excess of one cubic centimeter volume per crystal, the same being composed of a material of the class consisting of silver halides and mercurous chloride and then deforming such ingot to the desired shape.

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The following references are of record in the file of this patent:

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