

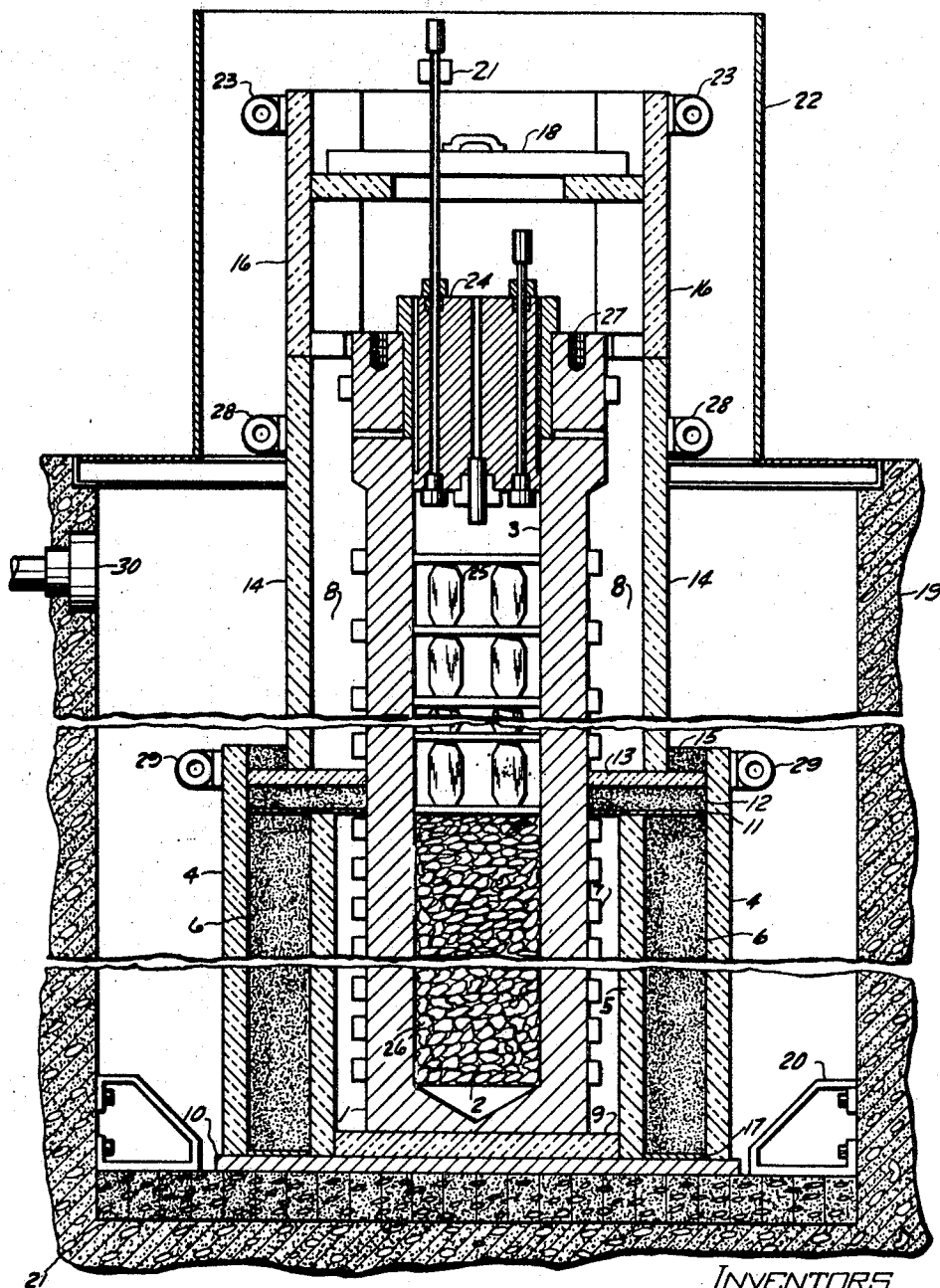
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AUTOCLAVE FOR GROWING QUARTZ CRYSTALS

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1

2

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AUTOCLAVE FOR GROWING QUARTZ CRYSTALS
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This invention relates in general to autoclaves and more particularly to autoclaves for growing quartz crystals.

The piezoelectric property of quartz whereby changes in crystalline structure are caused by applied voltages and the reverse reaction whereby tension and compression strains induce voltages, makes quartz particularly useful as a transducer to convert between different forms of energy. However, the efficiency of conversion between mechanical and electrical energy varies quite sharply with frequency. Since the frequency of maximum efficiency is determined by the dimensions of the quartz, it is possible to use crystal units as filters to select desired frequencies. A variety of useful devices can be derived from the properties of quartz and crystal units have many applications in electronic apparatus.

Natural quartz crystals of the size and quality required for electronic apparatus are quite scarce. Suitable crystals to meet the large industrial demand are found only in remote areas of Brazil. This source of supply is unpredictable because the mining operations are scattered over wide areas in the interior of the country and the quartz stones are usually mined by individuals on a free-lance basis since there are no large mining operations. Another factor, noted during World War II, was the danger that this supply might become unavailable or its effectiveness reduced.

Quartz plates made from natural quartz are quite expensive since adding to the cost is the fact that a great part of the quartz is lost during production due to imperfections in the stones and irregularity in the shape which requires further processing to conform to the dimensions of the finished plates. On the other hand, the technique of growing synthetic crystals has progressed to the point where the synthetic crystals have fewer cracks and impurities and because of their uniform dimensions are particularly suited to modern production methods. The development of man-made crystals provides a reliable source of crystal units for electronic applications at a somewhat lower cost.

The successful growing of quartz crystals is dependent upon several factors, including the ability to maintain two isothermal zones within a vertically extending autoclave. The bottom or nutrient zone where the quartz chips go into solution must be maintained at a temperature considerably above the upper or growing zone holding the seed plates. The heat input to the zones and the heat losses must be carefully controlled to maintain the two isothermal zones in a substantially constant thermal condition.

The object of this invention is an autoclave for growing quartz crystals having two isothermal zones with a sharp temperature gradient between the zones.

In accordance with the general features of this invention, the upper and lower zones of the autoclave for growing quartz crystals are maintained at different temperatures by carefully controlling the heat input and heat losses. The autoclave insulation is designed to effect isothermal conditions in the two zones by producing a sharp temperature gradient between the zones and by controlling the aforementioned heat losses. The lower zone insulation is a complete package which is easy to remove and re-

place. It consists of two concentric inner and outer walls of a soft structural insulating material such as an asbestos combination sheet type with a material or high insulation value between the walls. The inner wall is spaced away from the heater to provide breather space and prevent burn-outs due to "hot spots."

The upper zone is similarly insulated with a wall of insulating material such as an asbestos combination sheet type and is readily detachable from the lower zone insulation. The upper zone insulation is composed of detachable sections, an upper section and a lower section, and a removable cover lift which facilitates cooling. The temperature differential between zones is sharpened by the design of the intervening insulation. The zones are separated by a thin annular metal plate covered on top with high insulation value material and an annular plate of insulating material of an asbestos cement type which supports the upper zone and another layer of high insulation value material.

The package units of insulation used in the autoclave are readily serviced and interchangeable, and reproduce the exact properties required in successive runs.

Other objects and advantages will be apparent from the following detailed description when considered in conjunction with the accompanying cross-sectional view of the autoclave for growing quartz crystals. The drawing has been broken to adequately present the main features therein but in practice the autoclave itself is approximately 10 feet in length.

With reference to the drawing, the autoclave 1 for growing quartz crystals has two zones, a bottom or nutrient zone 2 and an upper or growing zone 3. The bottom zone is maintained isothermally by employing two circular insulating walls or boxes, an outer 4 and an inner 5, with a high insulating value material 6 between the two to minimize heat losses and a metal base 17 to contain the intervening insulation. The inner wall 5 is spaced away from the heaters 7 to provide breathing space 8 and thus prevent burn-outs. The bottom zone is secured to an insulating pad 9 and a metal base 10 which rests on fire-brick 21.

A steep temperature differential between the zones is obtained by controlling the heat input and by using a thin annular metal plate 11 covered on top with high value insulation 12 and an annular baffle 13 of an insulating sheet material which supports the upper insulation 14 and another layer of high value insulation 15. The upper zone has a detachable insulating top box 16 and a cover lift 18 to facilitate cooling when the autoclave is shut down.

For safety reasons, due to the high pressure developed in the autoclave, it is placed in a concrete lined pit 19, using flanges 20 to guide it into position. On completion of a run, the heating of the autoclave is discontinued and auxiliary cool-down apparatus is inserted through the door 21 in the protective metal shield 22. After the cool-down period, the protective metal shield 22 is first removed and then the upper top box section of insulation 16 is removed by means of eye bolts 23 revealing the autoclave head 24. An operator can then proceed to withdraw the synthetic crystals 25 and recharge the autoclave with seed crystals and nutrient 26 without disturbing the autoclave insulation.

For maintenance purposes on the autoclave or to service the insulation, the above steps are repeated and the autoclave including its insulation is picked up by means of lifting bolts 29 after the electrical connections have been disconnected from the control box 30. The autoclave and the packaged insulation are taken from their concrete lined pit 19 to an inspection pit where the upper zone insulation 14 is removed by grasping the eye bolts 28 provided for that purpose and lifting straight up. The baffle

3

parts 11, 12, 13, and 15 are removed by hand and the lower zone insulation 4, 5, and 6 is lifted up as a unit by eye bolts 29 leaving the autoclave ready for maintenance.

It is to be understood that the above described arrangements are simply illustrative of the application of the invention. Numerous other arrangements may be readily devised by those skilled in the art which will embody the principles of the invention and fall within the spirit and scope thereof.

What is claimed is:

1. An apparatus for growing quartz crystals including an autoclave having an upper crystal growing zone and a lower nutrient zone, means for maintaining the two zones under isothermal conditions with a steep temperature gradient between the zones, and means for holding quartz crystal seeds, comprising:

- inner and outer circular walls of insulation surrounding and spaced away from the lower zone of the autoclave;
- loosely arranged material of high insulation value positioned between the walls;
- an insulating circular wall surrounding and spaced a predetermined distance from the upper zone of the autoclave;
- a heating element surrounding and fixed to the autoclave at each crystal growing zone;
- means separating the insulation and the heating elements of the two zones including an annular metal plate, loosely arranged high insulation value material on said plate, and an annular plate of insulating material above the loosely arranged insulating material supporting the lower end of the insulating wall of the upper zone, the upper end of the outer wall of the lower zone overlapping the lower end of the insulat-

4

ing wall of the upper zone and being spaced therefrom; and

loosely arranged insulation on the annular insulating plate in the space between the overlapped wall ends.

2. An apparatus according to claim 1 wherein:

- the wall for the upper zone comprises two detachable sections, one resting on top of the other,
- a removable cover for the top section,
- an outer removable metal shield surrounding the top section,
- the inner and outer walls forming part of a detachable unit, and
- the detachable sections and unit, each respectively including means for facilitating their separate handling.

References Cited by the Examiner

UNITED STATES PATENTS

1,646,213	10/27	Otis	263—46
1,698,480	1/29	Mulholland	263—46
1,879,676	9/32	Fulcher	263—46
2,146,429	2/39	Hawkins	263—46
2,631,091	3/53	Kuentzel et al.	23—290
3,051,558	8/62	Jost	23—273

OTHER REFERENCES

"Natural Quartz from the Laboratory" by Corwin, Journal of Chemical Education, vol. 37, No. 1, January 1960, pages 11 to 14.

Lawson: Journal of Applied Physics, vol. 22, #12, December 1951, pages 1444 to 1447.

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