Nov. 26, 1963 YOSHIHIRO SHIMULA 3,112,398

DEVICE FOR COOLING THE SPECIMEN IN X-RAY DIFFRACTION APPARATUS

Filed Feb. 14, 1961

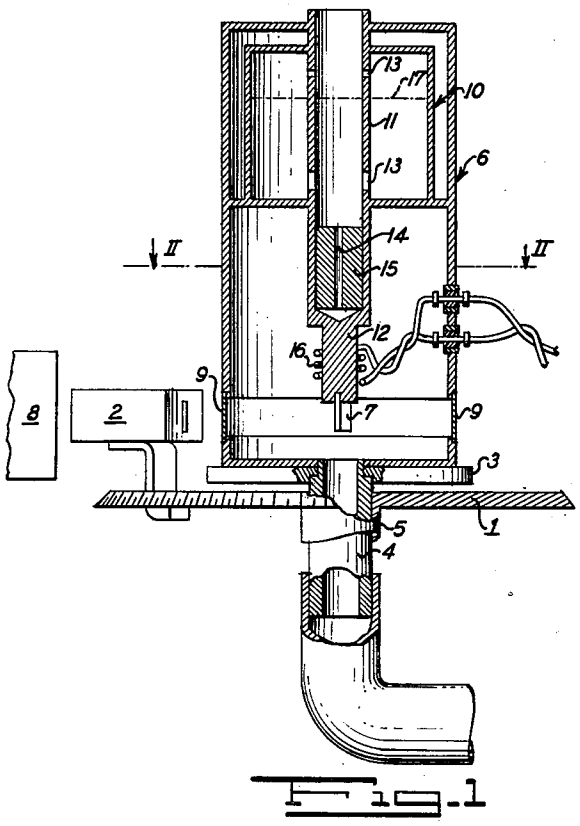


Fig. 1

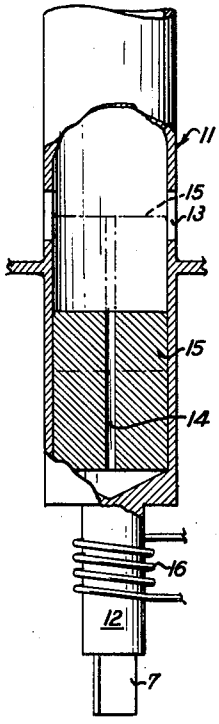


Fig. 3

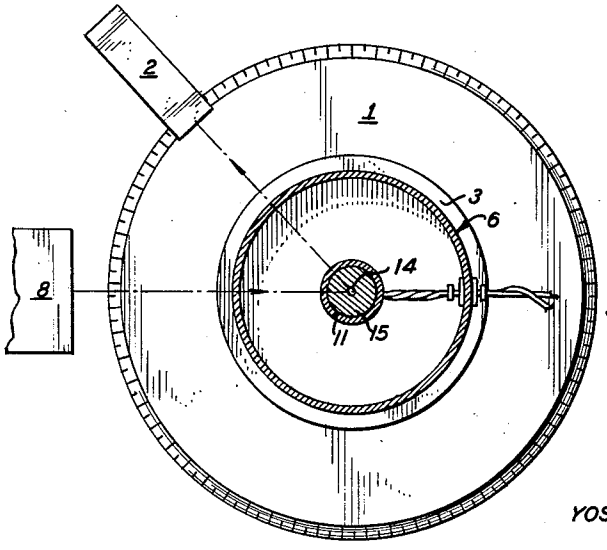


Fig. 2

INVENTOR

YOSHIHIRO SHIMULA

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DEVICE FOR COOLING THE SPECIMEN IN X-RAY DIFFRACTION APPARATUS

Yoshihiro Shimula, 19 Tenjincho, Nakano-ku, Tokyo, Japan

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This invention relates to cooling devices and more particularly to devices to cool a specimen set in X-ray diffraction apparatus.

For making an analysis of a specimen as to its crystal structure by employment of X-ray diffraction, it is required that the specimen be maintained at a relatively low temperature between ambient temperature and a temperature close to absolute zero, or that the temperature of such specimen be continuously varied within these limits.

Means have been proposed and utilized for the above purpose, wherein an end of a thermally conductive metal rod is soaked in a cooling medium such as liquid air, a specimen to be subjected to measurement is attached to the other end of said rod whereby said specimen is cooled, and a heating wire is wound onto the specimen and the metal rod whereby the temperature of the specimen is adjusted.

Such means, however, were deficient in that the direct winding of electric heating wire around the specimen not only causes lack of uniformity in temperature in the specimen at various portions thereof, but also the wire interrupts the rays during the X-ray application. Another disadvantage is that, as the electric heating wire wound onto said metal rod is heated, the evaporation of the employed cooling medium will be abruptly intensified and, when such evaporation is controlled to a certain degree, desired variations in the temperature of the specimen covering a wide range is not achievable.

It is an object of the present invention to provide a cooling device for a specimen set in an X-ray diffraction apparatus avoiding such defects as described above.

Another object of this invention is to provide a device for readily varying the temperature of a specimen subjected to measurement within desired temperature limits in an X-ray diffraction apparatus.

A further object of the present invention is to provide a device to cool a specimen provided in an X-ray diffraction apparatus, wherein the device is capable of providing a great temperature difference between the specimen and the cooling medium being employed with a small calorific value by utilizing the evaporation of the cooling medium, consequently varying the temperature of the specimen through a wide range without excessively evaporating the refrigerant due to the heat of a heating means.

Still a further object of this invention is to provide a device for cooling a specimen in an X-ray diffraction apparatus, which device is simple and accurately operable.

The advantages as described heretofore, other merits and the novel feature of the present invention will be fully understood from the following description made in conjunction with the accompanying drawings of an embodiment of this invention. In the accompanying drawings:

FIGURE 1 is a front view in longitudinal section of an improved specimen cooling device in the X-ray diffraction apparatus according to this invention;

FIGURE 2 is a view in section, taken on the line II—II of the device shown in FIGURE 1, and;

FIGURE 3 is an enlarged view in section of a portion of the device shown in FIGURE 1.

In the X-ray diffraction apparatus shown, a Geiger-Muller counter 2 is mounted at the side edge of a goniometer 1, and a shaft 4 of a specimen bed 3 is passed through

the shaft 5 of the goniometer 1 coaxially therewith, so that the goniometer 1 and the specimen bed 3 are in an interlocking revolution with an angular velocity ratio of 2 to 1. An airtight casing 6 is mounted on the specimen bed 3 and a specimen 7 provided in a plate shape is set in the center. The X-rays are applied to the specimen 7 from the X-ray tube 8 secured to the side of the goniometer 1 through the X-ray transmitting window 9 of the casing 6 as shown with chain lines in the drawings, whereby the X-ray diffracted at the surface of the specimen is detected by the counter 2. Since the specimen 7 and the counter 2 are rotated at an angular velocity ratio of 2 to 1, by prearranging the surface of the specimen in a position normal to the bisector of an angle formed by straight lines drawn to the center of the goniometer 1 respectively from the target of the X-ray tube and the front slit of the counter, said diffracted X-rays are constantly incident upon the counter, and thus the relationship between the X-ray diffraction angle of the specimen and the intensity of the diffracted X-ray is obtainable.

In the airtight casing 6, the upper surface of the upper bottom is hermetically sealed with a thermos 10 and the interior thereof is made vacuous. The thermos 10 is provided with a metal cylindrical body 11 formed of a material such as stainless steel at the center portion of the thermos. Said specimen 7 is so secured as to adhere closely to the end of the projection 12 formed at the lower end of the metal cylindrical body 11. The metallic cylindrical body 11 is provided with apertures opening into the interior of the thermos, together with a tubular metallic float of material such as stainless steel having a small hole extending therethrough from the top to the bottom, which float is inserted into the cylindrical body closely fitting relation but slidably movable therein. Electric heating wire 16 is coiled about the lower end of the projection 12.

The interior of the thermos 10 is filled with any suitable cooling medium such as liquid air or the like, and such cooling medium fills also the inner portion of the metallic cylindrical body 11. The metallic float 15 sinks to the bottom of said cylindrical body as shown in the drawings with solid lines, until an electric current is applied to the electric heating wire 16. The casing 6 to admit the lower portion of the cylindrical body 11 and the specimen 7, is kept vacuous, for example, by means of a vacuum conduit passing through the shaft 4. Therefore, the thermal content of the specimen is effectively removed by the cooling medium 17 and by conduction through the projection 12 and the float 15. Moreover, since transmission of heat from the outside is very limited, the specimen is cooled down to a temperature close to that of the cooling medium.

Upon application of an electric current to the heating wire 16, however, since the heat generated by the heating wire is removed by the cooling medium through the processes described above, a great variation in temperature is caused between the bottom portion of the cylindrical body 11 and the portion upon which is coiled the heating wire 16, whereby the temperature of the specimen 7 rises. Besides, because a large amount of heat discharged from the heating wire 16 passes to the cooling medium in the bottom section of the cylindrical body 11, whereat it collects after passing through the small hole 14 of the float 15, vaporization of the cooling medium in this section is effected and a vapor layer is formed in the section under the float 15. Consequently, the float is pushed upward by the vapor pressure to the position shown in FIG. 3 with chain lines. Thus, the thermal content of the specimen 7 and the heating wire 16 will pass to the cooling medium through the tubular portion 12, the lower side wall of the cylindrical body 11 and the float 15. Since, however, the side wall of the cylindrical

body 11 is formed of a relatively thin metal sheet having a small cross-sectional area with a strong resistance to thermal conduction, a great variation in temperature developed at this portion will further expedite the rise of temperature of the specimen, and the vaporized coolant will be scattered over the liquid surface of the coolant. In other words, the float is located at the bottom of the cylindrical body 11 when the heating wire is not being energized with current, so that the thermal content of the specimen 7 is then transmitted to the cooling medium 17 from the tubular portion 12 directly through the float 15 having a large sectional area, whereby the specimen is effectively cooled to a very low temperature.

However, upon application of an electric current to the heating wire, the calorific content of the heating wire is transmitted to the coolant, so that the said float is pushed upward by the vapor pressure, whereupon, the thermal content of the specimen and of the heating wire is absorbed by the coolant solely through the wall of the cylindrical body having a small sectional area. Consequently the thermal resistance is increased, so that the temperature of the specimen rises. The temperature of the specimen is therefore variable as desired by means of regulating the electric current applied to the heating wire.

As set forth heretofore, with the present invention it is intended that the float immersed in the cooling medium be moved by utilizing the vaporization of the cooling medium. As the thermal content is varied by the heat of such heating means as the heating wire 16, simultaneously the thermal conductive resistance is also varied, whereby a large difference in temperature between the specimen and the cooling medium is developed with a small calorific value. Accordingly, an excellent effect in variation of the temperature of the cooling medium covering very wide range is obtainable without any fear of excessive vaporization of the cooling medium by the heat discharged from the heating device employed.

While one embodiment of this invention in conjunction with the X-ray diffraction apparatus has been described for the purpose of illustration, it should be understood that the invention is not limited to the exact apparatus illustrated and described since modifications thereby may be suggested by those skilled in the art, without departure from the scope of the invention.

What I claim is:

1. Apparatus for controllably varying the temperature of a specimen; said apparatus comprising support means of thermally conductive material for supporting said specimen, heating means operatively associated with said support means for controllably heating the latter and thereby said specimen, cooling means adapted for lowering the temperature of said specimen, and connecting means connecting said cooling means to said support means for removing heat from the latter and thereby from said specimen, said connecting means including means between said support means and cooling means and having a thermal conductivity which varies in response to heat generated by said heating means, the latter means comprising a tube of thermally conductive material from which is suspended said support means, a float of thermally conductive material displaceable in said tube away from said support means, and a fluid in said tube beneath said float and adapted for being vaporized by heat applied to the fluid for raising the float in the tube to modify the combined heat conductivity of the float and tube.

2. Apparatus as claimed in claim 1 wherein said cooling means comprises a container and a cooling fluid in said container, said cooling fluid also constituting the fluid in said tube.

3. Apparatus as claimed in claim 2 wherein the heating means is a heating coil mounted on said support means.

4. Apparatus as claimed in claim 3 wherein said cooling means is a thermos arrangement on and encircling said tube and wherein said tube is provided with at least one opening communicating with said thermos arrangement whereby said fluid passes freely therebetween.

5. Apparatus as claimed in claim 4 wherein said float is provided with a bore passing therethrough for the passage of said fluid.

6. Apparatus as claimed in claim 5 comprising X-ray generating means and measuring means operatively associated with said support means for determining the crystal structure of said specimen.

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