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HIGH TEMPERATURE FURNACE FOR X-RAY DIFFRACTOMETER

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Fig. 1.

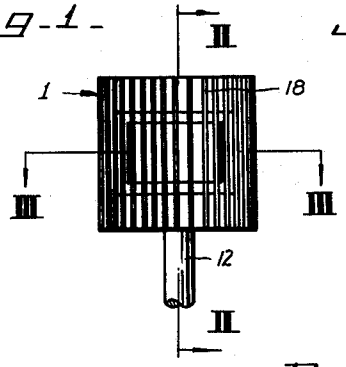


Fig. 2.

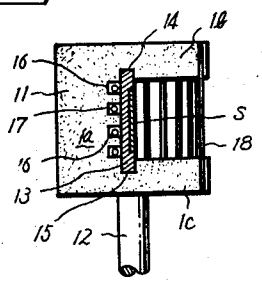


Fig. 3.

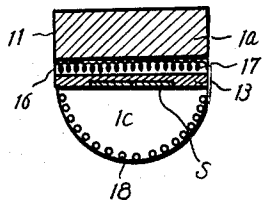
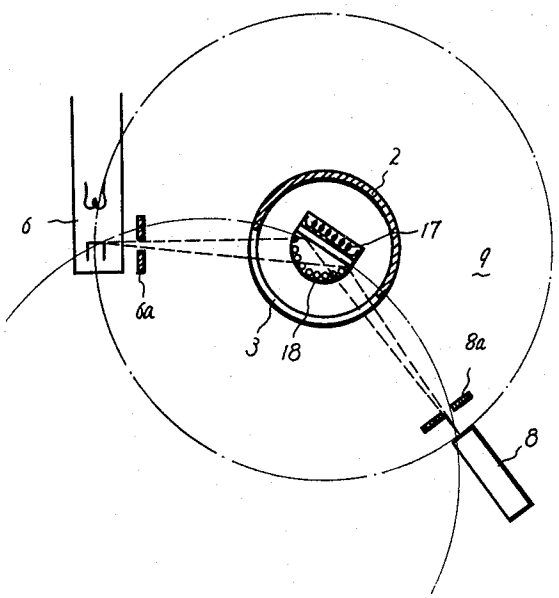


Fig. 5.



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Fig. 4

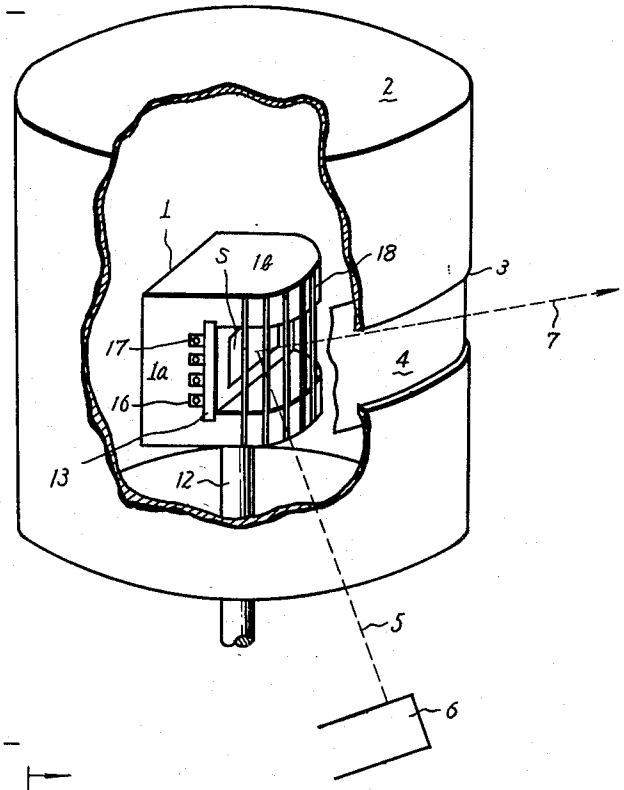
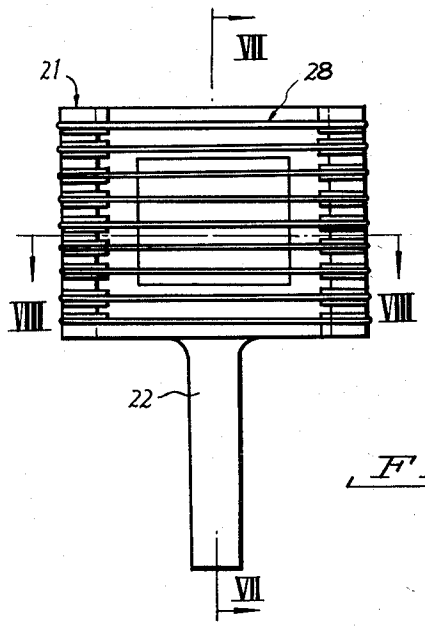


Fig. 6



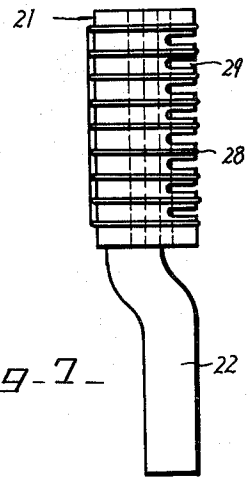


Fig. 7

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Fig. 8

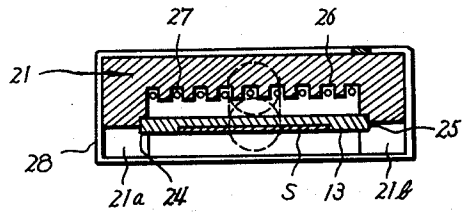


Fig. 9

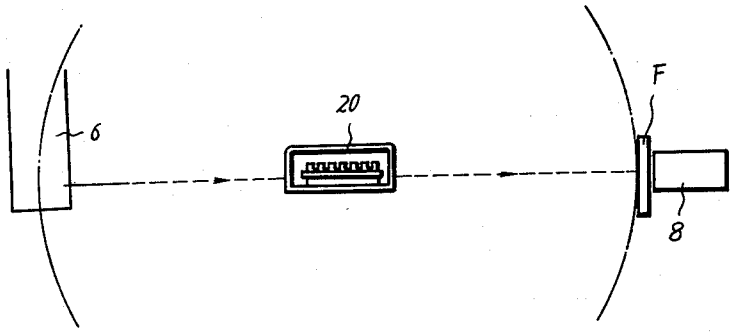
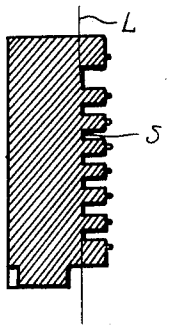


Fig. 10



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Fig. 11.

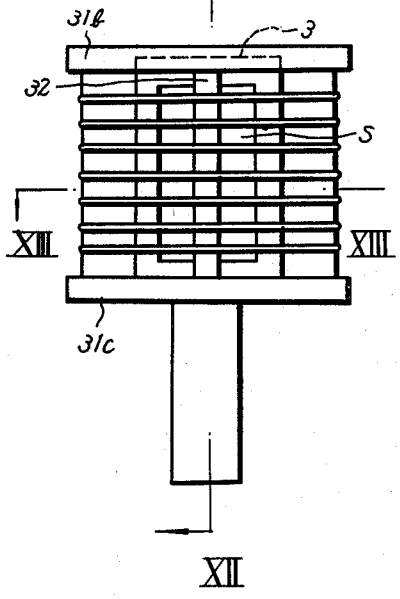


Fig. 12.

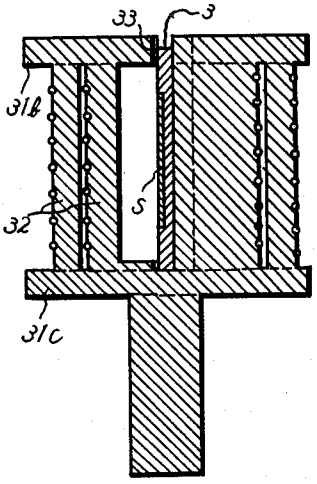


Fig. 13.

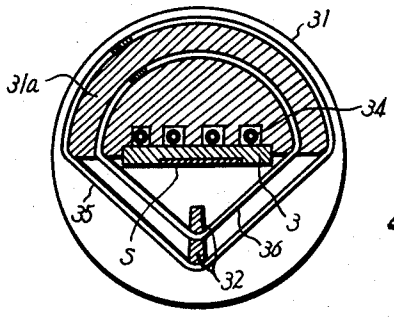
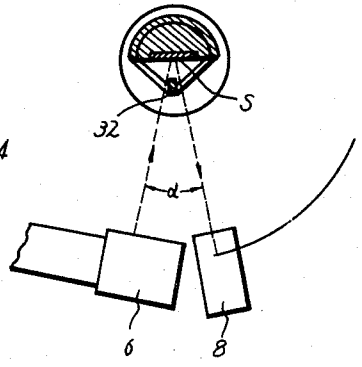


Fig. 14.



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HIGH TEMPERATURE FURNACE FOR X-RAY DIFFRACTOMETER

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4 Claims. (Cl. 250—51.5)

The present invention relates to high temperature furnaces for an X-ray diffractometer.

It is already well known to use an X-ray diffractometer to determine the crystal structure of a specimen by means of X-rays. The X-ray diffractometer generally consists of an X-ray generator, a goniometer on which the specimen is mounted, an X-ray detector and an electronic circuit. For the purpose of investigating the crystal structure at elevated temperatures or for investigating the change of the crystal structure in accordance with variation of the temperature, it is necessary to heat the specimen which is to be investigated. A principal object of the present invention is to provide an improved high temperature furnace for an X-ray diffractometer.

It has been difficult to heat the surface of a specimen irradiated by X-ray beams to a desired elevated temperature in the past since the specimen is generally heated from the opposite surface by means of a heating wire.

It is a further object of the present invention to provide an improved furnace for heating the surface of specimen irradiated by X-ray beams to a desired temperature.

It is a further object of the present invention to provide a furnace which uniformly heats the entire specimen.

It is a further object of the present invention to provide a device for heating the specimen from the front of its surface irradiated by X-ray beams without disturbing the beams incident on or diffracted by the surface of the specimen mounted on the goniometer.

It is a further object of the present invention to provide apparatus for heating the specimen to the desired temperature and for determining the crystal structure by an X-ray diffraction method.

It is a still further object of the present invention to provide simple devices for heating the specimen in order to measure the crystal structure in accordance with the variation of temperature.

It is yet another object of the present invention to provide a high temperature furnace for an X-ray diffractometer, which is capable of adjusting the position so as to satisfy the Bragg equation.

Other objects and advantages of the invention will be apparent during the course of the following description and in order that the invention may be more readily understood and carried into effect, the invention is hereinafter described by way of example and with reference to the accompanying drawings which illustrate preferred embodiments of the invention.

FIG. 1 is an elevation view showing an embodiment of the furnace according to the invention;

FIG. 2 is a view in sectional elevation along the line II—II of FIG. 1;

FIG. 3 is a sectional view along the line III—III of FIG. 1;

FIG. 4 is a perspective view of the furnace shown in FIG. 1, the furnace being housed in a shield casing a portion of which is broken away;

FIG. 5 is a schematic diagram of an X-ray optical system to be used in the determination of the crystal structure in employing the furnace illustrated in FIG. 1;

FIG. 6 is an elevation view showing a second embodiment of the present invention;

FIG. 7 is a vertical section taken on line VII—VII of FIG. 6;

FIG. 8 is a sectional view taken along line VIII—VIII of FIG. 6;

FIGS. 9 and 10 illustrate the method of adjustment in mounting the heating device shown in FIG. 6;

FIG. 11 is an elevation view showing a third embodiment of the present invention;

FIG. 12 shows a sectional elevation view taken along line XII—XII of FIG. 11;

FIG. 13 is a sectional view taken on line XIII—XIII of FIG. 11; and

FIG. 14 illustrates the furnace shown in FIG. 11, the furnace being mounted on a goniometer in order to measure the crystal structure and an X-ray tube and a counter are shown close to each other at a minimum angle.

Referring to the drawing in detail, and in particular to FIGS. 1, 2 and 3, showing a first embodiment of the furnace according to the present invention, the furnace is generally denoted by numeral 1. A base body 11 of the furnace 1 consists of a vertical rectangular portion 1*a* and half-circular end portions 1*b* and 1*c* projecting outwardly from the upper and lower ends of the vertical portion 1*a*. The three portions 1*a*, 1*b*, 1*c* are made of heat resisting material such as alumina and are formed as a one piece body. The base body 11 has a shaft 12 to be mounted on a goniometer. The specimen S to be studied by X-ray diffraction and to be heated is mounted on a specimen supporting member 13, which is mounted in the furnace 1. For this purpose, there are grooves 14 and 15 in the inner corners formed by the vertical portion 1*a* and the half-circular projected portions 1*b* and 1*c* in order to insert the specimen supporting member 13 into these grooves from the side of the furnace 1. The specimen supporting member 13 is formed as a dish having a depression formed in the center portion of a surface. Mounted in the depression of the member 13 is the specimen to be investigated which is in the form of the plate or powder and the outer surface of the specimen supported in the depression of the member 13 is coincident with the outer surface of the member 13.

The member 13 is mounted in the furnace in such a way that the extension of the axis of the base body 11 and the shaft 12 passes through the surface of the specimen attached to the member 13. A plurality of grooves 16 are arranged on the vertical surface of the base body 11 adjacent to the specimen supporting member 13 and these grooves are in parallel, separated from each other and arranged at a right angle to the axis of the shaft 12. The electric heating wires 17 which are made of, for example, platinum-rhodium and are used for heating the specimen from the back side thereof are positioned in the grooves 16. Extending between the upper projected portion 1*b* and the lower projected portion 1*c* are a plurality of electric heating wires 18, which are made of the same material as the wire 17 mentioned above, to heat the specimen from the front thereof. These heating wires may be connected in series with each other and connected to a suitable electric source which is not shown in the drawing. By providing grooves in the periphery of the half-circular portions 1*b* and 1*c* and inserting the wires therein, it is possible to avoid movement of these wires.

FIG. 4 is a view in perspective, showing the furnace 1, as above described mounted in a shield case 2. It is, however, needless to say that the furnace according to the present invention can be employed without using such a shield case.

The shield case 2 is made of metallic material such as, for example, nickel, the shield case having the form of a cylinder which is provided with a window 3 at the center portion of the side of the cylinder extending through about a half-circle thereof. The window 3 is covered by metal foil 4 as, for example, nickel having a suitable thickness to admit X-ray beams. The shield case makes it possible to minimize the thermal radiation from the furnace. The X-ray beam 5 coming from an X-ray tube 6, passes through the window 3 of the shield case 2 and

then falls upon the surface of the specimen S mounted in the furnace 1. The beams 7 reflected from the specimen pass through the window 3 of the shield case 2 and into a counter 8, which is shown in FIG. 5.

FIG. 5 illustrates an X-ray optical system having the furnace 1 showing in FIG. 1 mounted on a goniometer in order to investigate the crystal structure by X-ray diffraction.

The furnace 1 is longitudinally fixed to a shaft of the goniometer by means of the shaft 12 of the furnace so as to rotate the furnace together with the shaft of the rotating table of the goniometer. On the periphery of the rotating table of the goniometer, a counter 8 such as a Geiger-Müller counter is mounted and the X-ray tube 6 is positioned to the side portion of the goniometer. The distances from the center of the specimen mounted in the furnace to the target of the X-ray tube and to the front surface of the counter are of the same length.

In the X-ray diffractometer using the X-ray optical system shown in FIG. 5, the Bragg-Brentano focusing method is applied, and thus the intensity received by the counter is increased.

An X-ray beam coming from the target of the X-ray tube 6 passes through a slit 6*a* and through the window 3 of the shield case and then falls upon a surface of the specimen mounted in the furnace 1. The rays reflected from the specimen pass through the window 3 and through another slit 8*a* and into the counter 8. The specimen and the counter are rotated about the same central axis by means of a synchronous motor and a gear mechanism at the rotational ratio of 1 to 2. Since the counter is always inclined at the angle of 2θ from the beam incident upon the surface of the specimen when it makes an angle of θ between the surface of the specimen and the incident beam, the incident rays on the specimen are diffracted when the Bragg equation is satisfied and the diffracted X-ray is received by the counter by which the intensity of the reflected X-ray is measured.

In the above mentioned procedure, according to the present invention, the specimen S is heated by the heating wires 17 from the back thereof and by the heating wires 18 from the front of specimen surface. The specimen S may be heated by these wires to elevated temperature, for example at about 1500° C., and maintained substantially at uniform temperature as a whole. It is, therefore, capable of measuring precisely the crystal structure under the condition of the desired elevated temperature.

Employing the shield case 2 together with the furnace 1 according to the invention increases the heat efficiency in the furnace and prevents the heating of the means arranged outside of the furnace because the heat dissipation from the furnace is kept by the shield case 2 to a minimum.

In using the furnace of the invention, the beams from the X-ray tube 6, passing through the gaps of the heating wires 18 arranged in the front of the specimen S, reach the surface of the specimen and also the reflected ray from the specimen surface, passing through the gaps of the heating wires 18, reach the counter 8. Thus, it is clear that a large amount of beams fall upon heating wires 18 and are reflected therefrom. Since the heating wires 18 are arranged at intervals from the specimen surface, as mentioned above, and their positions are improper to satisfy the Bragg equation, the diffracted X-ray from the heating wires 18 do not enter the counter 8.

The surface of the specimen S mounted in the furnace is properly positioned with respect to the rotating table 9 if the surface of the specimen is aligned with a straight line connecting the slit 6*a* with respect to the X-ray tube and the slit 8*a* with respect to the counter 8 when the X-ray tube 6 and the counter 8 are brought into diametrically opposite positions in the goniometer 9.

Referring to FIGS. 6, 7 and 8, illustrating a second embodiment of the furnace according to the invention, the cross section of the base body 21 is substantially

of C-shape consisting of a vertical flat portion and projected portion 21*a* and 21*b* and there are a plurality of grooves 26 on the inner surface of the vertical flat portion of the base body 21. The heating wires 27 are arranged in grooves 26. A plurality of heating wires 28 are wound around the outside of the base body 21 at right angles to the shaft 22 of the furnace. The specimen supporting member 13 on which the specimen S is mounted is inserted into the grooves 24 and 25 which are provided in the inner surfaces of the projected portions 21*a* and 21*b*. In addition, there are grooves 29 between the wires 28. The depth of the groove 29 extends further than the plane defined by the surface of member 13 when the member is mounted in the furnace.

FIG. 9 shows a method for mounting the furnace 20 shown in FIG. 8 on the goniometer. The X-ray tube 6 and the counter 8 are positioned diametrically opposite one another with respect to the goniometer, the furnace 20 being mounted on the goniometer and a plate or screen coated by fluorescent material on which a reference line L (FIG. 10) is indicated is arranged in front of the counter 8. If the surface of the specimen S is coincident with the reference line L, of the plates or screen F, when X-ray beams are directed from the X-ray tube to one side of the furnace 20, it will be properly positioned relative to the goniometer.

It is important in the furnace shown in FIG. 6 that the heating wires 28, which are placed in front of the surface of the specimen, be perpendicular to the shaft of the furnace. Namely, since these wires are arranged parallel to the specimen surface, X-ray disturbance caused by wires 28 is made uniform regardless of the rotation of the goniometer.

FIGS. 11, 12 and 13 show a third embodiment of the furnace according to the invention, which is capable of heating the specimen to a higher temperature than in the other embodiments.

In the third embodiment of the furnace the base body 31 consists of a half circular cylindrical portion 31*a* and upper and lower circular flange portions 31*b* and 31*c*. A center line, passing through a center of the vertical flat surface of the half cylinder, passes through a center of both flange portions.

In front of the supporting member 3 and therefrom, is a support 32 extending between the upper and lower flange portions 31*b* and 31*c*. The specimen supporting member 3 on which the sample S to be investigated is mounted is inserted into a slot 33 provided in the upper flange portion 31*b* along the vertical plane of the half cylinder portion of the base body 31.

A plurality of grooves perpendicular to the axis of the base body are arranged in the surface of the vertical flat plane of the base body. As mentioned above, these grooves serve for setting the heating wires 34 to be used for heating the specimen from its back side. The further heating wires 35 and 36 are wound parallel around the half circular surface of the cylindrical portion 31*a* and the outer side of the support 32 and are perpendicular to the axis of the base body 31.

The heating wires 36 may be arranged in the same horizontal plane as each of the heating wires 35 and arranged closer to the specimen as shown in FIG. 13.

As is clear from FIG. 14, there is an angle α between the X-ray tube to a center of the specimen and the counter 8 to a center of the specimen when the X-ray tube and the counter are adjacent to each other. Since the support 32 comes into the range of such an angle α, the beams coming from the X-ray tube to the surface of specimen and the diffracted rays from the surface of specimen to the counter are not disturbed by the support member 32.

While the invention has been described in its preferred examples and embodiments, it is to be understood that changes can be made thereto within the purview of one

skilled in the art without departing from the spirit of the invention as defined in the appended claims.

What I claim is:

1. In an X-ray diffractometer having an X-ray source and a counter, a furnace containing a specimen mounted on a supporting plate, said furnace comprising: a heat resistant base member provided with a receptacle adapted for accommodating said plate, said plate being accommodated in said receptacle with the specimen therein having a surface adapted for being irradiated by X-rays from said X-ray source, said base member being further provided with grooves adjacent said receptacle, heater coils in said grooves for heating said specimen from a side of the plate remote from the surface of the specimen to be irradiated, further heating coils on the other side of the plate and facing the surface of the specimen to be irradiated, said further heating coils being adapted to furnish additional heat to the specimen, said X-ray source being operatively positioned with respect to said specimen to irradiate the same with X-rays, the further heating coils being in the path of the X-rays and being in spaced relation with respect to one another to permit passage therethrough of X-rays both from said source and diffracted from said specimen, and means on the base member maintaining said further heating coils in position.

2. In an X-ray diffractometer as claimed in claim 1, means on said base member providing an axis of rotation therefor, said surface of the specimen to be irradiated being a flat surface substantially coplanar with said axis, said further heating coils extending parallel to said axis.

3. In an X-ray diffractometer as claimed in claim 1, means on said base member providing an axis of rotation therefor, said surface of the specimen to be irradiated being a flat surface substantially coplanar with said axis, said further heating coils extending transverse to said axis.

4. In an X-ray diffractometer as claimed in claim 1 wherein the first said coils are perpendicular to said further coils.

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