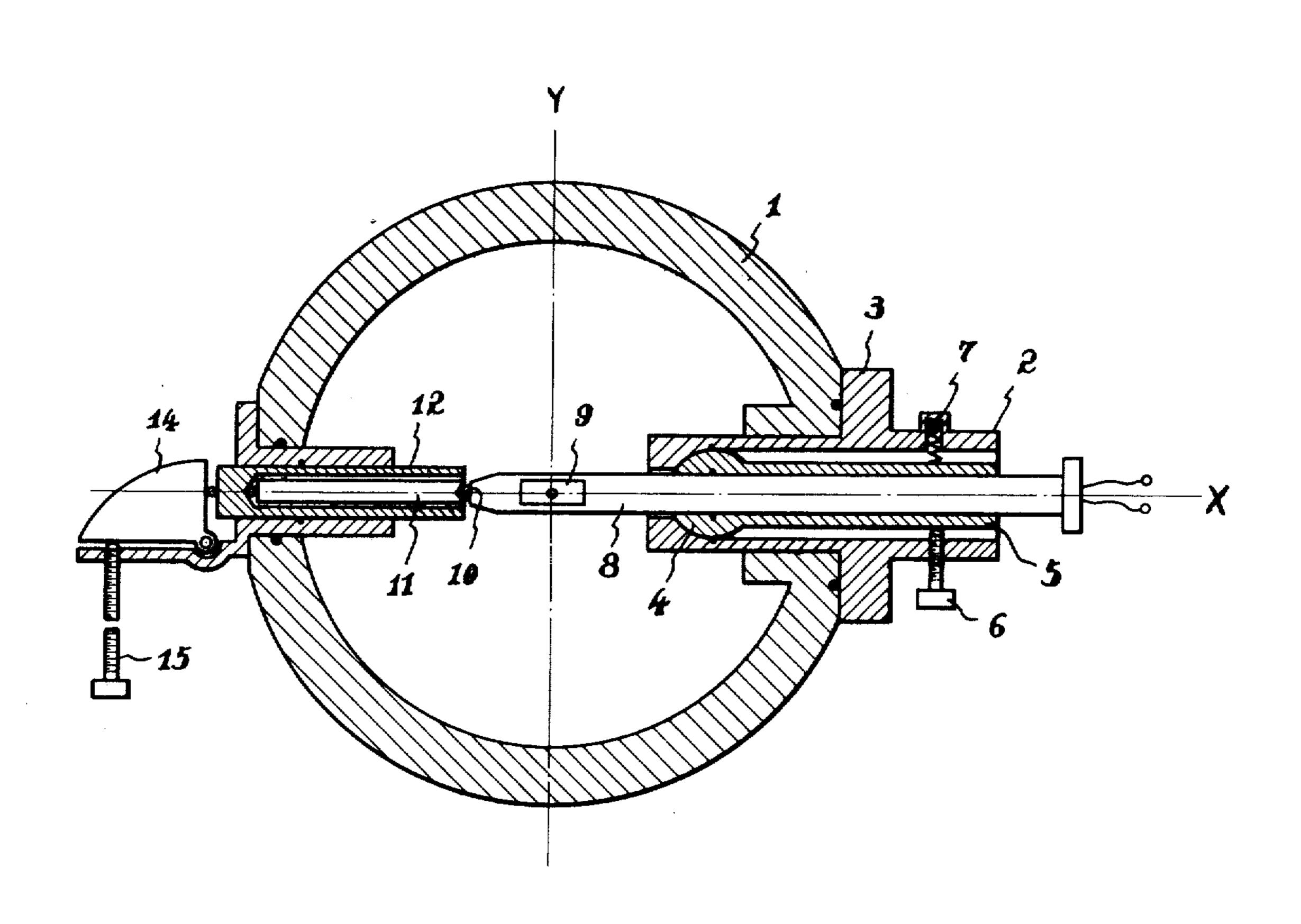
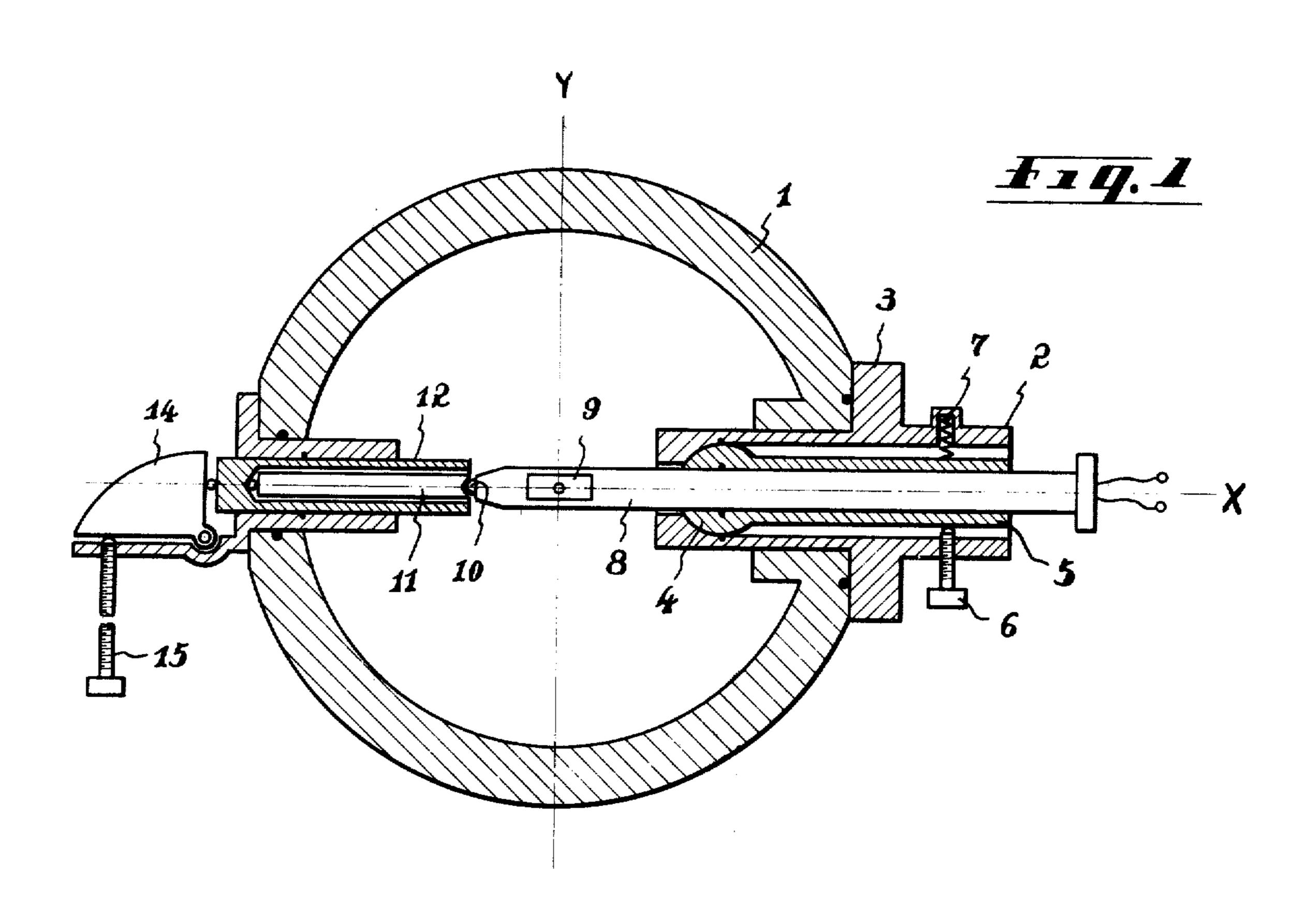
Nukui et al.

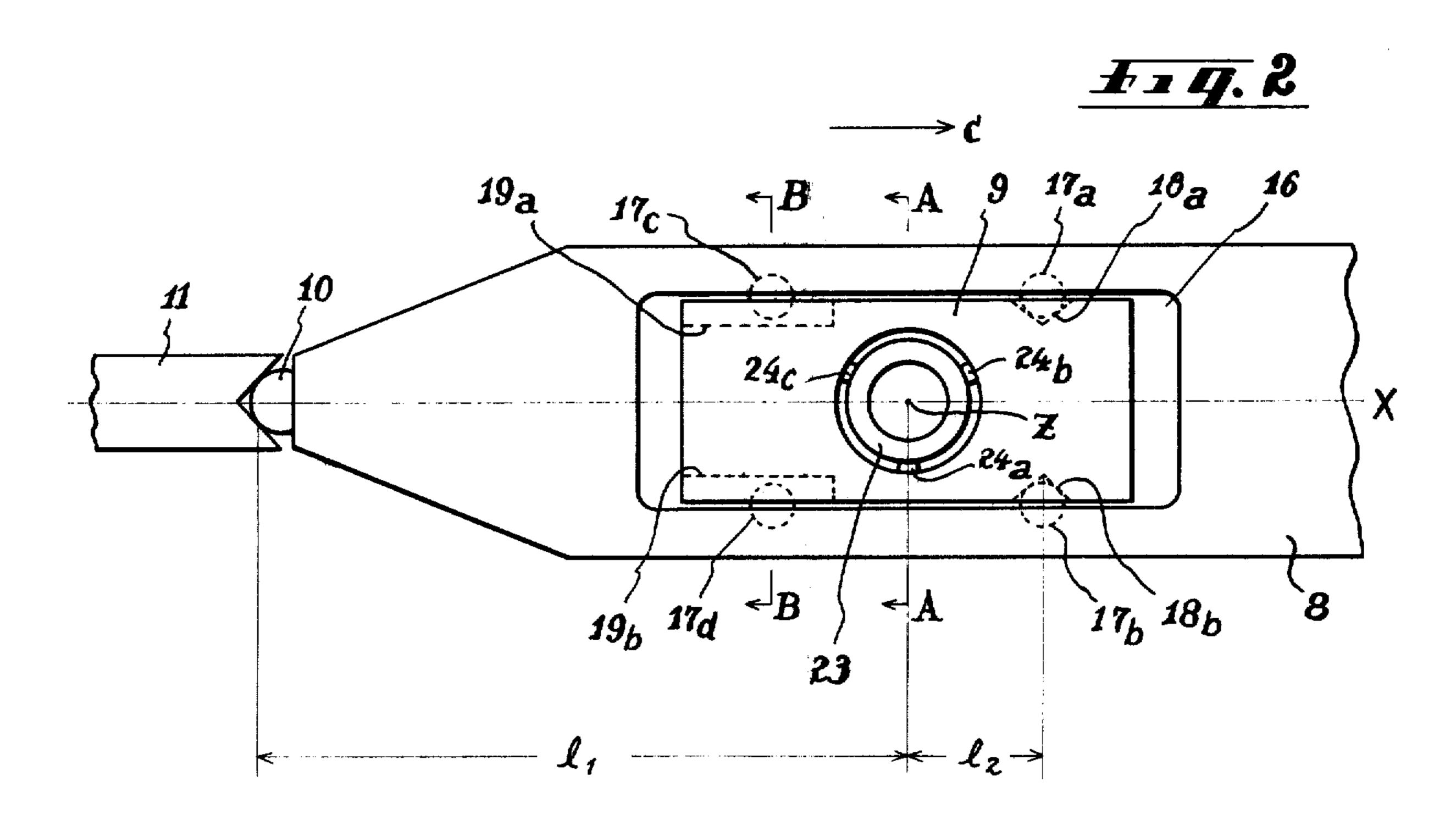
[45] July 22, 1975

[54]	SPECIMEN HEATING AND POSITIONING DEVICE FOR AN ELECTRON MICROSCOPE		3,778,621	12/1973	
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[22]	Filed:	Dec. 5, 1973			
[21]	Appl. No.:	421,978	[57]		ABSTRACT
[30]	Foreign Application Priority Data  Dec. 14, 1972 Japan		A specimen heating and positioning device having a specimen stage with a specimen holder mounted thereon. The specimen stage positions the specimen at		
[52] U.S. Cl			the optical axis. The specimen holder, including a specimen and heating coil, is disposed so that the		
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[51] [58]			shifting of	he specin	nen holder due to thermal expan- is offset by a corresponding shift-
• •		arch 250/439, 440, 441, 442,	shifting of t sion or con	he specin	nen holder due to thermal expan- is offset by a corresponding shift-
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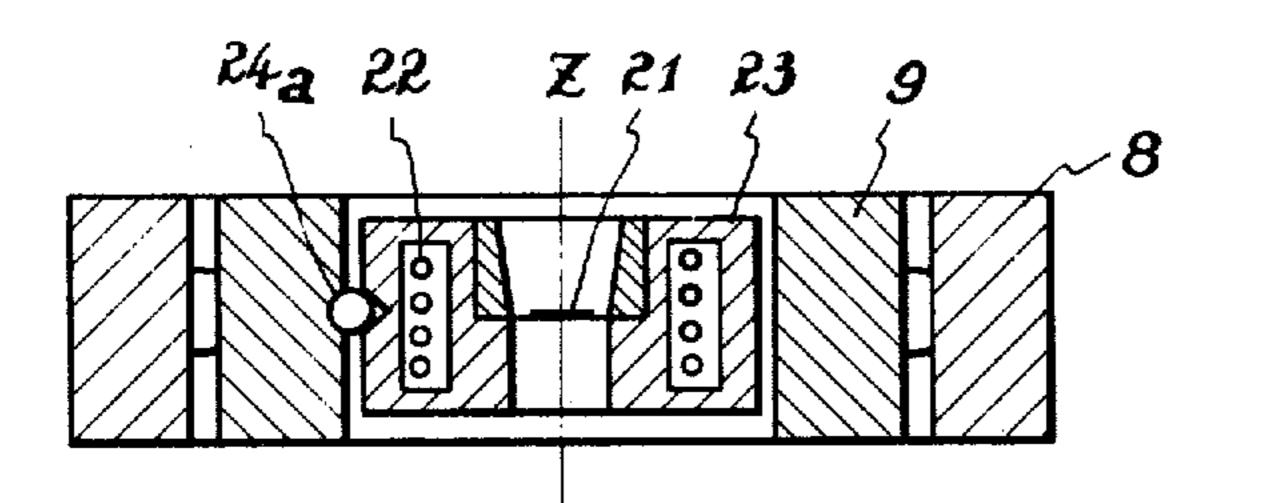
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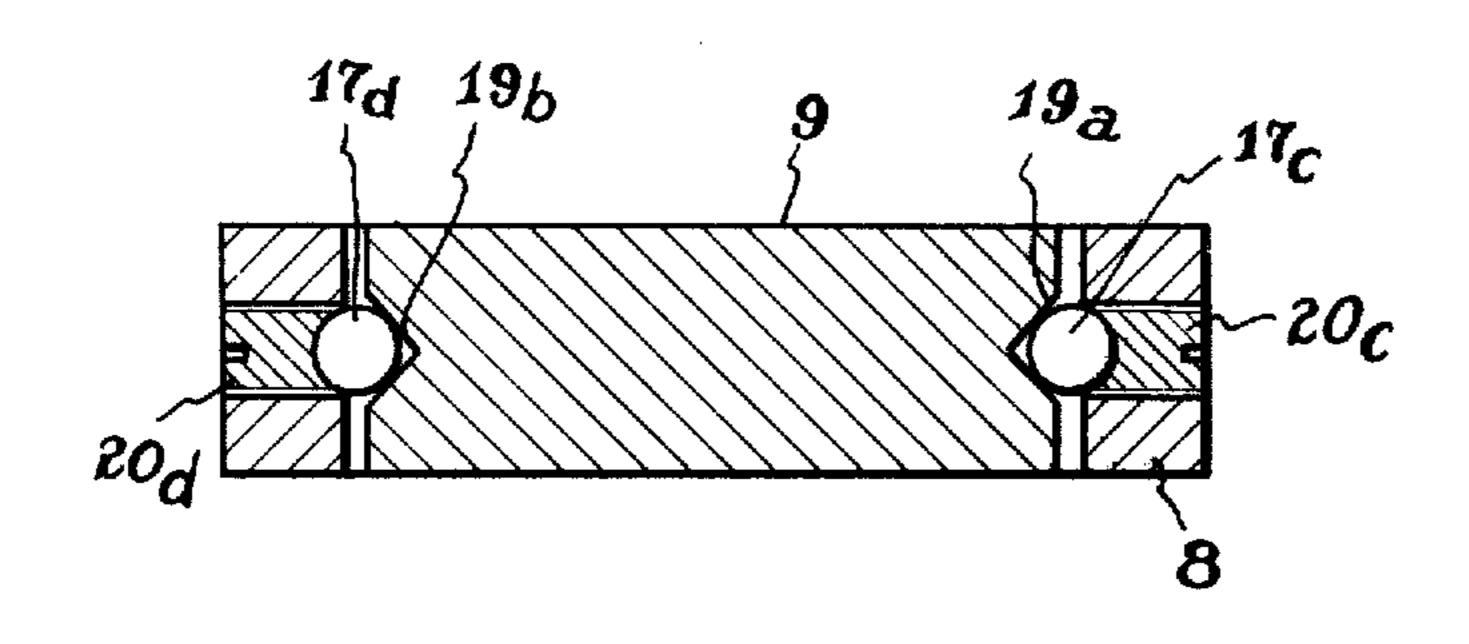


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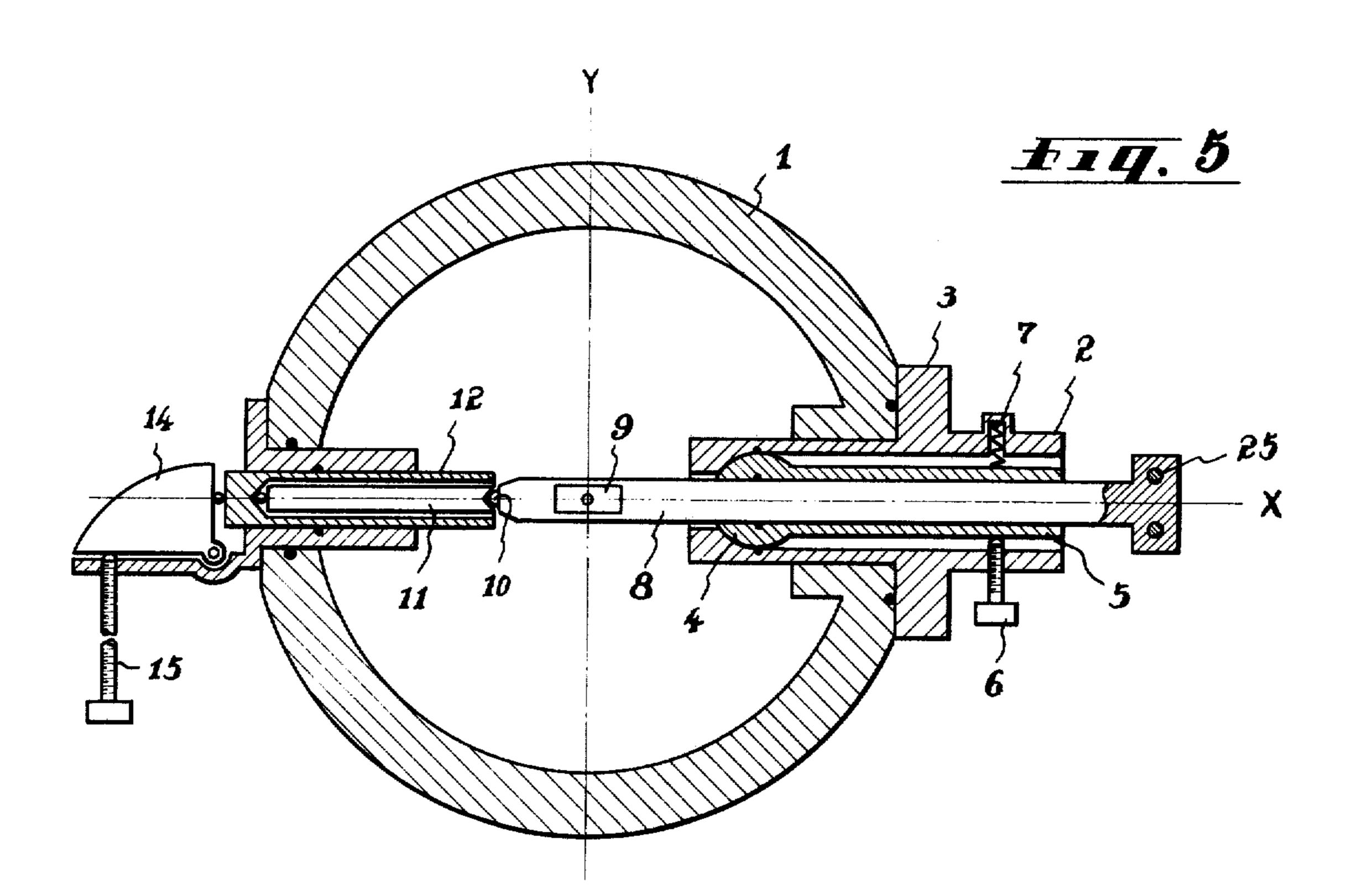
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£14.3



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## SPECIMEN HEATING AND POSITIONING DEVICE FOR AN ELECTRON MICROSCOPE

The present invention relates to a specimen heating and positioning device for electron microscopes or the like.

A specimen must be set in a magnetic field of an objective lens for high resolution electron microscopy. In such case, a device for positioning the specimen can be classified into two types with respect to the way of insertion of the specimen. One is the top entry type in which the specimen is inserted from an upper part of an objective lens, and another is the side entry type in which the specimen is inserted laterally into the objective lens. Both types have respective features, but the side entry type is now used more frequently because of easy operation providing larger angles of tilt than the top entry type. From another viewpoint, since one end of a side entry specimen stage is held to a microscope column, the distance between the column and the specimen must be extremely long.

For this reason, the specimen largely drifts perpendicularly away from the optical axis as the thermal expansion or contraction due to the change in temperature at the specimen stage occurs; it follows, therefore, that in observation of the specimen being heated, its resolving power remarkably lowers due to the drift.

An advantage of this invention is therefore to provide a specimen heating device for electron microscopes capable of observing the heated specimen with high resolving power.

Another object of this invention is to provide a specimen heating device for electron microscopes or the like, which is capable of compensating the drift of a specimen due to the thermal expansion or contraction of the specimen stage.

Briefly, according to this invention, a specimen heating and positioning device for an electron microscope or the like comprises a specimen stage adjustably fixed to the microscope column for positioning the specimen along the optical axis. The stage is arranged to expand 40 11. on heating along an axis perpendicular to the optical axis and away from a first reference point fixed relative to the optical axis. A specimen holder is mounted in the specimen stage. The holder is arranged to expand on heating along the same axis along which the specimen holder expands away from a second reference point fixed relative to the specimen stage and on the opposite side of the optical axis from the first reference point. Means are provided for heating the specimen. The distances between the reference points and the optical axis can be selected such that as the specimen holder and specimen stage are heated, the thermal expansion of the specimen stage and the specimen holder are approximately equal, thus maintaining the specimen substantially at the optical axis.

Other objects and advantages of the present invention will be better understood from the following detailed description referring to the accompanying drawings in which:

FIG. 1 is a cross-sectional view of one embodiment of this invention;

FIG. 2 is a plane view showing a substantial part in FIG. 1;

FIG. 3 is a cross-sectional view taken along line 65 A—A in FIG. 2;

FIG. 4 is a cross-sectional view taken along line B—B in FIG. 2; and,

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FIG. 5 is a cross-sectional view showing another embodiment of this invention.

Referring to FIG. 1, a section of a microscope column 1, for example, the objective lens yoke has mounted therein a rotating body 2 rotatable about an axis intersecting the electron optical axis of the microscope perpendicular thereto (hereafter the "X axis" as shown on the drawing). A knob 3 attached to the external end of the rotating body 2 serves to rotate the body 2 about its X axis. A supporting member 5 terminating on a sphere 4 is supported in a spherical socket of the rotating body 2 so that it is rotatable about a center in the sphere 4. A screw 6 threaded in the rotating body, and the tip of the screw directly touches a side of the supporting member 5. The rotating body 2 is biased against the screw by a spring 7.

A specimen stage 8 passes through the said supporting member 5 and is positioned across the optical axis. A specimen holder 9 is mounted in the specimen stage to be positioned at or near the optical axis. An end of the stage abuts a connecting rod 11 through a ball or bearing 10 made of thermally insulating material as ruby. The connecting rod is set inside a cylinder 12 disposed in the column 1 and is capable of shifting through the cylinder 12 in a longitudinal direction, and may also be slightly tilted within the cylinder 12. The end of this cylinder outside the column touches a bell crank 14, positionable by a screw 15. Thus, by turning 30 the knob 3 of the rotating body 2, the supporting member 5, and the stage 8 turn about the axis X and the specimen is tilted in a certain direction and at a certain angle with respect to the axis X. Further, by adjusting the screw 6, the supporting member 5 rotates centering 35 the sphere 4. Accordingly, the specimen is moved along the axis Y perpendicular to the X axis. Again, adjusting the screw 15, the stage 8 or the specimen shifts in a direction of the axis X through the movement of bell crank 14, the cylinder 12 and the connecting rod

The present invention is characterized by a mechanism which holds the specimen holder 9 to the specimen stage 8. In FIGS. 2, 3, and 4 an opening 16 bigger than the specimen holder 9 is formed in the specimen 45 stage 8 and the said holder is accommodated in this hole, supported by balls 17a, 17b, 17c and 17d made of thermal insulating material such as ruby. A pair of cone-shaped sockets 18a and 18b and a pair of Vshaped grooves 19a and 19b are cut into opposite sides of the specimen holder 8. In this case, the cone-shaped sockets 18a and 18b are, in FIG. 2, formed on the right side of the optical axis Z, perpendicular to axes X and Y, namely, on the side opposite the side where the end of the stage 8 touches the connecting rod 11 thus placing the optical axis between said sockets 18a and 18b and said end. The balls or bearings 17a and 17b are accommodated in the cone-shaped sockets 18a and 18b, respectively, while the balls 17c and 17d are positioned in the V-shaped grooves 19a and 19b, respectively, and all bearings are kept in position by screws 20a, 20b, 20c and 20d respectively, (20a and 20b not shown). As a result, the contact points of the balls 17a and 17b are fixed on the stage 8; while the balls 17c and 17d can be freely shifted permitting the specimen holder to expand away from sockets 18a and 18b.

In the center of the specimen holder is a specimen cartridge 23 which includes the specimen 21 and a

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heating coil 22 held by grooves and ruby balls 24a, 24b and 24c placed therein.

In this embodient, provided an electric current is supplied to the heating coil 22 to heat the specimen 21 up to high temperature, for example 1,000° C, the specimen holder 9 and the specimen stage 8 are heated (in spite of being supported by the balls 17a, 17b, 17c, 17d, 24a, 24b and 24c made of thermal insulating material) by slight thermal conduction and radiation. Thermal expansion, of course, results. The heating device according to this invention is designed so that the expanding directions of the stage 8 and the holder 9 are opposed and the specimen has little, if any drift.

Since the stage 8 thermally expands away from the contact point of its tip with the connecting rod 11, the 15 center of the specimen 21 shifts to the direction shown by an arrow marked C in FIG. 2. The degree of this shift for this case corresponds to the thermal expansion of the length  $l_1+l_2$ , where  $l_1$  is a distance between the center of the specimen and the contact point above described,  $l_2$  is the distance between the center of the specimen and the ruby balls 17a or 17b, measuring these distances along the shifting axis.

On the other hand, the specimen holder 9 expands to reverse direction of the arrow marked C, away from the 25 ruby balls 17a and 17b, thus the center of the specimen shifts in the same direction in an amount corresponding to the thermal expansion for the length  $l_2$ . If the shifting distances due to the thermal expansion of both members are equal by virtue of pertinent selection of their 30 dimensions and materials, the drift of the specimen can be substantially prevented. Since the temperature of the specimen holder will be much higher than that of the specimen stage, even if these members are made of identical material, the center of the specimen can be 35 held at a stationary position.

We have found that where the specimen stage 8 and the specimen holder 9 were both made of phosphor bronze and the specimen was heated to  $1,000^{\circ}$  C, the temperature of the specimen stage and the specimen 40 holder were increased 98° C and 495° C with respect to the temperatures before heating. No drift in the center of the specimen occurred provided the aforementioned length  $l_1$  was 19mm and  $l_2$  was 4.7 mm.

FIG. 5 shows another embodiment of this invention. 45 In this embodiment a heater 25 is also accommodated into the specimen stage. This heater is used in order to heat the stage 8 up to a certain temperature prior to the observation of a heated specimen by electron microscope. By virtue of this preheating the stage, the period of time required for reaching the thermal equilibrium stage during heating the specimen can be shortened, and hence also even a slight drift during equilibrium can be excluded.

In this embodiment, the position of the heater 25 is 55

not limited to the end of the stage 8 as shown in the drawing, but may be adjacent to the specimen holder 9. In addition, it is preferable to arrange any thermal insulator between the specimen stage 8 and the supporting member 5 to prevent heat conduction to the column 1.

In the practical application of this invention, diverse modifications may be realized. For instance, though a fixed point of the specimen stage 8 was previously selected at its tip, it may be on the right of the axis Y in FIG. 1. Further, the specimen holder and the specimen cartridge which were independently formed as in the earlier embodiment, may be unitary or integral.

Having thus described the invention with the detail and the particularity as required by the Patent Laws, what is desired protected by Letters Patent is set forth in the following claims.

We claim:

- 1. A specimen heating and positioning device for an electron microscope or the like having an electron optical axis comprising a specimen stage adjustably fixed for positioning a specimen along the optical axis, said stage arranged to expand on heating on an axis passing through the optical axis away from a first reference point fixed relative to the optical axis and spaced therefrom, a specimen holder mounted in said specimen stage for holding the specimen and arranged to expand on heating along the same axis along which the specimen stage expands in a direction away from a second reference point fixed relative to the specimen stage and on the opposite side of the optical axis from the first reference point, means for heating the specimen, the distances between the reference points and the optical axis being selected such that as the specimen holder and specimen are heated, incidental to the heating of the specimen, the expansion of the stage and holder are approximately equal thus maintaining the specimen substantially at the optical axis.
- 2. A specimen heating device according to claim 1 in which means are provided for heating the specimen stage.
- 3. A specimen heating and positioning device according to claim 1 wherein the said specimen holder is held on the said specimen stage by bearings made of thermal insulating material.
- 4. A specimen heating device according to claim 1 wherein the specimen holder mounted in said specimen stage is secured by four ball bearings, two bearings being seated in cone-shaped sockets formed on opposite sides of the said specimen holder and the adjoining portions of the specimen stage therein defining the second reference point and two being positioned in V-shaped grooves on opposite sides of the specimen holder and adjoining portions of the specimen stage.