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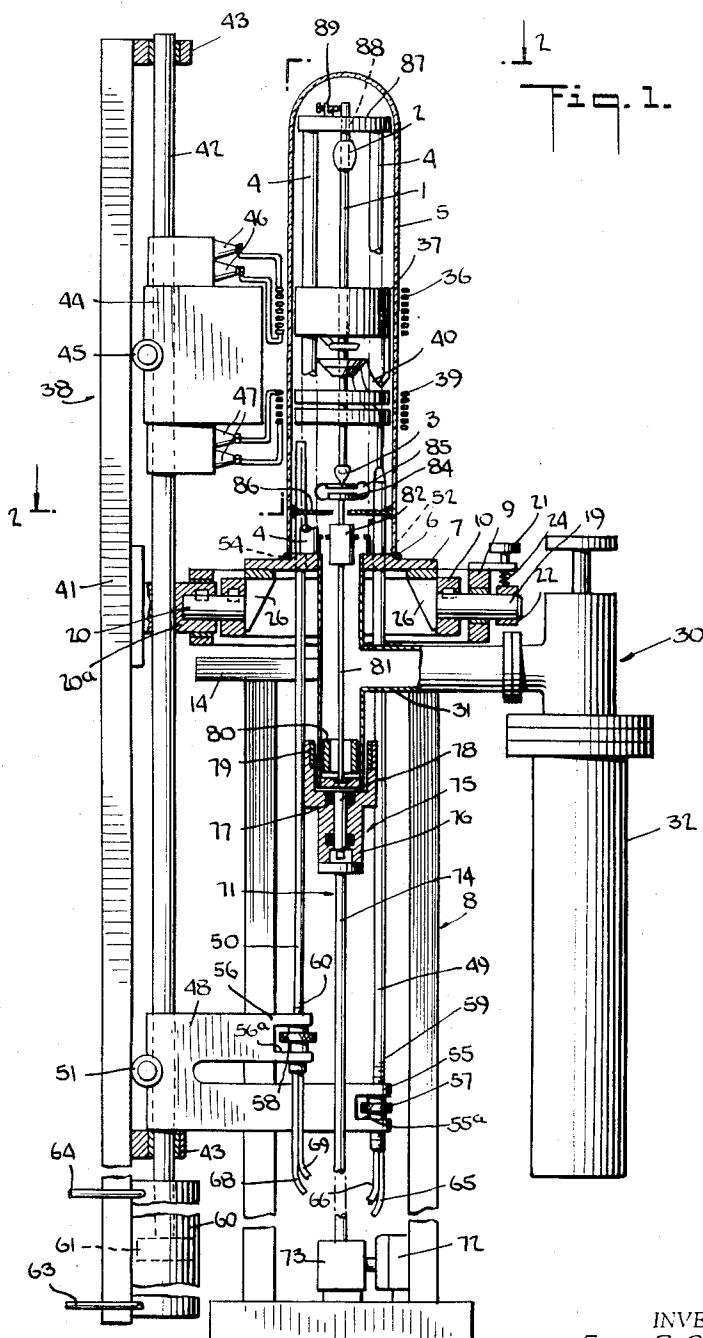
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ZONE PURIFICATION DEVICE

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2 Sheets-Sheet 1



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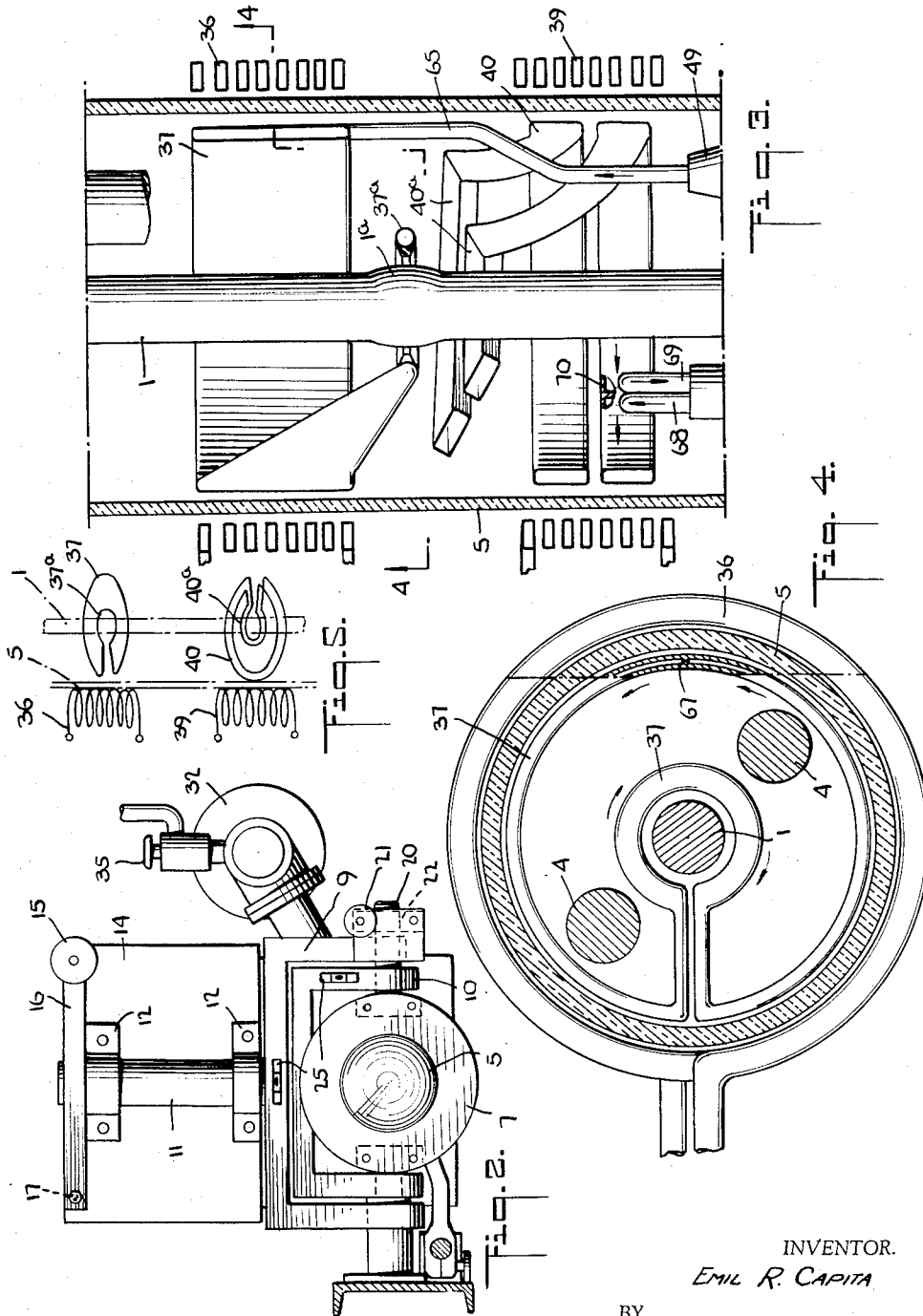
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ZONE PURIFICATION DEVICE

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12 Claims. (Cl. 219—10.43)

The present invention relates to induction heating in a high vacuum or an inert atmosphere and more particularly to a device for inductively melting a short zone of an elongated specimen and for moving the melted zone along the specimen.

The melting of a short zone of an elongated specimen in a vacuum or in an inert atmosphere is used in known processes of zone purification for semiconductor or metallic materials such as germanium, silicon, iron, etc. In the zone purification of such rods a short zone of the rod is melted and the molten zone is then moved longitudinally of the rod. The moving molten zone acts as a distributor of impurities in the rods to a high degree or to increase or decrease the impurity or solute concentration in predetermined portions of the rods as required. In order to provide for a high degree of purity or to permit a predetermined distribution of the impurities, it is necessary to carry out the zone purification in high vacuums or inert atmospheres and to provide for movement of the molten zone along the rods with a minimum amount of vibration or movement of the specimen rods themselves. Zone purifiers now in use do not provide the necessary combination of a neutral atmosphere and a smoothly operating zone motion. The apparatus of the present invention is an improvement upon known types of zone melting devices which provides for an improved control of the zone motion while the specimen rod is mounted in a high vacuum or an inert atmosphere. A novel device is provided which moves the molten zones along the rods while the rods themselves are mounted with freedom from vibration and longitudinal motion.

Accordingly, an object of the present invention is to provide an improved means for zone purification.

Another object of the present invention is to provide a zone purification device which provides for vibration-free mounting of the specimen.

Another object of the present invention is to provide an improved induction type zone purifying device.

Another object of the present invention is to provide an improved induction type zone purifying device having movable induction heating coils.

Other and further objects of the invention will be obvious upon an understanding of the illustrative embodiment about to be described, or will be indicated in the appended claims, and various advantages not referred to herein will occur to one skilled in the art upon employment of the invention in practice.

A preferred embodiment of the invention has been chosen for purposes of illustration and description and is shown in the accompanying drawings, forming a part of the specification, wherein:

Fig. 1 is a side elevational view partially in section of the zone purification device of the present invention;

Fig. 2 is a sectional view taken along the line 2—2 of Fig. 1;

Fig. 3 is an enlarged fragmentary sectional view of the induction heating coils;

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Fig. 4 is a sectional view taken along line 4—4 of Fig. 3; and

Fig. 5 is a schematic diagram of the coils.

The rodlike specimen 1 of germanium or silicon or other material is mounted between holders 2 and 3 on support rods 4 within a transparent airtight cover or jar 5. The jar 5 is mounted by an airtight seal indicated at 6 on a platform 7 which is supported on a stand indicated generally at 8.

In order that the specimen 1 may be positioned vertically during the zone purifying operation, the platform 7 is attached to the stand 8 by adjustable gimbals 9 and 10. As seen in Fig. 2, the gimbal 9 is pivotally mounted by its shaft 11 in bearings 12 on the top 14 of the stand 8 and the position of gimbal 9 is adjusted by threaded adjuster screw 15 which engages the stand top 14 to position the arm 16 on gimbal shaft 11 against the force of compressed spring 17. The gimbal 10 is rotatably mounted in gimbal 9 on shafts 19 and 20 and its position is adjusted by the rotation of threaded adjuster screw 21 which engages the arm 22 fixedly mounted on the outer end of gimbal shaft 19 and which moves the arm against the force of the compressed spring 24. Level gauges 25 (Fig. 2) are mounted on the gimbals to assist in their adjustment so that the platform 7 which is attached to inner gimbal 10 by brackets 26 may be adjusted to a horizontal position.

In order to provide an atmosphere within the jar 5 which is inert with respect to the specimen 1, a vacuum system 30 is connected to the interior of the jar 5 through an outlet tube 31. In the preferred embodiment the vacuum system 30 comprises a diffusion pump 32 with a back-up pump (not shown) connected thereto through valve 35 in the conventional manner. The vacuum apparatus connected to the outlet tube 31 comprises a conventional high-vacuum pumping arrangement and a further description is therefore unnecessary. The vacuum system 30 is the preferred means for providing a pure atmosphere about the specimen 1; however, with certain specimens and where inert gases of extremely high purity are obtainable, the vacuum system 30 may be replaced by a connection to a source of inert gas.

The induction heating means used to melt a short longitudinal zone of the specimen 1 comprise a primary coil 36 positioned outside the jar 5 and a secondary coil 37 energized thereby and positioned within the jar 5 adjacent to the specimen 1. A mounting system 38 is provided for the coils 36 and 37 which moves the coils longitudinally of the specimen 1 in synchronism with each other and which also is adapted to move auxiliary induction coils in synchronism with the heating coils 36 and 37. As illustrated in Figs. 3-5, the secondary heating coil 37 comprises a single turn having an inner portion 37a closely spaced from the specimen 1 to provide for efficient heating of the specimen 1 so that the zone 1a may be melted by the currents induced therein by the heating coil 37 without auxiliary heating elements.

In the embodiment illustrated, additional coils are illustrated in the form of molten zone supporting coils 39 and 40. Coil 39 is a primary coil mounted outside the jar 5 and coil 40 is a secondary coil energized by the primary coil 39 and mounted within jar 5. The secondary coil 40 is mounted below the molten zone 1a and it induces currents therein which are repulsed by the currents in coil 40 so that the repulsion between the two currents tends to support the molten zone 1a against its natural tendency to flow downwardly on specimen 1. Coil 40 is shown with two turns each having a portion 40a bending inwardly adjacent to the specimen 1. As will now be more fully described, the mounting system 38 supports the four coils 36, 37, 39, and 40 in a fixed rela-

relationship with respect to each other and movable with respect to the specimen 1.

The mounting system 38 is fixedly connected to the platform 7 in perpendicular relationship therewith by its support arm 41, which is attached to the outer end 20a of gimbal shaft 20. The support 41 will therefore be adjusted to a perpendicular position when the platform 7 is adjusted to a horizontal position by adjustor screws 15 and 21. Coil support rod 42 is slidably mounted in bearing brackets 43 on the support arm 41. The primary heater coil 36 and the primary auxiliary coil 39 are mounted on bracket 44 which is adjustably positioned on the support rod 42 by clamping screw 45. Coils 36 and 39 are attached to and supported by suitable terminals 46 and 47 which are adapted for connection to suitable oscillators (not shown). The secondary coils 37 and 40 are also mounted on the support rod 42 through the intermediation of bracket 48 and support rods 49 and 50, respectively. The bracket 48 is adjustably positioned on the support rod 42 by the clamping screw 51. The support rods 49 and 50 slide in airtight bearings 52 and 54 in the platform 7 so that they are enabled to reciprocate vertically with respect to the specimen 1. A threaded connection is provided between the support rods 49 and 50 and the arms 55 and 56 of the bracket 48 to allow for fine adjustments of the positions of secondary coils 37 and 40 with respect to each other and with respect to the primary coils 36 and 39. The threaded connection comprises nuts 57 and 58 which engage the threaded portions 59 and 60 of support rods 49 and 50 and which are rotatably positioned in slots 55a and 56a of the bracket arms 55 and 56.

As each of the coils 36, 37, 39, and 40 is connected to the support rod 42, the coils will move in synchronism as the rod 42 moves in its bearing brackets 43. In the preferred embodiment, the support rod 42 has a hydraulic cylinder 61 at its lower end which is adapted to move the support rod 42 up and down as is required for the particular purifying operation being used. A piston 62 is connected to the support rod 42 and the admission of hydraulic fluid under pressure to either inlet 63 or 64 moves the support rod 42 and the attached coils up or down with respect to the specimen 1.

The secondary coils 37 and 40 are preferably hollow and are water cooled. The cooling system for coil 37 comprises a supply tube 65 adapted for connection to a suitable water or other coolant source and a drain tube 66. The tubes 65 and 66 pass through the hollow center of the support rod 49. The tubes 65 and 66 have at least their bottom portions formed of rigid metal to support the secondary coil 37 and they are connected in the electrical center of the hollow secondary coil 37 to provide a ground potential at this point. As seen in Fig. 4, a baffle 67 is provided between the connection of tube 65 and tube 66 so that the coolant passes completely around the coil 37 in the direction of the arrows as it flows from the supply tube 65 to the drain tube 66. The secondary coil 40 is also mounted on and cooled by a water supply tube 68 and a drain tube 69. These tubes are connected at opposite sides of a baffle 70 (Fig. 3) which requires the coolant to flow completely around the secondary in passing from the supply tube 68 to the drain tube 69. Tubes 68 and 69 are connected to the electrical center of the secondary 40 to ground this point.

A drive system adapted to rotate the specimen 1 or its longitudinal axis to equalize the melting effects is indicated generally at 71. The drive system 71 comprises a drive motor 72 with a suitable speed reducer 73 which drives the vertical drive shaft 74. A magnetic coupling member 75 is mounted on the upper end of the drive shaft 74 which comprises a housing 76 rotatably mounted on bearings 77 which engage the fixed shaft 78 on the bottom of the outlet tube 31. Permanent magnets 79 on the housing 76 are magnetically coupled to permanent magnets 80 on the gear shaft 81 which is ro-

tatably mounted within the outlet tube 31 and which is coupled to the lower specimen holder 3 by step-down gearing indicated at 82. In order to avoid the transmission of any vibration from the gear train 82 to the specimen 1, the coupling between the specimen 1 and the last gear train 82 is a flexible coupling 84 with spring members 85.

The upper specimen support 2 is rotatably mounted on the support bracket 87 by a bearing indicated at 88. A locking screw 89 is provided to prevent the rotation of the upper end of specimen 1 when it is desired to rotate only the lower portion to stir the molten zone 1a. A splash pan 86 is mounted beneath the specimen 1 to protect the gear train from metal splashes.

The operation of the above-described zone purification device will now be described.

The platform 7 is first adjusted to a horizontal position by the operation of the adjusting screws 15 and 21 on the gimbals 9 and 10. Specimen 1 is then mounted on the holders 2 and 3 and the airtight cover 5 is placed in position on platform 7 over the support rods 4. The atmosphere within the airtight cover 5 is now purified by the creation of a vacuum therein by the operation of the diffusion pump 32 and the backing pump. These pumps draw the air out of the airtight cover 5 through the outlet tube 31 to form a substantially complete vacuum within the airtight cover 5 and surrounding the specimen 1. The hydraulic cylinder 6 is now used to move the coil support rod 42 and the attached coils 36, 37, 39, and 40 to their desired starting position with relation to the specimen 1. This position will depend upon the particular purification process being used with the particular specimen 1 mounted in the zone purifying device. When the coils have been moved to the starting position, the oscillators which are connected to the primary coils 36 and 39 through terminals 46 and 47, respectively, are energized. The specimen heating currents are now induced in the secondary 37 and in the specimen 1. A short longitudinal zone 1a (Fig. 3) of the specimen 1 is now melted by the current induced in the specimen 1 from the secondary 37 of the heating coils. A repulsive supporting force is provided for the molten zone 1a by the currents induced therein by the auxiliary secondary coil 40 which is positioned beneath the melting coil 37 and the molten zone 1a.

Once the molten zone 1a has been formed on the specimen 1, the zone purification process requires the molten zone 1a to be moved longitudinally along the specimen 1. This is done by admitting hydraulic fluid through the hydraulic cylinder inlets 63 or 64 to move the coil support rod 42 upwardly or downwardly, respectively, as is necessary for the particular purification operation being performed and at a speed which is controlled by controlling the rate of flow of the hydraulic fluid into the hydraulic cylinder 60. Since the primary coils 36 and 39 are rigidly attached to the support rod 42 on bracket 44 and since the secondary coils 37 and 40 are also rigidly connected to the support rod 42 by means of the bracket 48, the coils will maintain a constant relationship with respect to each other as they are moved along the specimen 1. During the motion of the coils, the airtight cover 5, which is preferably a glass jar, acts as an insulator between the primary and the secondary coils. In order to equalize the heating effect of coil 37 around the circumference of the specimen 1, specimen 1 is rotated by drive motor 72. Preferably the drive motor 72 is a variable speed motor so that the rate of rotation of the specimen 1 may be adjusted.

During the purifying operation, the overheating of the secondary coils 37 and 40 is prevented by the admission of coolant to the coils through the inlet tubes 65 and 68, respectively.

When the zone purification has been completed by the movement of the molten zone 1a along the specimen 1 to the extent required, drive motor 72 and coils 36 and

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39 are de-energized along with the diffusion pump 32 and the backing pump. The air pressure is then raised within the cover 5 to correspond to the outside pressure and the cover 5 is removed to permit the removal of the specimen 1.

It will be seen that the present invention provides an improved zone purification device which provides for the zone purification of a rodlike specimen in a pure atmosphere with a minimum amount of vibration and with no longitudinal motion of the specimen. The zone purifying device permits the use of heating coils as well as auxiliary coils and provides for the synchronized movement of all the coils so that they all may be operated with corresponding motions. The heating coil is adapted to initiate the zone heating without auxiliary heating means so that the starting of the zone purifying operation is simplified. Cooling means are provided for the secondary coils adjacent to the specimen to permit their efficient operation with high induced currents without overheating. The zone purifying device of the present invention is also adapted for automatic operation so that the melted zone on the specimen may be moved longitudinally of the specimen in the desired pattern and so that the specimen may be simultaneously rotated on its longitudinal axis with a minimum of supervision.

As various changes may be made in the form, construction and arrangement of the parts herein without departing from the spirit and scope of the invention and without sacrificing any of its advantages, it is to be understood that all matter herein is to be interpreted as illustrative and not in a limiting sense.

Having thus described my invention, I claim:

1. A zone purification device for refining an elongated solid metallic body comprising the combination of an airtight enclosure adapted to contain an elongated specimen of the solid metallic body, a transformer including a primary coil outside said airtight enclosure and a secondary heating coil within said airtight enclosure adapted to encircle closely a portion of the specimen, and said primary coil and said secondary coil connected to a common movable support means whereby the secondary coil may be moved along the specimen while maintaining a fixed position relative to said primary coil.

2. A zone purification device comprising the combination of an airtight enclosure adapted to contain an elongated specimen, a transformer including a primary coil outside said airtight enclosure and a secondary heating coil within said airtight enclosure adapted to encircle a portion of the specimen, said primary coil and said secondary coil connected to a common movable support means whereby the secondary coil may be moved along the specimen while maintaining a fixed position relative to said primary, a second transformer having a primary coil outside said airtight enclosure and a secondary coil within said airtight enclosure adapted to encircle the specimen at a position spaced from said first-mentioned primary coil, and said primary coil and said secondary coil of said second transformer being mounted on said movable support whereby they move in a fixed relationship to each other and to the primary coil and the secondary coil of said first transformer.

3. A zone purification device comprising the combination of an airtight enclosure adapted to contain an elongated specimen, a transformer including a primary

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coil outside said airtight enclosure and a secondary heating coil within said airtight enclosure adapted to encircle a portion of the specimen, said primary coil and said secondary coil connected to a common movable support means whereby the secondary coil may be moved along the specimen while maintaining a fixed position relative to said primary and said secondary coil being hollow and being adapted for connection to a source of coolant.

4. The zone purification device as claimed in claim 1 in which said airtight enclosure is coupled to an air evacuating means.

5. The zone purification device as claimed in claim 2 in which said secondary coils are each hollow and are each adapted for connection to a source of coolant.

6. A zone purification device comprising the combination of an airtight enclosure, an outlet on said enclosure, spaced holders mounted within said enclosure and adapted to support an elongated specimen therebetween, one of said holders being rotatably mounted, a drive means operatively coupled to said one holder, and heating means for said specimen comprising a primary coil outside said enclosure and a secondary coil within said enclosure and adapted to encircle a portion of a specimen mounted in said holders.

7. The device as claimed in claim 6 in which said drive means comprises a drive motor positioned outside said enclosure which is coupled to said one holder through a wall of said enclosure by an airtight coupling.

8. The device as claimed in claim 7 in which said airtight coupling comprises a magnetic coupling.

9. A zone purification device comprising the combination of an airtight enclosure, a pair of spaced specimen holders in said enclosure adapted to mount an elongated specimen therebetween, an outlet on said enclosure, an adjustable support for said enclosure movably mounted whereby said specimen may be positioned vertically, a transformer including a primary coil outside said enclosure and a secondary heating coil within said enclosure adapted to encircle the specimen, a common support means for said coils attached to said enclosure support and movable therewith, and said coils connected to said support means through the intermediation of a reciprocable member mounted for motion parallel to the specimen whereby the secondary coil is movable along the specimen with the primary coil maintained in fixed relationship to the secondary coil.

10. The device as claimed in claim 9 in which said enclosure support comprises a pair of gimbals rotatably mounted on perpendicular axes.

11. The device as claimed in claim 9 which further comprises a hydraulic drive means operatively connected to said reciprocable member and adapted to move said reciprocable member in opposite directions.

12. The device as claimed in claim 9 in which said specimen holders are rotatably mounted, and one of said holders is operatively connected to a drive means.

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