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An Ultrahigh Vacuum Reflectometer for Use with Extreme Ultraviolet Synchrotron Radiation

by

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An Ultrahigh Vacuum Reflectometer for Use with Extreme Ultraviolet Synchrotron Radiation[≠]

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Abstract

An ultrahigh vacuum reflectometer is described. It may be used for studies of reflectance, transmittance, and photoemission on samples evaporated *in situ*. The angle of incidence can be varied from 0° to $\pm 90^\circ$ in steps of 7.5° for sample and detector independently. The whole system can be rotated about the axis of the incident light for study of polarization effects. With the highly polarized continuum of synchrotron radiation as a light source and a normal incidence monochromator the reflectometer allows measurements in the extreme ultraviolet for wavelengths down to 300 Å for s and p polarized light.

1. Introduction

The use of synchrotron radiation as a light source allows measurements with polarized light in the extreme ultraviolet (XUV) region[1] under ultrahigh vacuum (UHV) conditions. This opens a broad field of interesting studies. The advantages of polarized light and UHV in this region have been shown recently in investigations of optically excited plasma oscillations in thin films and the polarization dependent photoeffect at the Deutsches Elektronen-Synchrotron (DESY)[2,3]. Polarized light is advantageous also in measurements of optical constants[4,5]. Through independent measurements of reflectance with the light electric vector parallel (p) and perpendicular (s) to the plane of incidence, it is possible to check the internal consistency of the measured optical constants. The accuracy of the determination of optical constants is in general higher for measurements made with polarized light than for those made with unpolarized light. Only polarized light allows meaningful measurement of the optical constants of anisotropic crystals.[6]

Adsorbed layers of the order of a monolayer thick can strongly influence XUV reflection measurements and the determination of optical constants from them. Therefore it is desirable to carry out reflection measurements on clean samples in UHV. Since the pressure in the DESY synchrotron is about 10^{-6} Torr, which is orders of magnitude lower than the pressure in conventional XUV sources[7], UHV conditions in an experimental chamber can be relatively easily obtained through differential pumping.

2. Degrees of Freedom

Figure 1 illustrates the degrees of freedom which the reflectometer offers. Light highly polarized with its electric vector in the plane defined by axes 1 and 2 is incident along axis 2. The figure shows the case where the electric vector is perpendicular to the plane of incidence of the sample (s polarization). The sample may be turned about axis 1 and this axis is movable so that the sample can be removed from the beam for direct measurement of the incident light. The detector can be turned on an axis coaxial to that of the sample. Sample and detector can be moved independently in steps of 7.5° from 0° to 360°. The complete arrangement may be rotated about axis 2 so that the other polarization (p polarization) or an arbitrary degree of polarization may be elected.

Figure 2 is a photograph of the reflectometer outside of the vacuum system. It is nearly in the position drawn in Fig. 1. Axis 1 goes from the lower right to the upper left. Light is incident through the hole in the bearing on the left. The diameter of the ring is 25 cm. Its size is determined by the sizes of the vacuum chamber (diameter 30 cm) and the detector. The detector, a Bendix multiplier, is at the top of the photograph.

3. Detector

We have chosen as detector an open electron multiplier (Bendix M306) for the following reasons:

1. The sensitivity of the multiplier photocathode falls sharply for wavelengths above about 1200 Å. This eliminates straylight problems due to long wavelength synchrotron radiation.
2. The multiplier may be baked out to 200° C.
3. The M 306 multiplier can be modified for direct photoelectric measurements on metal films[8]. For such measurements the grid normally positioned over the cathode is removed and a microscope cover glass is inserted in the position of the cathode sheet. Metal films may then be evaporated on the cover glass to provide photocathodes evaporated *in situ*.

A disadvantage of the magnetic photomultiplier is its strongly inhomogeneous cathode sensitivity. Since the beam emerging from a monochromator (particularly one used with synchrotron radiation) is not homogeneous, light displaced or inverted on the multiplier cathode will introduce spurious results. With such a detector absolute reflectance measurements are extremely difficult[9]. One can, however, make relative reflectance measurements at various angles. In order to obtain optical constants from reflection measurements made in this way, measurements must be made for at least three different angles. With absolute reflectance values two angles suffice in principle. Normally one attempts to make even absolute measurements at as many angles as possible, say 5 or 10, in order to increase the accuracy of the optical constant determination through internal consistency checks. If many angles are measured there is no disadvantage in working only with relative reflectances.

4. Technical details

A special difficulty exists for a reflectometer which is to operate in UHV. There are no rotary UHV feedthroughs which make possible a rotation requiring a considerable torque, which are free of play, and which yield an exactly reproducible motion. Therefore, we have chosen to set the angles of the sample and detector in a stepwise manner, where the steps are given by a prescribed angular setting. We have given up the advantages of a continuous choice of angles. Exact measurements of the critical angle[9] or the Brewster angle are thus impossible. For measuring methods which use fixed angular settings such a device has the advantage of making accurate angular placements easy to attain. In addition, by giving up continuous angular measurements, we have been able to design a reflectometer which allows the polarization of the incident light to be changed without alteration of the sample and detector angles.

Optimal adjustment of a reflectometer is difficult[9]. It is even more complicated than usual when using synchrotron radiation since health physics regulations prevent us from working at the reflectometer while synchrotron light is falling upon it. Our adjustment is a two step process. First the reflectometer must be internally adjusted, i.e. the axes 1 and 2 must be perpendicular and the sample detector angles must be accurate. Secondly (and more difficult in our case), axis 2 must coincide with the axis of the incident light. These two adjustments can be carried out separately.

Figure 3 shows a cross section through the vacuum system perpendicular to the synchrotron plane, i.e. a side view. The synchrotron light emerging from our monochromator is incident from the left. The angle setting device which is mounted on the turning ring TR is not shown. The ring is shown in s position (dashed: p position). TR is held in two ball bearing races K1 and K2 mounted on the supports ST. The frame TR may be rotated about the axis of the incident light (s-p rotation) by means of a "wobble-stick" rotary feedthrough DF. At position T the ring TR together with the bearings can be separated from the supports. The internal adjustment of the reflectometer can thus be performed outside of the vacuum chamber. This adjustment and the subsequent adjustment of the reflectometer relative to the synchrotron light are greatly facilitated by using a laser. The screws S_1 allow the reflectometer to be adjusted with respect to the incident light.

A top view of the apparatus is shown in Fig. 4. Here the angle stepping device is included. The detector (multiplier MP) is fastened on arm A on the other end of which is mounted a counterweight G. For photoelectric measurements the sample holder PH and the counterweight are removed and MP is slid to the middle of A. M and P are gears coaxial with the multiplier and sample axis which fix the angle of the multiplier and sample respectively. The drive for the angle selection device is provided through the magnetic feedthroughs MD and the keys S. The principle of this drive is illustrated in Fig. 5. With one revolution of the keys S the gear is advanced one tooth. The ratchet R, which is under pressure from the leaf spring F, guarantees the accuracy of the angle steps. In Fig. 5 the key is uncoupled so that the reflectometer may be driven to the other polarization direction. The gears of the multiplier and sample have different diameters but each has 48 teeth which define the angular step of 7.5°.

Figure 6 shows how the sample may be removed or inserted in the light beam. The sample PR sits on a sled S in frame H. The sample may be heated by resistance wires in HE. By sliding S to the left the sample may be removed from the light path which is perpendicular to the plane of the figure and is indicated by M. Sliding the sample sled is carried out as follows. Hook B is fixed to a nut N which is on threaded axis A of a magnetic rotary feedthrough.

B is engaged in a slit in cylinder Z. By turning the feed-through clockwise, Z and (by means of the rods ZS) the sample sled are moved to the left. Upon finishing the sample movement B is rotated 180° counter-clockwise to position B'. With the hook in position B' the reflectometer polarization direction can be changed without hindrance. By rotating B further counter-clockwise the hook may be engaged on the other side of Z and the sample driven to the right and into the light path.

The accuracy of the angle settings is mainly determined by the machining of the two gears, particularly that of the sample stepping device. The angular accuracy of the reflectometer has been checked trigonometrically with a beam of laser light reflected from a sample mirror and by using autocollimation of a laser beam. The step angle of 7.5° is accurate and reproducible to $\lesssim 0.2°$. A gear machined to close tolerances would certainly improve this. Inaccuracies in the angle due to slight misalignment of the various components of the reflectometer or misalignment with the synchrotron radiation beam may bring the total error in an angular setting to as much as 0.5°. Errors of the latter types can be taken into account in data reduction through their systematic appearance in curves of reflectance versus angle for angles above and below the synchrotron plane.

5. Ultrahigh Vacuum

The reflectometer is constructed of nonmagnetic stainless steel. It is used in a modified Varian VT-102 UHV system in conjunction with a normal incidence monochromator designed for use with DESY synchrotron radiation. The monochromator has been described by Skibowski and Steinmann[10]. The VT-102 contains a 270 l/sec ion pump and a titanium sublimation pump. We utilize a turbomolecular pump for forepumping. Ions produced by the operation of the ion pump caused spurious results with our electron multiplier until "optical" baffles were inserted in the vacuum chamber between its ion pump and the reflectometer assembly.

The exit slit of our monochromator (at present 0.5 mm X 3.0 mm with a length of 10 mm) allows a pressure differential to be maintained between the reflectometer vacuum system and the monochromator. With a pressure of 10^{-6} Torr in front of the slit a pressure in the reflectometer chamber of 1×10^{-9} Torr has already been reached.

The reflectometer has been baked out to 150 - 200° C for extended periods and operated in a vacuum of 10^{-9} Torr without serious difficulties due to "freezing" of moving parts. ("Molykote" lubricant has been used on several bearing surfaces.)

6. Measurements

The first measurements with the reflectometer discussed here were studies of the optical excitation of a plasma resonance in Al films at a pressure of 1×10^{-9} torr. A pronounced resonance in reflectance and photoemission with p polarized light was found at the Al plasma wavelength (835 Å) for the first time[2]. A strong influence of Al_2O_3 surface layers on the resonances was observed. Measurements on Al films were carried out earlier with a prototype of the reflectometer described here which was only suitable for measurements down to 10^{-6} torr[2]. The prototype was also used to measure the optical constants of Ge in the wavelength region 300 to 500 Å with both s and p polarized light[5].

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Footnotes

≠ This paper is based in part on a portion of the doctoral thesis of B. Feuerbacher submitted to the Universität München, München, Germany. The experiments were performed at the Deutsches Elektronen-Synchrotron DESY, Hamburg, Germany. The work received support from the Deutsche Forschungsgemeinschaft.

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Figure Captions

1. The degrees of freedom offered by the reflectometer.

   1-- axis for coaxial driving of sample and detector

   2-- axis allowing rotation to change polarization, axis of incident light.

2. The reflectometer outside the vacuum system. Incident light enters through the bored axis on the left.

3. Side view of the reflectometer.

   TR turning ring

   K1, K2 ball bearing races

   T removable frame mounts

   ST supports

   S1, S2 screws for adjustment and fastening

   FK flexible coupling

   FB bellows

   DF "wobble-stick" feedthrough

4. Top view of the reflectometer.

   TR turning ring

   PH sample holder assembly

   MP multiplier

   A, G multiplier arm and counterweight

   M, P gears for advancing MP and PH

   S gear keys for M and P

   MD magnetic feedthroughs

5. Angle stepping arrangement.

   S drive key

   R ratchet

   F spring

6. Sample in - out arrangement.

   PR reflectance sample

   M incident light beam

   B hook connected to magnetic feedthrough

   other symbols explained in text

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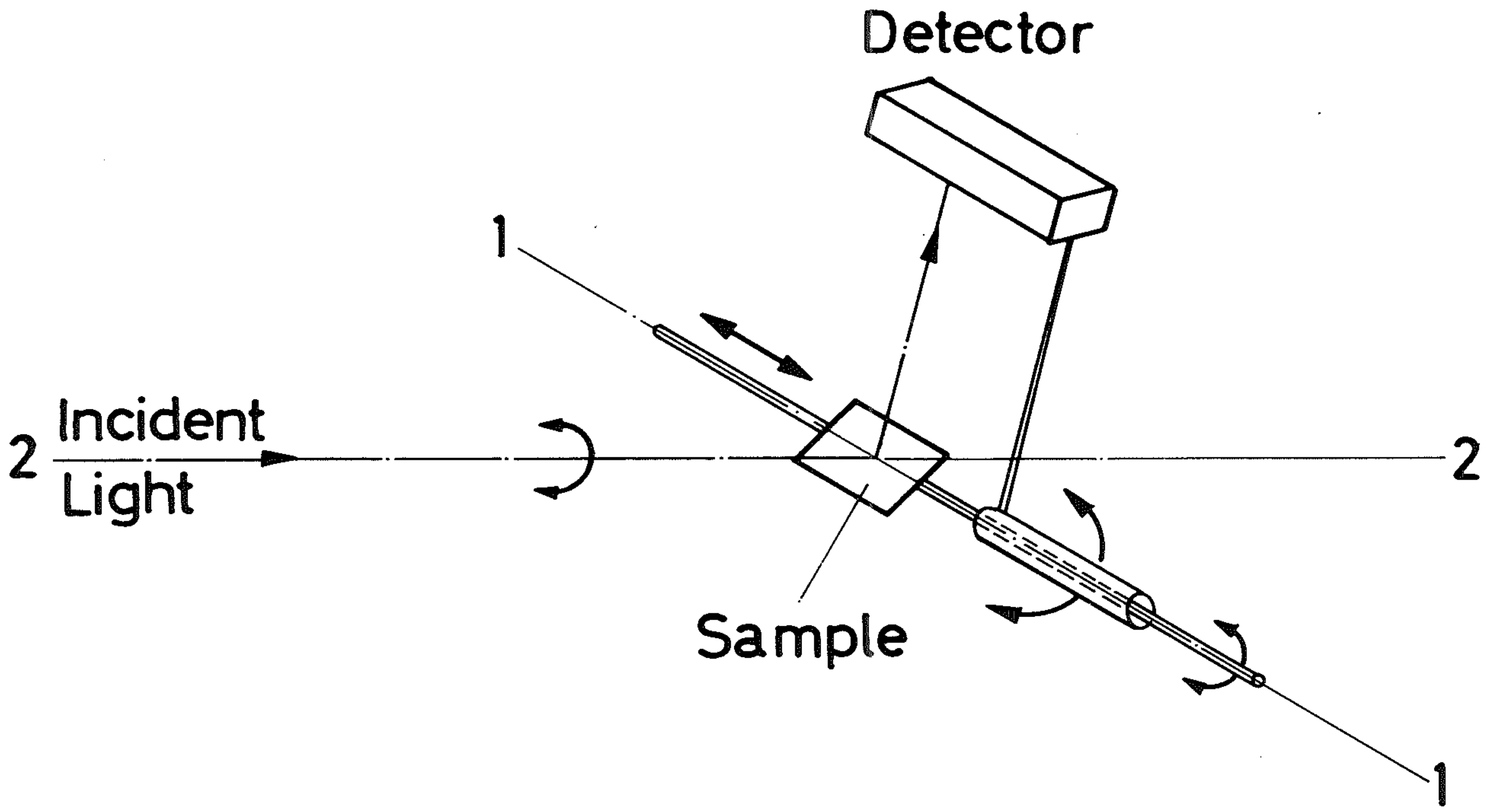
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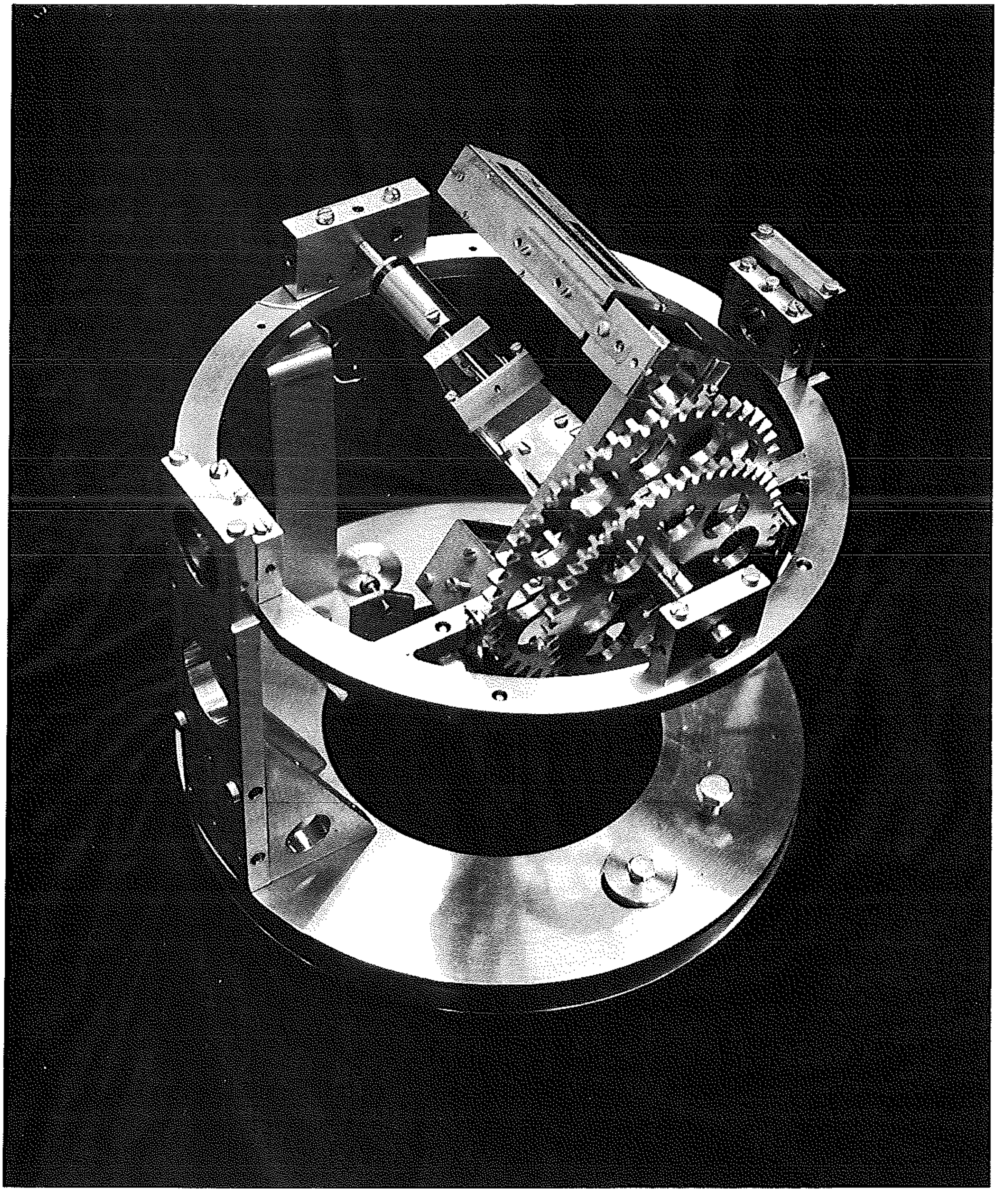

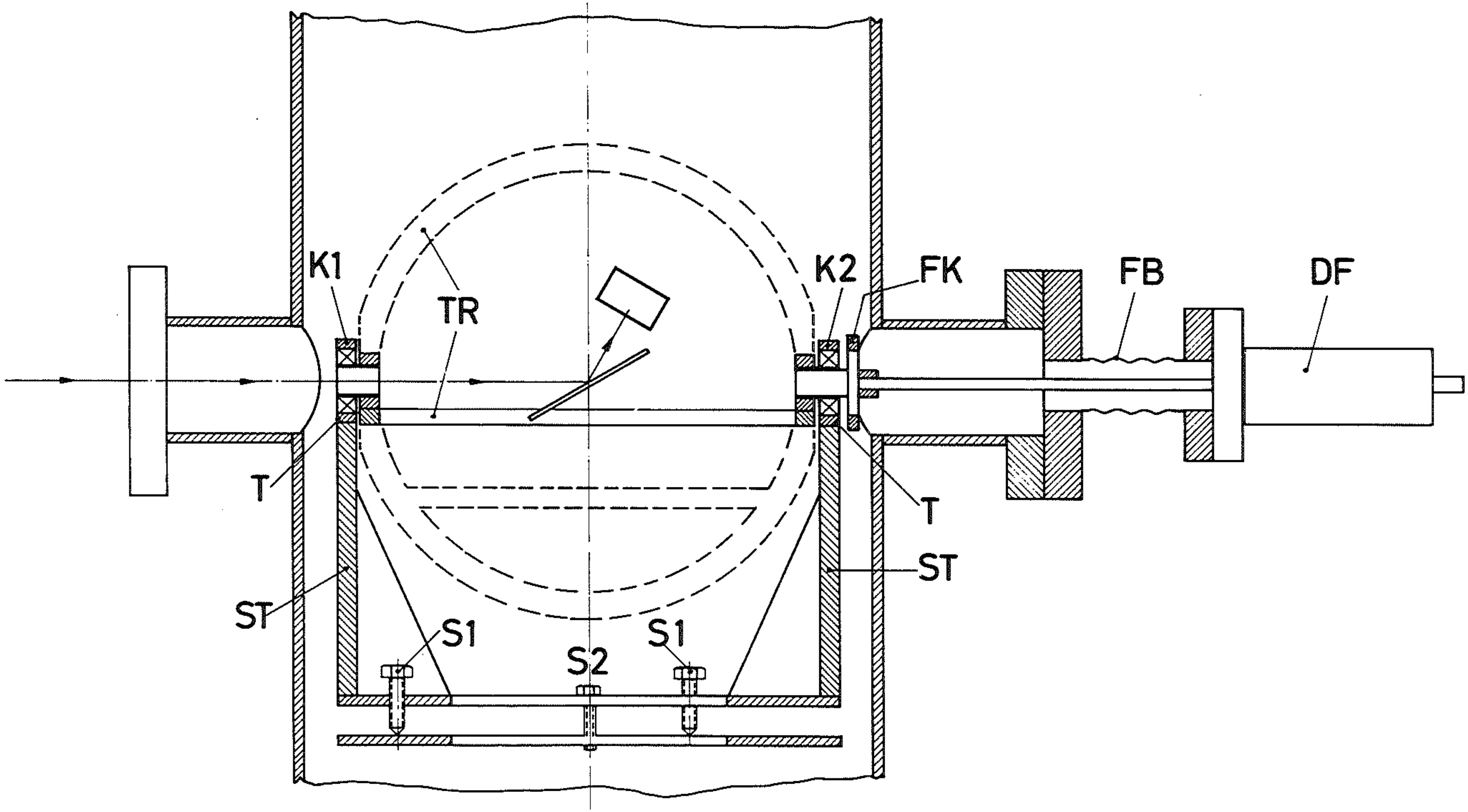
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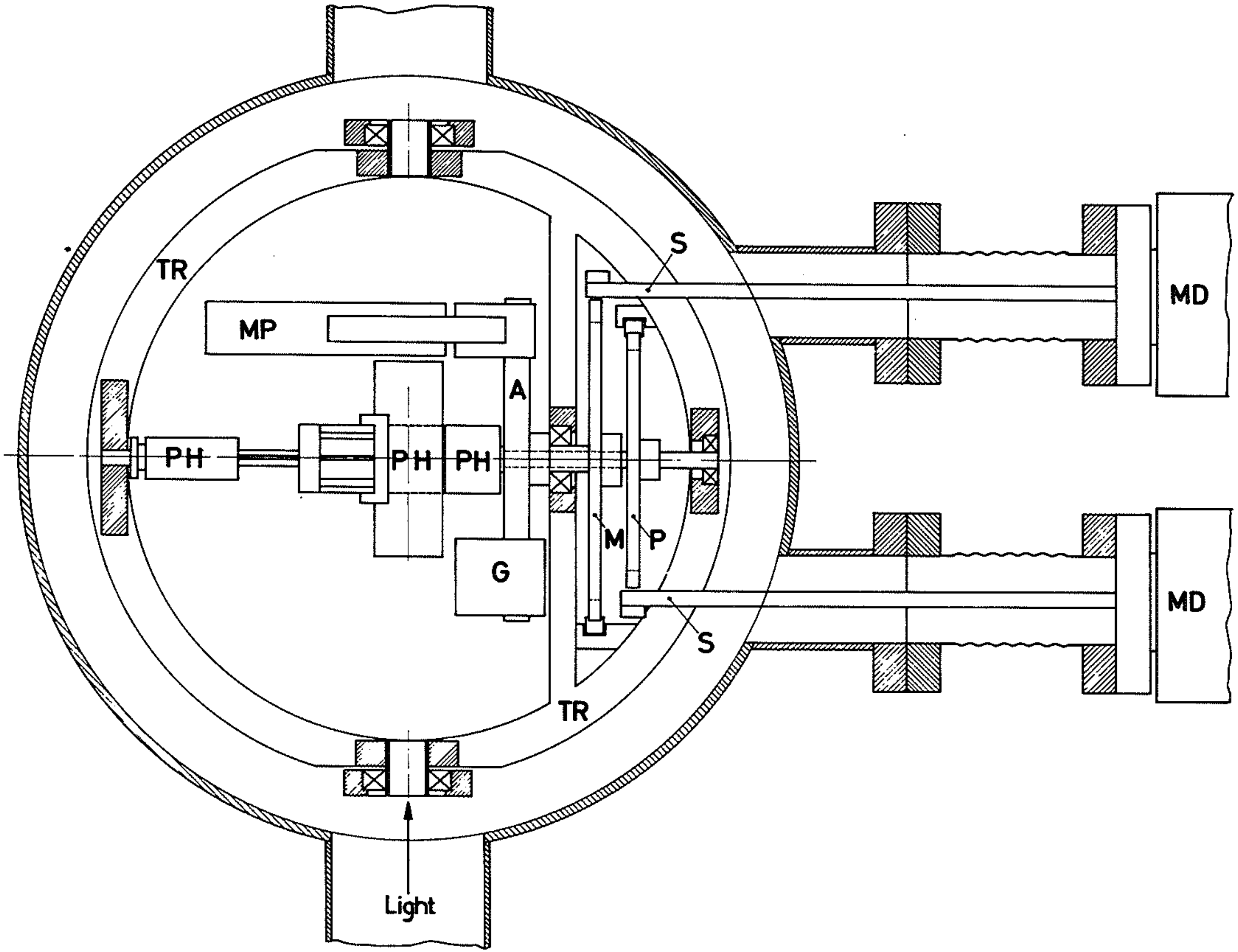

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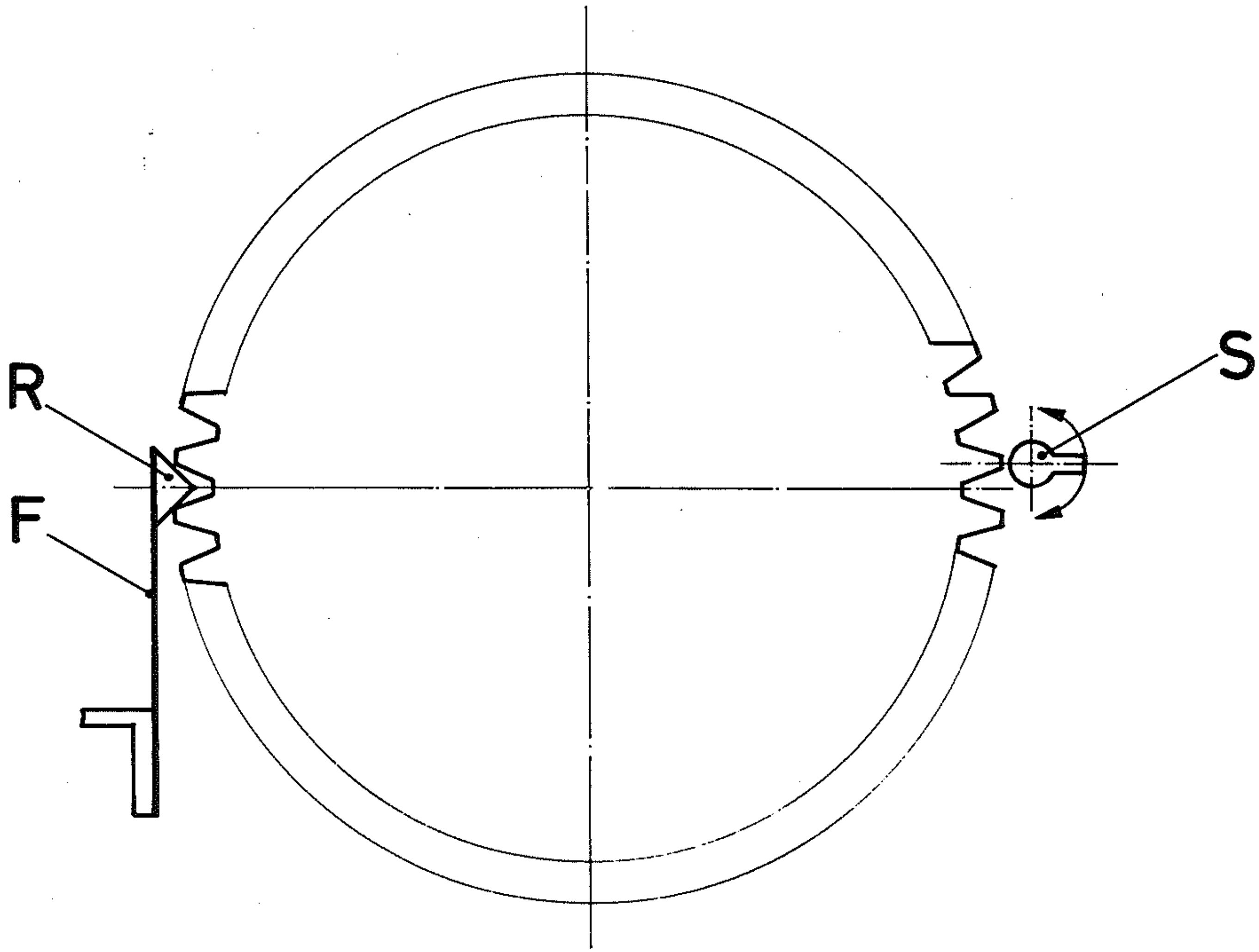
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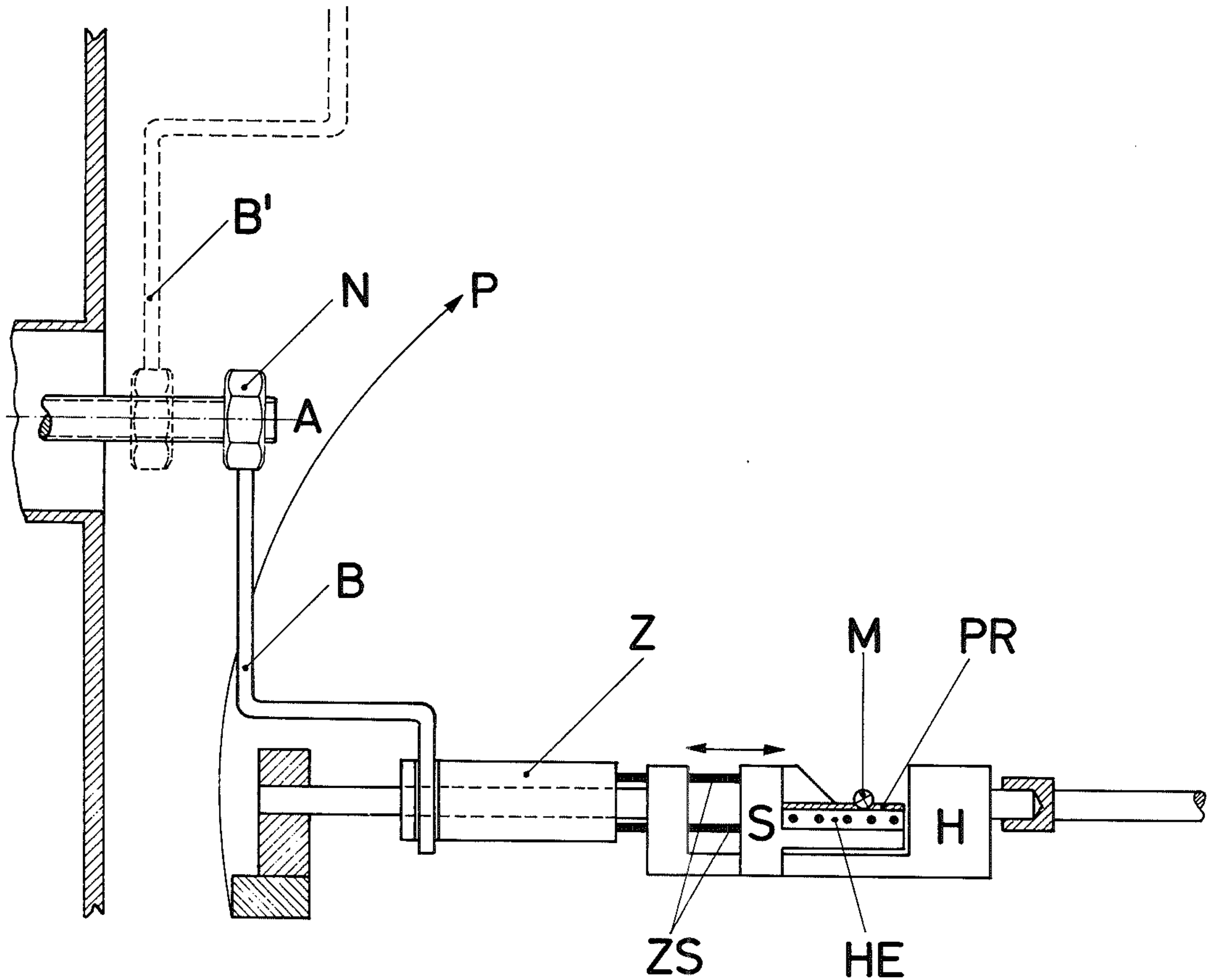
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