# United States Patent [19]

**Schmidt**

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[45] **Feb. 12, 1974**


[54] **SURFACE ANALYSIS**


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[52] **U.S. Cl.** ..... **250/41.3,** 250/41.9 SE

[51] **Int. Cl.** ..... **B01d 59/44**

[58] **Field of Search**..... 250/49.5 P, 41.9 SE, 41.3



[56] **References Cited**

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*Primary Examiner*—James W. Lawrence

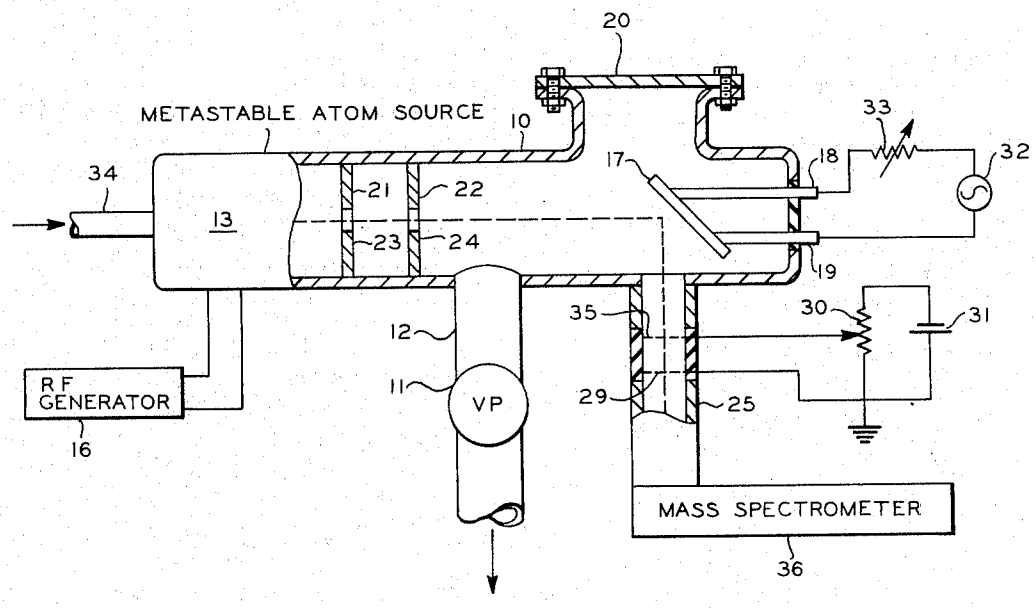
*Assistant Examiner*—C. E. Church



[57] **ABSTRACT**

Surfaces are analyzed to determine materials adsorbed thereon by directing a beam of atoms or molecules excited to metastable levels onto the surface. Ions of the adsorbed material which are produced are analyzed, such as by a mass spectrometer.


**4 Claims, 2 Drawing Figures**



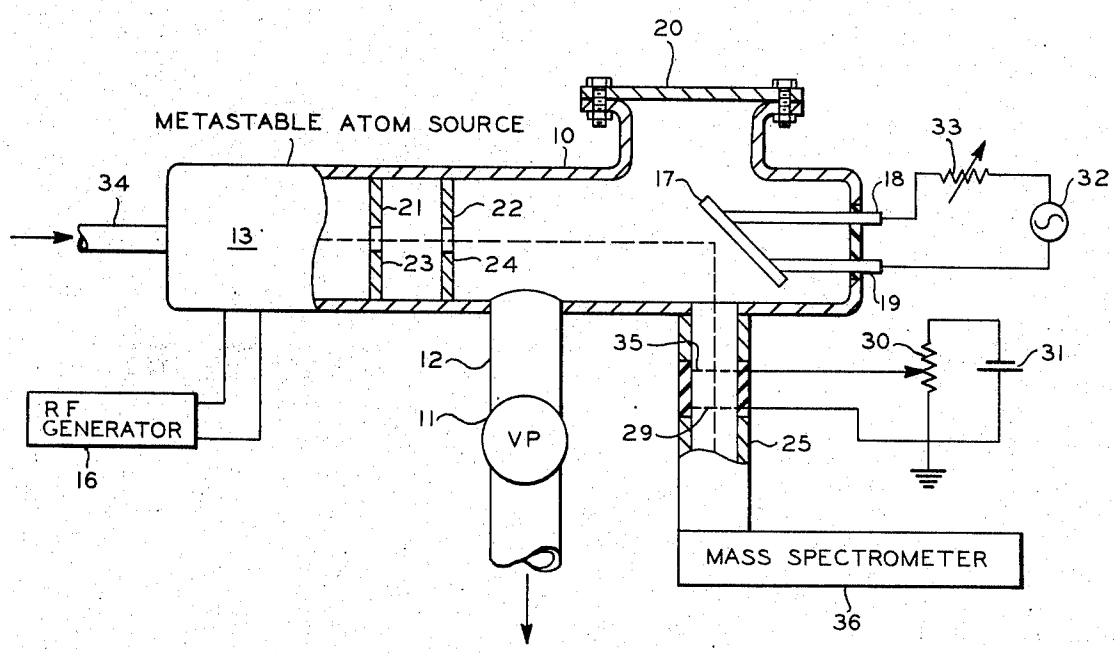



FIG. 1

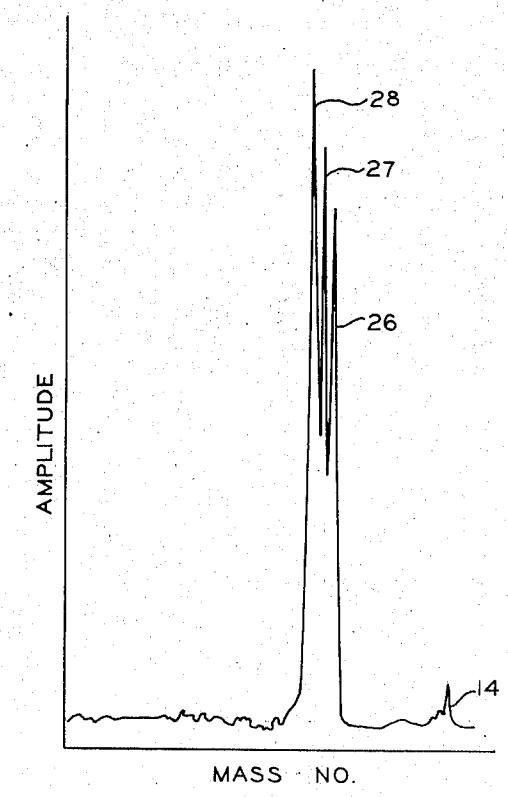



FIG. 2

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## SURFACE ANALYSIS

It is desirable to be able to analyze the surface of a body to determine materials adsorbed thereon and any catalytic effect produced by the body. Such an analysis is useful in the study of catalytic materials and catalytic reactions, for example. Analyses of this type are also useful in the study of selective adsorbents.

In accordance with this invention, a method is provided for studying the surfaces of bodies to determine materials adsorbed thereon. This is accomplished by bombarding the surface with a beam of atoms or molecules which are excited to metastable levels. The excited atoms or molecules ionize and eject materials which may be absorbed on the surface. The resulting ions are analyzed, such as by the use of a mass spectrometer, to provide information concerning the materials adsorbed and any catalytic effect of the surface. Apparatus useful in carrying out such a method is also provided in accordance with this invention.

In the accompanying drawing, FIG. **1** is a schematic representation of an embodiment of apparatus constructed in accordance with this invention. FIG. **2** is a graphical representation of a typical output signal from a mass spectrometer obtained in the practice of this invention.

Referring now to the drawing in detail and to FIG. **1** in particular, there is shown a housing **10**, the interior of which is maintained at a reduced pressure. This is accomplished by means of a vacuum pump **11**, the inlet of which is connected to the interior of housing **10** by a conduit **12**. A metastable atom or molecule source **13** is provided in one end of the housing. A stream of atoms or molecules to be energized is introduced into the source through a conduit **34**. A radio frequency generator **16** is connected to the source to establish a radio frequency discharge to excite the atoms or molecules introduced through conduit **13** However, other types of equipment can be employed to produce the excited atoms or molecules, such as equipment employing charge transfer, low voltage arc discharge, or cold cathode discharge, for example.

A surface to be studied is positioned in the opposite end of housing **10**. In the illustrated embodiment, a metal plate **17** is so positioned by means of support rods **18** and **19** which extend through the end of housing **10**. Housing **10** can be provided with an access port **20** to permit plate **17** to be installed. Plates **21** and **22**, which have respective apertures **23** and **24** therein, are disposed within housing **10** so that the apertures are in alignment between source **13** and plate **17**. This serves to direct a beam of excited atoms or molecules to plate **17**.

A conduit **25** communicates with housing **10** at a location adjacent plate **17** so that ions ejected from the surface of the plate are directed through the conduit. Conduit **25** communicates with the inlet of a mass spectrometer **36**. The mass spectrometer serves to detect the masses of the ions produced when the metastable particles impinge on plate **17**. A pair of spaced screens **35** and **29** can be disposed in conduit **25**. Screen **35** is connected to the contactor of a potentiometer **30**, and screen **29** is connected to one end terminal of the potentiometer. A voltage source **31** is connected across the end terminals of potentiometer **30**. By suitable adjustment of the contactor of the potentiometer, only ions having energy in a particular range are permitted to pass through screens **35** and **29** to the inlet of the mass spectrometer. The mass spectrometer can be any instrument capable of detecting the masses of the ions introduced therein. The masses of interest can be scanned periodically.



In some operations, it is desirable to maintain plate **17** at an elevated temperature. This can be accomplished by passing current through the plate from a source **32** which is connected in circuit with the plate by means of a variable resistor **33**. If plate **17** is constructed of an electrically conductive material, the plate itself may provide sufficient resistance to generate the desired amount of heat. Otherwise, an electrical heating element can be secured to the back of the plate. If tests are to be conducted at sub-ambient temperatures, a cooling coil can be connected to plate **17**.

In the operation of the apparatus of FIG. **1**, plate **17** is initially contacted by a material or materials to be studied. The surface is then positioned in housing **10** and subjected to bombardment by the excited atoms or molecules. The generated ions are representative of the material adsorbed on the surface and the state in which the material is adsorbed.

In a specific example of this invention, a platinum surface was exposed to an atmosphere of ethylene. The surface was then bombarded by helium atoms excited to the metastable state in a radio frequency discharge of about **7** Mhz. The resulting mass spectra of the ejected ions is illustrated in FIG. **2**. This is a spectrum representative of ethylene where mass **28** is due to a singly charged ethylene ion. The peaks at mass numbers **14, 26** and **27** represent ethylene fragments. It is thus apparent that some ethylene was adsorbed on the surface, but was not polymerized because no polymer spectrum was obtained.

If chemicals which undergo a reaction in the presence of a catalyst are contacted in the presence of the catalyst, ions representative of the reaction product are observed when the catalyst is bombarded by energized particles in accordance with this invention. The invention is thus quite useful in the direct study of catalytic reactions. Adsorbents can also be investigated to determine materials that are selectively adsorbed. Various molecules and atoms excited to the metastable level can be utilized in accordance with this invention. Numerous examples of such molecules and atoms are described in "Molecular Beams," Ross, Interscience Publishers, New York, 1966, page 172.

While this invention has been described in conjunction with presently preferred embodiments, it obviously is not limited thereto.

What is claimed is:

**1.** The method of analyzing a surface of a body having a material adsorbed thereon, which method comprises the steps of establishing a source of atoms or molecules which are excited to metastable levels, passing a beam of said excited atoms or molecules through a focusing means to impinge onto the surface to be analyzed so as to ionize the material adsorbed on said surface and eject from said adsorbed material ions representative of the material adsorbed on said surface, passing a beam of the ions so ejected from the adsorbed materail to an analysis means removing from the ions passed to the analysis means ions which have an energy level outside a predetermined range, and analyzing the remaining ions in a mass spectrometer.

**2.** The method of claim **1** wherein the source is established by subjecting helium to a radio frequency electromagnetic field to produce metastable helium atoms.

**3.** The method of claim **2** wherein the surface comprises platinum having a hydrocarbon adsorbed thereon.

**4.** The method of claim **1** wherein the surface comprises a solid material.

* * * * *

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,792,251 Dated: February 12, 1974

Thomas W. Schmidt

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 2, line 57, delete "materail" and insert therefor —— material ——; and column 2, line 57, after "means" insert a comma —— , ——.

Signed and sealed this 2nd day of July 1974,

(SEAL)
Attest:

EDWARD M. FLETCHER,JR.
Attesting Officer

C.MARSHALL DANN
Commissioner of Patents