United States Patent

Rupp, Jr. et al.

[15] **3,691,453**

[45] **Sept. 12, 1972**

[54] **COMPACT MICROWAVE SPECTROMETER**

[72] Inventors: **Louis William Rupp, Jr.**, Warminster, Pa.; **Walter Michael Walsh, Jr.**, Berkeley Heights, N.J.

[73] Assignee: **Bell Telephone Laboratories, Incorporated,** Murray Hill, Berkeley Heights, N.J.

[22] Filed: **April 28, 1970**

[21] Appl. No.: **32,538**

[52] **U.S. Cl.** .....**324/0.5 R**, 331/107 G

[51] **Int. Cl.** **G01n 27/78**

[58] **Field of Search**.....324/0.5 A, 0.5 AC, 0.5 AH; 331/107 G

[56] **References Cited**

UNITED STATES PATENTS

3,582,778 6/1971 Faulkner 324/0.5

OTHER PUBLICATIONS

L. W. Rupp, W. M. Walsh & A. Steinfeld– Simplified Microwave Frequency Electron Spin Resonance Spectrameter, Am. Journal of Physics, 38(2), Feb. 1970 pp.238– 242.

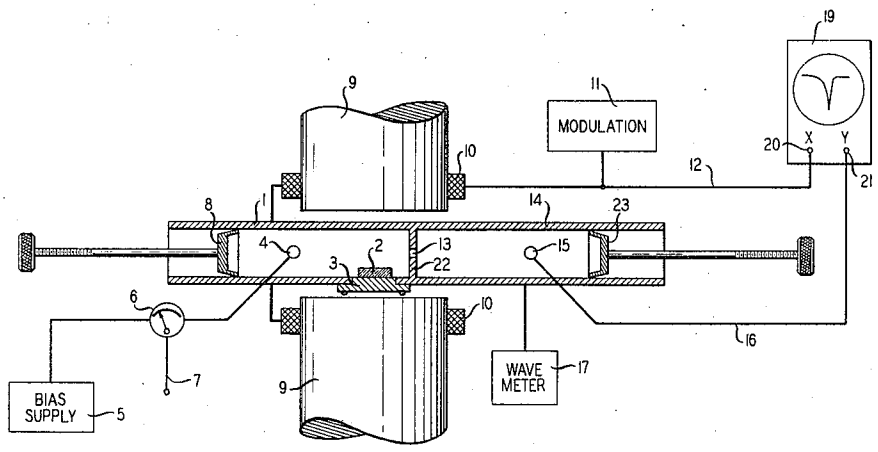
Primary Examiner—Michael J. Lynch

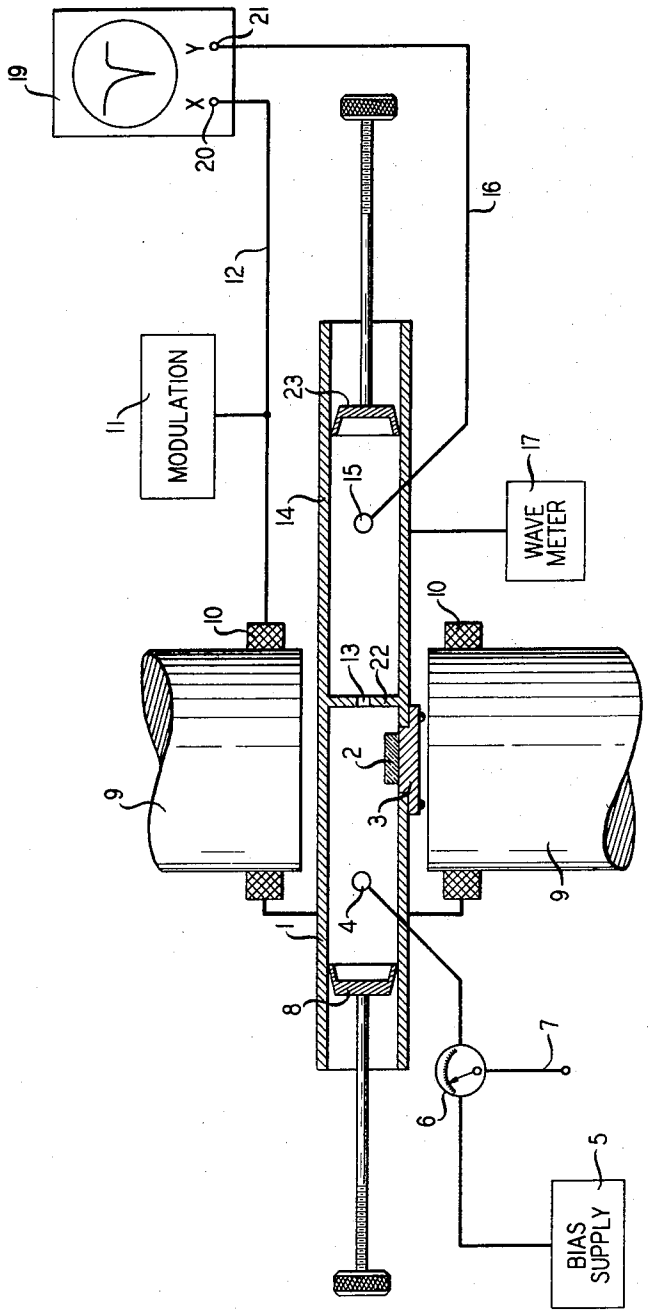
Attorney—R. J. Guenther and Edwin B. Cave

[57] **ABSTRACT**

The development of a simple and inexpensive microwave spectrometer for the study of spin resonance phenomena has been made possible by the inclusion of a solid state microwave energy source within the same resonant cavity as the sample to be investigated. A hypersensitive condition of oscillation of the microwave source has been found which leads to a spectrometer rivaling the sensitivity of much more complex and expensive conventional apparatus. Development models constructed using Gunn type microwave oscillator diodes show that, in addition to the use of conventional microwave detection, the output signal can be detected by observation of the diode bias current, obviating the use of a separate microwave detector.

2 Claims, 1 Drawing Figure





INVENTORS L. W. RUPP JR.
W. M. WALSH JR.
BY George S. Indig
ATTORNEY

COMPACT MICROWAVE SPECTROMETER

BACKGROUND OF THE INVENTION

1. Field of the Invention

The invention lies in the field of apparatus for measurement of the microwave absorption spectra of matter.

2. Description of the Prior Art

The microwave absorption of matter in a magnetic field is used extensively as a diagnostic tool by chemical, physical and biological investigators. A sample of the matter to be studied is placed in a d.c. magnetic field and irradiated with a microwave electromagnetic field. As the d.c. magnetic field is slowly varied, resonant absorption of the microwave energy is observed which is directly related to the electronic structure of the sample. From this, information about the sample can be derived. The simplest form of apparatus required for such a measurement includes a source of microwave energy such as a klystron, a length of waveguide within which the sample is fixed and a microwave detector. More sensitivity can be realized if the portion of waveguide within which the sample is located is made into a cavity by, for instance, the introduction of transverse walls with small coupling irises. However, this increased sensitivity brings along with it electronic complexity. Since two cavities are now involved, the cavity within the klystron and the sample cavity, an AFC loop between the microwave detector and the microwave source must be introduced to prevent the drift of the source frequency relative to the resonant frequency of the cavity containing the sample. Such drifts or fluctations would yield spurious signals. Instruments of this sort costing many thousands of dollars are now being marketed by instrument manufacturers for research use.

SUMMARY OF THE INVENTION

It has been found that the spectrometer can be greatly simplified if the microwave source and the sample are placed in the same cavity. If this is done the frequency of the generated microwave energy cannot drift relative to the resonant frequency of the sample cavity, since the generated frequency is defined by this same cavity. This allows the elimination of the AFC loop and allows the construction of a simple compact and inexpensive spectrometer. Since this requires that the source be in the magnetic field supplied to the sample, a source whose frequency does not change appreciably with changing magnetic fields is required. It was found that semiconducting microwave oscillator diodes meet this requirement. In the low to medium ranges of the microwave spectrum high field domain (Gunn) type devices are suitable. At higher frequencies, into the millimeter range, LSA, IMPATT or tunnel devices may be used.

Greatly increased sensitivity has been observed in some experimental models when the diode voltage is reduced below that voltage producing maximum power output. This "hypersensitive" operation leads to spectrometer sensitivity rivaling that of much more expensive apparatus. The attainment of this hypersensitive operation depends upon the characteristic of the particular device used and on the electrical loading.

Under favorable circumstances an output signal can be derived directly from the observation of the source bias current, thus eliminating the need for a separate microwave detector. Microwave spectrometers built along the lines described above may be used for production line testing (e.g., detection of impurities) or as a pedagogical tool in undergraduate and graduate college laboratories as well as for scientific research.

BRIEF DESCRIPTION OF THE DRAWING

The drawing is a schematic representation of a microwave spectrometer of the disclosed type with ancillary instrumentation.

DETAILED DESCRIPTION OF THE INVENTION

Basic Spectrometer

The drawing is a schematic representation of a basic spectrometer of the type disclosed here. The sample cavity **1** is a length of waveguide which, in the experimental model constructed, was rectangular in cross section. Situated within the cavity is the sample to be measured **2**. The sample **2** may be placed within the cavity through an entrance port provided **3** or other insertion means so arranged as not to require the disassembly of the spectrometer. Also placed within the cavity is the microwave oscillator diode **4** which serves as the source of the microwave electromagnetic field. In the low to medium ranges of the microwave spectrum, high field domain (Gunn) type devices are most suitable. At higher frequencies, into the millimeter range LSA, IMPATT or tunnel devices may be used. This source **4** is energized by a d.c. current from the bias supply **5**. This bias supply may be provided with a current measuring device **6**, which may be simply a series resistor. One end of the cavity is formed by a transverse conducting wall **22** and the other end by a sliding short **8** which is used to tune the cavity. The cavity may also be provided with other tuning devices such as a tuning screw, which may be a metal screw protruding through the wall of the cavity. This screw may be employed to adjust the frequency of the cavity or distort the field pattern within the cavity in order to achieve optimum coupling to the sample. Once the required cavity length has been determined either theoretically or experimentally, the sliding short **8** may be eliminated and a fixed short circuit wall can be substituted or, in some cases, the cavity can be terminated in an open circuit by merely cutting the waveguide to the appropriate length.

A d.c. magnetic field is applied to the sample by a magnet whose pole pieces **9** are illustrated. In order to observe the resonant absorption the magnitude of the magnetic field is varied by small amounts relative to the large d.c. field by means of the modulating coils **10**. The modulating coils are supplied by the modulation source **11** which also may supply an output **12** to the X-axis input **20** of a display device **19** such as an oscilloscope. In order to observe the magnitude of the microwave field, a microwave detector **15** can be used. In this case a small iris opening **13** is supplied in the conducting transverse wall **22** and the detector diode **15** is placed within a cavity **14** with its own sliding short **23**. This sliding short is used to optimize the coupling of the microwave field to the detector **15**. The detector cavity **14** can also be supplied with a frequency measuring device such as a cavity-type wave meter **17**. If a

cavity-type wave meter is used, the detector cavity can be made to exhibit the frequency changes which occur during the resonant absorption of the sample by adjusting the wave meter **17** to the point where the meter **17** has a large change of absorption with frequency. The detected signal from the diode **15** can be lead to the Y-input **21** of the display device **19**. The display device can be provided with a suitable electronic amplifier in order to achieve the required level of sensitivity. Greatest sensitivity usually requires the use of a lock-in detector. It has been found in some cases, usually for the more efficient oscillator diodes **4** that the absorption of the sample **2** can be observed in the current which is drawn by the diode **4** from the bias supply **5**. In such cases an output **7** can be taken from the current measuring device **6** and lead to the Y-input **21** of the display device **19**. This eliminates the need for the microwave detector **15** and its cavity **14**.

Hypersensitive Operation

During work with experimental models such as illustrated above, it was found that the microwave oscillations could be made to exhibit a hysteresis effect. That is as the voltage on the bias supply **5** is increased from zero, no microwave oscillation is observed until the upward breakover point typically in the neighborhood of 8 to 10 volts in the devices used, is reached. As the voltage increases further, the power output of the diode **4** reaches a maximum and then starts to decrease. If the voltage is then slowly decreased oscillation is maintained to voltages less than the upward breakover point until the downward breakover point is reached, typically between 6 and 7 volts in the devices used. It was observed that in the region of oscillation between the upward breakover point and the downward breakover point, the sensitivity of the spectrometer shows a marked increase. This region of operation is referred to here as the hypersensitive region. The attainment of this hypersensitive operation depends primarily on the diode used and on the electromagnetic loading of the cavity. It is believed that relatively lightly doped Gunn and LSA devices are preferable for this hypersensitive operation [(doping x length) less than $\sim 10^{13}$ cm^{-2}].

Sensitivity

A standard measure of the sensitivity of microwave spectrometers is the minimum number of paramagnetic electron spins which can be observed with a one-to-one signal-to-noise-ratio, in a measuring device with a one cycle per second bandwidth, if the width of the absorption line is one oersted. According to this measure the most sensitive high quality experimental devices presently available can detect as few as 10^{11} spins. The spectrometer disclosed here, when biased for optimum power output, can detect as few as 10^{14} spins. However, when operated in the hypersensitive region, the sensitivity is increased by an order of magnitude and as few as 10^{13} spins can be observed. This level of sensitivity is quite useful for all but the most demanding research experiments.

The spectrometer presented in the drawing is intended to be merely illustrative of the various elements which may be employed in any particular apparatus design. In addition to the variations described above, the possible variation of cavity design and cross section known to the art are many fold but would be simple extensions of the basic ideas disclosed here.

What is Claimed is:

1. Method for the investigation of the properties of matter by measurement of the resonant interaction between electromagnetic fields of frequencies greater than 3 x 10^8 cycles per second and a portion of the matter comprising:
   a. placing the portion in a resonant cavity, which cavity includes a semiconductor microwave oscillator device as a source of the electromagnetic field;
   b. applying a variable magnetic field to the portion;
   c. applying a bias voltage to the semiconductor microwave oscillator device;
   d. adjusting the bias voltage of said oscillator device to a level at least 5 percent below the level of said bias voltage required to initiate the production of said electromagnetic energy so that said source of electromagnetic energy is oscillating in a hypersensitive state; and
   e. observing an output signal dependent upon the resonant interaction between the portion and the electromagnetic field.

2. Apparatus for the investigation of the properties of matter by measurement of the resonant interaction between electromagnetic fields of frequencies greater than 3 x 10^8 cycles per second and a portion of the matter subjected to a varying magnetic field, said apparatus comprising:
   a. a resonant cavity for the insertion of the portion;
   b. a semiconductor microwave oscillator device positioned in said cavity as a source of the electromagnetic field;
   c. insertion means required to insert the portion within the resonant cavity at a position which is favorable for the interaction of the portion with the electromagnetic field; and
   d. resonant signal output means including means for the measurement of the variation of the bias current through the semiconductor microwave oscillator device as correlated with the variation of the magnetic field.

* * * * *