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Attia

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[54] **METHOD AND APPARATUS FOR EVALUATING THE GAS CONTENT OF MATERIALS**

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[52] **U.S. Cl.** **250/281,** 250/287, 250/430, 250/457, 250/542, 250/544

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[58] **Field of Search** 250/281, 289, 457, 430, 250/542, 543, 544; 313/7, 174

[56] **References Cited**

UNITED STATES PATENTS

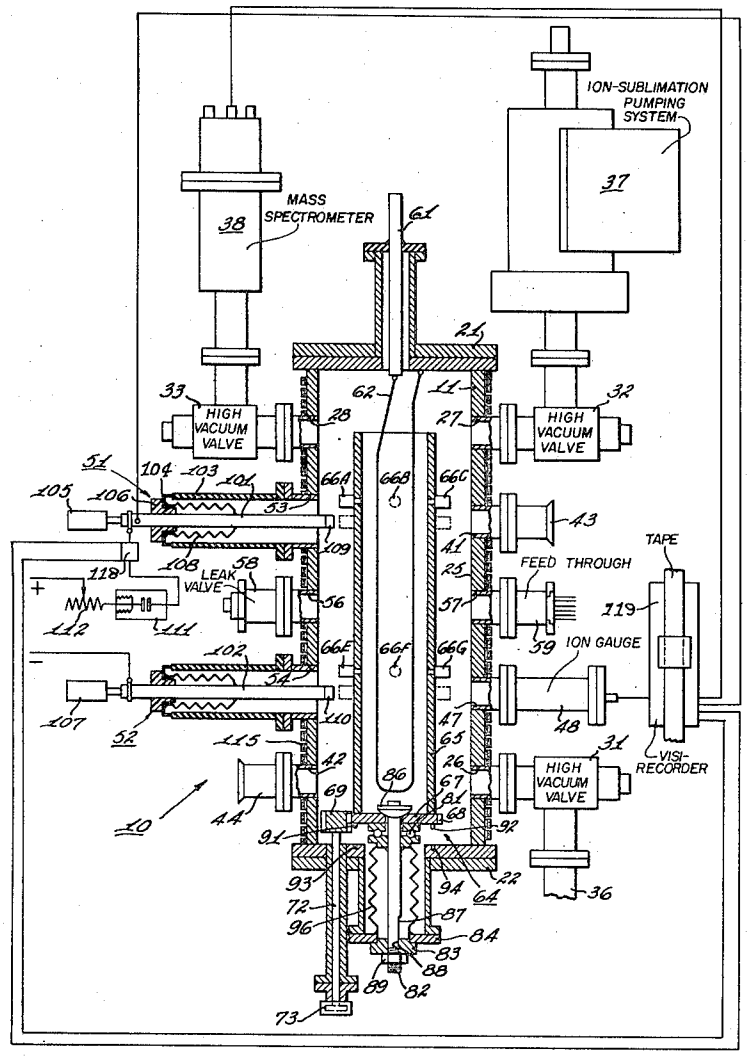
3,400,066	9/1968	Caswell et al.	313/174 X
3,591,827	7/1971	Hall	313/7
3,751,989	8/1973	Briggs	250/430

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[57] **ABSTRACT**

A highly sensitive gas analysis method and apparatus to practice the method is provided to evaluate the gas content and predict the expected performance of a vacuum interrupter using specific contact material analyzed.

11 Claims, 2 Drawing Figures



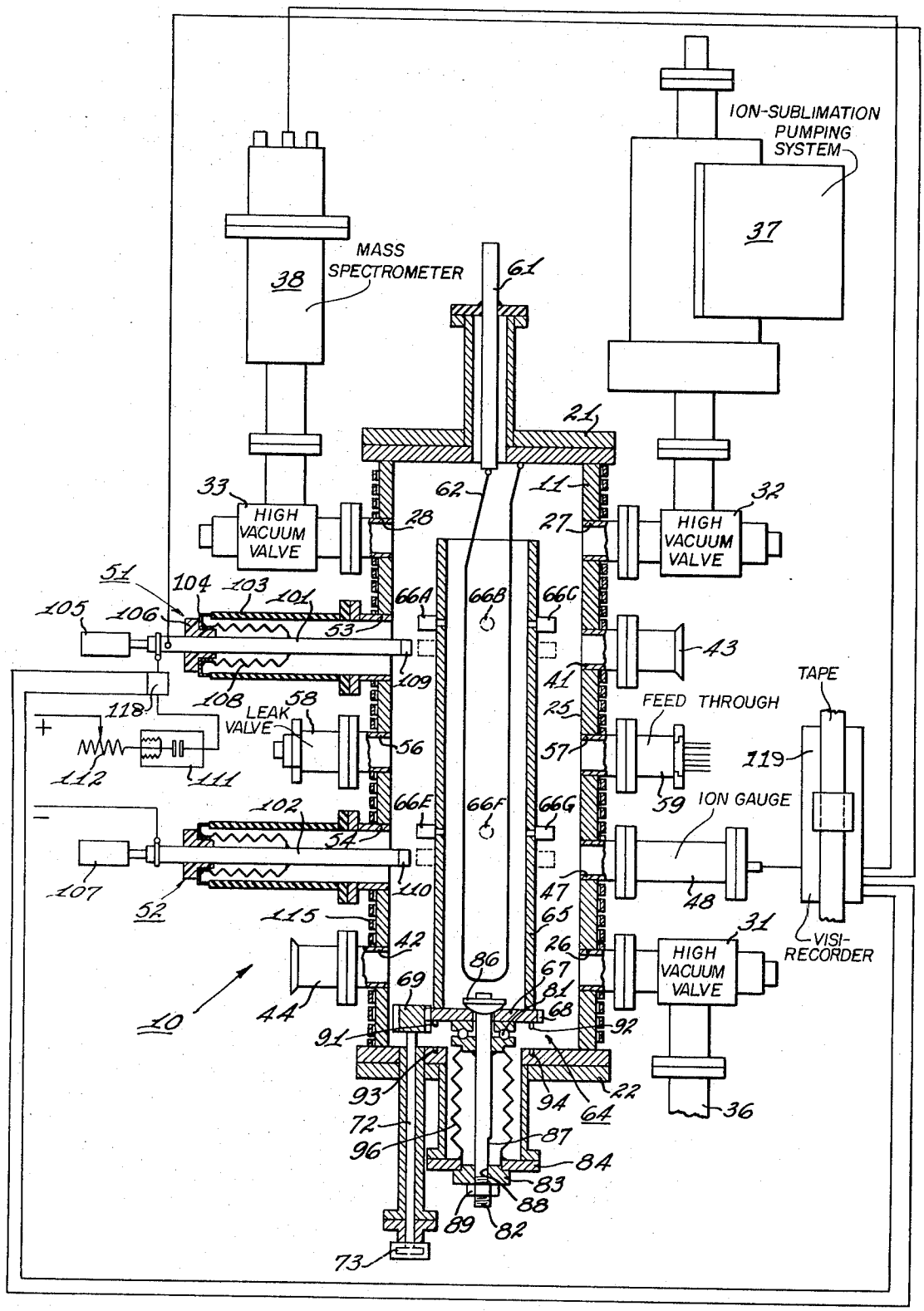

ION-SUBLIMATION PUMPING SYSTEM
37
MASS SPECTROMETER
38
61
21
HIGH VACUUM VALVE
33
28
HIGH VACUUM VALVE
32
27
62
11
51
104
103
101
105
106
53
66A
66B
66C
43
41
109
108
118
LEAK VALVE
58
56
112
111
FEED THROUGH
25
57
59
TAPE
119
ION GAUGE
102
66E
66F
66G
54
107
52
110
47
48
115
65
42
26
31
HIGH VACUUM VALVE
VISI-RECORDER
86
67
81
69
68
10
44
91
92
93
94
64A
22
36
72
96
87
84
83
88
89
82
73

Fig.-1

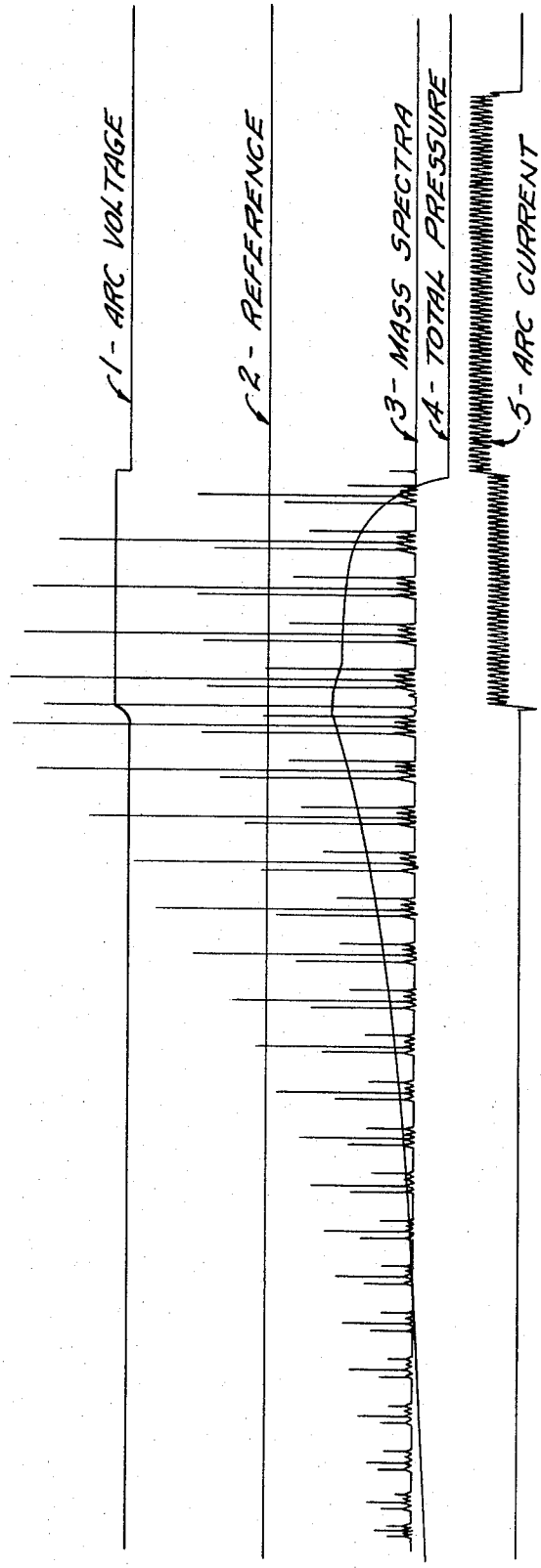

1- ARC VOLTAGE
2- REFERENCE
3- MASS SPECTRA
4- TOTAL PRESSURE
5- ARC CURRENT

Fig. 2

METHOD AND APPARATUS FOR EVALUATING THE GAS CONTENT OF MATERIALS

CROSS REFERENCES

Applicant is not aware of any patents or other information which relate to the present invention.

BACKGROUND OF THE INVENTION

Successful performance of vacuum interrupters is dependent upon the contacts, or more specifically, on the gas purity of the material of which the contacts are made. To predict the performance of vacuum interrupters it has been the practice to analyze contact samples individually in experimental interrupters. This method is costly and time consuming since one sample is analyzed at a time. In analyzing individual contacts in experimental interrupters, great care is required to maintain conditions similar for each sample analyzed so that useful comparison can be made. With this method, usually a larger variation or scatter of the data has been observed. Other conventionally known gas analysis methods, e.g., fusion analysis, Auger spectroscopy, X-ray analysis, etc., the gas density at the surface of the contact and in the bulk. Also, some of these methods fail to analyze certain gases (e.g., hydrogen by activation analysis and X-ray fluoresence). Further, the sensitivity of most conventional gas analysis methods is inadequate for the purpose at hand.

SUMMARY OF THE INVENTION

The system is composed of a vacuum chamber which is adapted to receive three high vacuum valves which operate to connect or disconnect an ion sublimation pumping system; another of the valves is connected to receive a mass spectrometer analyzer tube, while the third valve connects a roughing system to the vacuum chamber. Included in the system is an ion gauge and an arc initiating mechanism. The mass spectrometer is utilized to analyze the type (composition) of the gas occluded in the test sample as it is released by arcing while the ion gauge measures the total amount of the gas. An electrical feed-through is provided and is connected to an internal heater which is operable to effect a bakeout which precedes the testing and is intended to free the surfaces from adsorbed gas species and drive them to the pumping system. Within the vacuum chamber, a carrousel is operably disposed to carry test contact samples that are moved in a rotational path of travel to position the sample materials opposite the arc initiating anodes so that the sample materials in effect become the cathodes. With the system provided, it is possible to analyze several samples in one system run and to effect both exhaust, bakeout, etc., thus reducing the considerable time necessary in testing the samples in individual effort. Also, by analyzing the samples under the same conditions, a valid comparison between the test samples introduced can be obtained from the analyzed data. In addition, the described test setup allows accurate determination of the arcing time, arcing current and arc voltage, which are important factors for an accurate estimation of the gas content and for a valid prediction of the expected behavior of the contact material under evaluation.

It is therefore an object of the present invention to provide a method and means for determining the gas content of a plurality of material samples by d.c. arcing followed by partial and total pressure measurements.

Still another object of the present invention is to develop and provide a highly sensitive gas analysis system to evaluate the gas content and predict the expected performance of a vacuum interrupter using the specific contact material analyzed.

Yet another object of the present invention is to provide a system wherein a plurality of samples can be analyzed at the same time and under the same conditions.

A further object of the present invention is to provide a system for analyzing several material samples under the same conditions and compare them to a control or a standard sample, thus establishing a reference for comparison and the results obtained thus provide data having more meaningful information.

DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic representation of a system which may be utilized in practicing the method of the present invention; and,

FIG. 2 is a graph representative of a display obtained from a Visi-recorder indicating the various data obtained from a sample analysis.

DESCRIPTION OF THE INVENTION

As shown in FIG. 1, the system 10 includes a cylindrical housing 11 that is sealed by sealing end plates 21 and 22 to form a vacuum chamber 25 therein. Connected to communicate with the vacuum chamber 10 through ports 26, 27 and 28 are three high vacuum valves 31, 32 and 33, respectively. The valve 31 is operable to connect or disconnect a roughing system 36 which is utilized to initially evacuate the chamber 25 prior to utilization of an ion-sublimation pumping system 37. The ion-sublimation pumping system 37 is connected to the chamber 25 via the port 27. A mass spectrometer 38 is operably connected to the valve 33. Ports 41 and 42 are utilized to accommodate viewing windows 43 and 44 through which the operator may visually inspect the interior of the vacuum chamber 25. Another port 47 accommodates an ion gauge 48 for monitoring of the total pressure within the vacuum chamber 10 during the various processes (e.g., during pumping down, before arcing, during arcing, after arcing, etc.). Two arc initiating mechanisms 51 and 52 are accommodated in ports 53 and 54, respectively. In addition, two other ports 56 and 57 are provided in the vacuum chamber cylinder 11 which are adapted to receive leak valve 58 and a feed-through 59, respectively. The leak valve 58 is used to calibrate the mass spectrometer 38 while the feed-through 59 is utilized to introduce thermocouples (not shown) to measure the temperature during the bakeout process which precedes the testing of the samples. The sealing end plate 21 accommodates an electrical feed-through 61 which communicates with the interior of the vacuum chamber 25. The inner end of the electrical feed-through 61 has an operative electrical connection to one end of a molybdenum heater 62 which is grounded to the top sealing end plate 21. Within the vacuum chamber 25 is a carrousel 64 having an electrically conductive upstanding standard 65 which is adapted to carry eight material samples 66 of which six samples 66A, 66B, 66C, 66E, 66F and 66G are shown.

The material samples 66 to be tested are carried in two annular rows with each annular row having four test pieces spaced 90 degrees apart as depicted in FIG. 1. Thus the upper annular row is provided with material

samples 66A, 66B, and 66C, as well as one other sample (66D, not shown) all of which are spaced 90 degrees apart relative to adjacent samples. Similarly, the bottom annular row is provided with material samples 66E, 66F and 66G, as well as an additional sample (66H, not shown) all of which are also spaced 90 degrees apart relative to adjacent samples. The samples include samples for evaluation as well as control or standard sample.

In order to effect the testing of a sample, the carrousel 64 is rotated 90° so that the samples 66A and 66E are positioned adjacent the two arc initiating mechanisms 51 and 52. To effect such rotation of the carrousel 64, the base 67 of the carrousel is formed with a peripheral gear 68 that is adapted to mesh with a spur gear 69 when the carrousel 64 is in elevated released position as shown. The spur gear 69 is carried on the inner end of a mechanical feed-through shaft 72, the opposite outwardly extending end of which is provided with means 73 for effecting a rotation of the shaft 72. The means 73 which is preferred is a magnetic drive; however, the means 73 may be a handwheel, a handcrank or a gear whereby the power input to the shaft may be effected if so desired. When the carrousel 65 has been rotated 90 degrees to position the particular samples 66A and 66E into position for cooperation with the arc initiating mechanisms 51 and 52, the carrousel 65 will be lowered and effectively clamped in position. To this end the carrousel 65 is disposed to rotate on an antifriction bearing 81 carried by a draw-rod 82. In order to draw the carrousel 64 downwardly into clamped engagement with the sealing end plate 22, the draw-rod 82 extends through a sealing bushing 83 disposed in a sealing plate 84 and into the vacuum chamber through the carrousel base plate 67. The inner end of the draw-rod 82 is provided with a conical thrust washer 86, the convex surface of which is adjacent the base plate 67 to provide for a minimum of frictional engagement between the washer 86 and the base plate 67 during rotation of the carrousel 64. The lower end of the draw-rod 82 extends through the sealing bushing 83 and has a flat surface 87 which engages a flat 88 in the bore of the sealing bushing 83. Thus, the draw-rod 82 is permitted to move axially but is prevented from rotating by means of the engaging flat surfaces 87, 88. The antifriction bearing 81 is affixed to the rod 82 so as to move axially with the rod. For effecting the axial movement of the rod 82 for drawing the carrousel 64 downwardly into a fixed desired position, the lower end of the rod 82 is threaded and receives a nut 89. By rotating the nut 89 in one direction so as to engage the nut on the rod, the rod is caused to move axially downward thereby effecting engagement of the washer 86 with the base plate 67 to draw the entire carrousel 64 and the bearing 81 downwardly. As the carrousel 64 moves downwardly a pair of studs 91 and 92 disposed 180 degrees apart on the lower face of the base 67 engage in suitable openings 93 and 94 provided in the inner surface of the sealing end plate 22. Thus, when the carrousel 64 has been drawn downwardly the material samples, such as the samples 66A and 66E, will be moved into the position depicted by the broken line of the samples. In this position the studs 91 and 92 are engaged in the openings 93 and 94 in the end plate thereby locating the carrousel 64 in a fixed position. It is to be understood that while two studs 91 and 92 located 180° apart are illustrated there is also another pair of studs located 180° apart, all studs being spaced 90 degrees apart relative to adjacent studs. Thus, in any 90 degree position of the carrousel 64, the carrousel when in a lowered position will be held in that position. A bellows 96, tubular in form, is provided to maintain the vacuum seal of the chamber 25 and is welded at its innermost end to the lower race of the bearing 81 and to the lower sealing end plate 84.

With the carrousel 64 located in a locked position, the material sample, such as the material samples 66A and 66E as previously mentioned, are located opposite the electrodes of the arc initiating mechanisms 51 and 52. To effect electrical engagement of the electrodes 101 and 102 of the arc initiating mechanisms 51 and 52, respectively, with the material samples 66A and 66E, the electrodes 101 and 102 are axially movable into engagement with the samples. To this end, the arc initiating mechanisms 51 and 52 are identical and each are constructed with the sample design. Thus, the arc initiating mechanism 51 is comprised of an insulating ceramic housing 103 which is secured in sealed relationship to the flange of the port 53. The outer end of the ceramic feed-through is provided with a metallic flanged collar 104 that is sealed as being brazed or welded to a bellows 108. The feed-through electrode 101 is supported in a bearing 106 which is preferred to be a nylon bearing and that is disposed in substantially coinciding axial alignment with the sample 66A. The bellows 108 as previously mentioned is brazed to flanged collar 104 and at its inner end is also brazed to the peripheral surface of the feed-through electrode 101. The extreme inner end of the electrode 101 is provided with a contact 109 which serves as an anode during arcing. The axial inner movement of the electrode 101 is controlled by a solenoid 105 in a well-known manner which is activated on demand to cause the electrode 101 to move axially outwardly thereby separating the contact 109 from the material sample 66A under test and which is now serving as a cathode thereby initiating an arc. The bushing 106 is utilized to align the movable electrode 101 so that the contact 109 may be positioned to meet precisely with the material sample 66A when the contact 109 is in the closed position.

The successful use of the arc induced vaporization and mass spectrometric analysis of the released gases technique to evaluate the gas content of a material relies, in the first place, on the careful application of advanced vacuum technology so that effects such as desorption from the test chamber internal surfaces and gas leak through vacuum seals is negligible in comparison to the total amount of gas evolved by arcing. Desorption from the gas chamber internal surfaces is thought to be caused by the thermal quanta received by conduction or radiation from the arc active zone. It may also be caused by surface bombardment or by arc products which stream away from the arcing gap in ionized form and impinge on the bounding surfaces with a mean energy of 20 to 60 eV, which may be acquired by acceleration from a potential maximum in the cathode fall. Leaks at the test chamber seals are minimized by constructing the chamber and the parts from 304L stainless steel and using copper shear seals at the ports between the main chamber and the other sections of the system.

To reduce desorption effects from the chamber's internal surfaces, the test chamber is baked out before

testing, while being exhausted by an ion-sublimation pumping system, for a total time of 36 hours including 10 hours holding at 400°C. and 10^{-8} Torr. At the start of the exhaust cycle the test chamber is roughed by a mechanical pump furnished by a cryogenic trap to reduce pressure to a level where the ion-sublimation pumping system can be started usually at 10^{-4} to 10^{-5} Torr. The bakeout is then effected by energizing the heater **62**. The chamber may also be surrounded from the outside by an oven or heating tape **115** to insure a uniform bakeout of all the parts.

For arc erosion of the test samples, an electrical circuit composed of a regulated power supply with open circuit voltage of approximately 100 volts d.c. is connected in series with the arc initiation feed-through electrodes **101** and **102**, a back-up breaker **111** and a variable resistance **112** to control the current in the circuit. The arc initiation feed-through electrodes **101** and **102** are connected in the electrical circuit in a manner that when the sample **66**A, for example, is to be arced, the electrode **101** is connected to the positive polarity of the circuit and the electrode **102** is connected to the negative polarity. In this way, the sample **66**A during arcing would be a cathode and the contact **109** of the electrode **101** an anode. Likewise, when it is required to arc the sample **66**E the electrode **102** is connected to the positive polarity of the circuit and the electrode **101** to the negative polarity thus causing the test sample **66**E to be the cathode during arcing. The mention of the arc initiating feed-through electrodes is controlled by magnetic solenoids **105, 107.** In the normal position, the contacts **109** and **110** at the internal end of the electrodes **101** and **102**, respectively, and the samples are contacting or are in closed positions. During a sample testing, however, one solenoid is used, that is the one controlling the motion of the electrode to become an anode during arcing, while the other solenoid is not in use and the electrode connected to it is merely used to conduct the current to the carrousel **64** carrying the samples. The solenoid to be used and the control mechanism of the back-up breaker are electromechanically actuated and controlled by time delay relays. In operation, the back-up breaker is closed manually to permit the current to flow in the circuit. After an interval of approximately 1.5 seconds, an automatic cycle starts in which the arc initiating electrode facing the sample to be tested is parted to initiate an arc which is maintained for a predetermined arcing time interval of between 0.5 and 1 second. Thereafter the opening of the back-up breaker is accomplished to extinguish the arc.

A precision resistor **118** having a resistance of 0.05 ohms, for example, may be inserted between the back-up breaker **111** and the electrode **101** to measure the current in the circuit during the flow of current and during arcing. The voltage across the resistor **118** is measured using the Visi-recorder **119** which is initially calibrated to read in volts. The current, in amperes, will be equal to the voltage divided by 0.05. The arc voltage is equal to the potential difference between the movable electrode **101** and the ground potential and is measured using the Visi-recorder **119** which, as previously mentioned, is calibrated to read in volts.

During arcing the total pressure caused by the arc evolved gas and the composition of this gas is evaluated from the ion gauge and mass spectrometer readings respectively. Both the ion gauge and the mass spectrometer tube are furnished with Thoria coated iridium filaments and are operated at an emission current of 1 milliampere or less. In this way, it is possible to reduce to an insignificant value effects such as ion gauge and mass spectrometer pumping and new gas formation. The mass spectrometer is calibrated (using the major gases of interest, that is, H_2, CO, CH_4, CO_2, etc., which may be leaked during calibration to the systems through valve **58**) against the ion gauge which was previously calibrated against a McLoed gauge. During testing five parameters are of interest and are, therefore, measured. These parameters are: the arc voltage, the arc current, the duration of the arc, the total pressure caused by the arc released gas and the type (composition) of this gas. The data is acquired by using a six-channel Visi-recorder **119** and calibrating the different channels to read directly the parameters of interest. The arcing time is obtained from a knowledge of the Visi-recorder chart speed and then measuring the length of the arc current or voltage trace. The mass spectrometer a quadropole of 10^{-14} Torr. N_2 sensitivity and negligible response time is adjusted to scan repeatedly between mass 1 and 100 AMU. The scan time is approximately 0.2 seconds. FIG. 2 depicts a sample of a Visi-recorder display during an arcing event of a pure Cu. sample. Reading from right to left as viewed in FIG. 2, curve 1 is the arc voltage, the value of which is indicated by the step in the curve. Curve 2 is a reference time base intended to calibrate the chart speed. Curve 3 is the mass spectra of the arc released gases and show peaks at masses **2, 12, 15, 16, 28** and **44** indicating the revolution of H_2, CH_4, CO and CO_2. Curve 4 is the total pressure as measured by the ion gauge. Curve 5 is the arc current and shows two steps, the first indicates the establishing of the current in the circuit and the second indicates the value of the arc current. The arc duration is the length as compared to the reference time base of the second step in curve 5 representing the arc current or the step in curve 1 representing the arc voltage.

From a knowledge of the total pressure, the volume of the test chamber and the percentage composition of the release gaseous species, the type and quantity of the gas released during arcing is estimated. The gas content is calculated by relating this result to the estimated quantity of the material eroded during arcing, which is calculated from the average erosion rate (weight loss per unit of arc energy) and the arc energy dissipated during arcing. The average erosion rate is calculated by weighing the test sample before and after arcing to obtain the total erosion then dividing by the total arc energy the sample experienced during testing. Approximately 5 to 10 arcs should be drawn and the gas content evaluated after each arc to obtain an accurate value for average gas content. By including within the samples a standard or a sample known to have a certain amount of gas, the results obtained from the various samples may be compared with this standard and a reference may therefore be established. The homogenity of the gas content may also be checked by arcing the test sample to various depths from the surface and estimating the gas content versus the depth from the surface.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A method for evaluating material to be utilized in a vacuum interrupter comprising the steps of:

arranging the material in a vacuum chamber;
connecting the material in an electrical d.c. circuit in series with an electrode that is connected to the positive polarity of the circuit so as to function as an anode with the material serving as the cathode;
effecting a separation between the electrode and the material to draw an arc for a predetermined time interval;
interrupting the circuit to extinguish the arc;
determining the gas pressure of the gas evolved during arcing and the composition of the gas evolved; and,
comparing the results obtained to control data.

2. A method for evaluating material to be utilized in a vacuum interrupter comprising the steps of:
arranging the material in a vacuum chamber;
connecting the material in a d.c. circuit having a regulated power supply with open circuit voltage of approximately 100 volts and in series with an electrode that is connected to the positive polarity of the circuit and functions as an anode with the material functioning as the cathode;
position the material in closed circuit relationship with the electrode;
permitting current to flow through the closed circuit for approximately 1.5 seconds;
effecting a separation between the electrode and the material to initiate an arc;
maintaining the arc for a predetermined time interval;
determining the total pressure caused by the arc evolved gas and evaluating the composition of the gas evolved; and,
comparing the results obtained to control data.

3. A method for evaluating material to be utilized in a vacuum interrupter comprising the steps of:
arranging the material in a vacuum chamber;
baking out the vacuum chamber with the material therein for approximately 36 hours at 400°C and exhausting the chamber to a vacuum of 10^{-8} Torr.;
connecting the material as a cathode in a d.c. circuit composed of a regulated power supply with open circuit voltage of approximately 100 volts;
closing the circuit to permit current to flow in the circuit for approximately 1.5 seconds;
effecting a separation of the material from the anode portion of the circuit to draw an arc for a time interval of approximately 0.5 to 1 second;
measuring the arc voltage during the arcing period;
measuring the arc current during the arcing period;
measuring the duration of the arc;
measuring the total pressure caused by the arc released gas;
determining the composition of the gas released during the arcing period; and,
comparing the results obtained to control data.

4. A method for evaluating material to be utilized in a vacuum interrupter comprising the steps of:
arranging the material in a vacuum chamber as a cathode in a d.c. electrical circuit having a separable anode;
effecting a bake-out of the chamber while evacuating the chamber to a vacuum of 10^{-8} Torr.;
initiating current flow in the d.c. circuit for a time interval of approximately 1.5 seconds;
effecting a separation of between the anode and the cathode material to draw an arc for a predetermined arcing time interval of 0.5 to 1 second;
extinguishing the arc;
effecting a measurement during the arcing interval of the arc voltage, the arc current, the duration of the arc, the total pressure caused by the arc released gas and the composition of the released gas;
recording the measurements on a chart to provide a visual record; and,
comparing the results obtained to control data.

5. In apparatus for evaluating contact material to be utilized in a vacuum interrupter, said apparatus having a sealed housing in which a vacuum is established and comprising;
a support within said chamber for carrying the contact material to be evaluated;
a d.c. circuit including an electrode which is connected to the positive polarity of said circuit, said electrode being movable into and out of engagement with the contact material;
means connecting the contact material to be evaluated to the negative polarity of said d.c. circuit;
means operably connected to effect a separation between said electrode and the material to establish an arc; and,
means operably connected to the interior of said vacuum chamber to measure the arc voltage, the arc current, the duration of the arc, the total pressure caused by the arc released gas and the composition of the arc released gas.

6. Apparatus according to claim **5** wherein said measuring means comprising an ion gauge connected to the interior of vacuum chamber and operable to measure the total amount of the arc released gas; and,
a mass spectrometer connected to the interior of the vacuum chamber and operable to analyze the composition of the occluded gas in the contact material as it is released by arcing.

7. Apparatus according to claim **5** wherein said material support is angularly positionable within said vacuum chamber and operable to carry a plurality of material specimens for individually locating each specimen in axial alignment with said electrode; and,
drive means connected to effect the angular positioning movement of said support for locating a selected arc of said specimens in axial alignment with said electrode.

8. Apparatus according to claim **7** wherein said angularly positionable support includes a gear drive internally of said vacuum chamber and having actuating means operably connected thereto and extending externally of said vacuum chamber for effecting a positioning movement of said support for locating said material in alignment with said electrode.

9. Apparatus according to claim **8** wherein there is provided locking means operative to secure said support in a preselected angular position in which position the material carried by said support is locked in aligned relationship to said electrode; and,
mechanical means operatively connected to said support and extending outwardly of said vacuum in scaled relationship for effecting the release of said support from said locking means or for effecting the engagement of said locking means with said support.

10. Apparatus according to claim 9 wherein said locking means are interengageable components one of which is carried by said support and the other being on said housing;

mechanical operating means to move said support for effecting the disengagement of the support locking component from the locking component of said housing, said mechanical means being connected to said support and constructed and arranged to extend outwardly of said vacuum chamber in sealed relationship;

operating means external of said vacuum chamber and operatively connected to said mechanical operating means to actuate said mechanical operating means for moving said support in a locking or unlocking direction of movement.

11. In apparatus for evaluating the material to be utilized in a vacuum interrupter;

a sealed housing having a vacuum chamber;

a pumping system connected into said chamber to evacuate it;

a heater within said vacuum chamber and operable to effect a bakeout of said chamber;

a support within said chamber on which material to be tested is carried;

an electrode supported in sealed relationship by said housing and extending into said chamber, said electrode being axially positionable in engagement with the material supported within said chamber;

a d.c. circuit having its positive polarity connected to said electrode and its negative polarity connected to said material;

means to effect a separation of said electrode from the material to draw an arc; and,

means to measure the arc voltage, the arc current, the duration of the arc, the total pressure caused by arc released gas and the composition of the arc released gas.

* * * * *