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King, Jr. et al.

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[54] **METHOD FOR CLEANING A CRYSTAL MICROBALANCE**

[75] Inventors: **William H. King, Jr.**, Florham Park; **Gideon M. Varga, Jr.**, Clark, both of N.J.

[73] Assignee: **Exxon Research and Engineering Company**, Linden, N.J.

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[52] **U.S. Cl.** **134/1**; 29/25.35

[51] **Int. Cl.**[2] **B08B 7/02**; H04R 17/10

[58] **Field of Search** 73/67.8; 134/1; 29/25.35

[56] **References Cited**

UNITED STATES PATENTS

2,178,225	10/1939	Diehl et al.	29/25.35
2,846,874	8/1958	Horn	73/67.8
2,851,876	9/1958	Arnold	73/67.8 X
3,653,253	4/1972	Olin	73/28

Primary Examiner—Barry S. Richman
Assistant Examiner—Bradley R. Garris
Attorney, Agent, or Firm—Harold N. Wells; F. Donald Paris

[57] **ABSTRACT**

A method of removing solid particles from the active surface of piezoelectric crystal microbalances by driving the crystal into a multiplicity of vibrational modes. When used to measure mass on its surface, the crystal vibrates in the plane of the crystal surface. But, by applying a current substantially greater than used in making mass measurements and scanning across a range of frequencies spanning the natural clean resonant frequency of the crystal, a multiplicity of resonances are excited and substantially all of the solid particles are removed from the crystal surface.

7 Claims, 4 Drawing Figures

Fig.1.

TRANSISTOR OSCILLATOR CIRCUIT

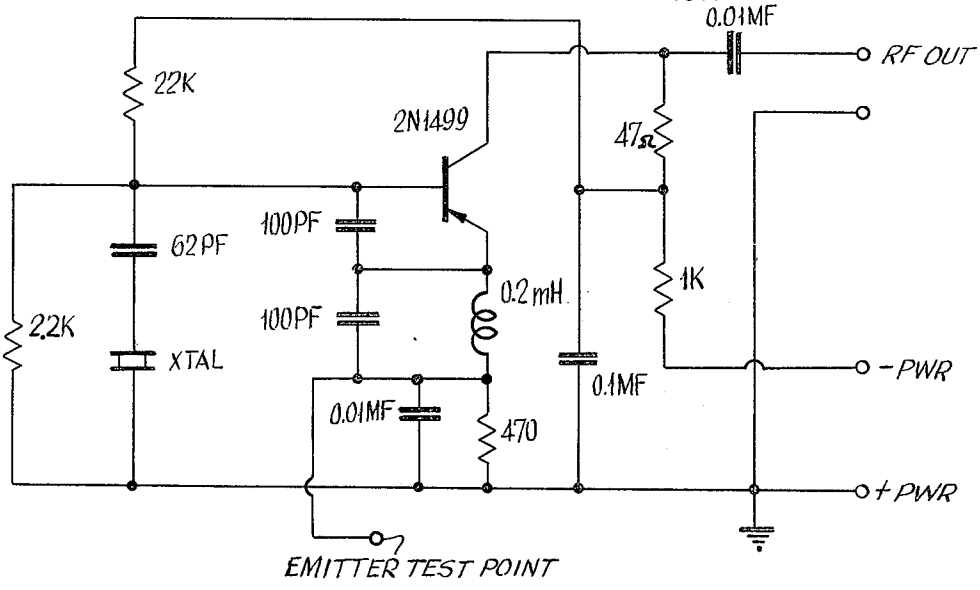


Fig.2.

CRYSTAL IMPEDANCE METER

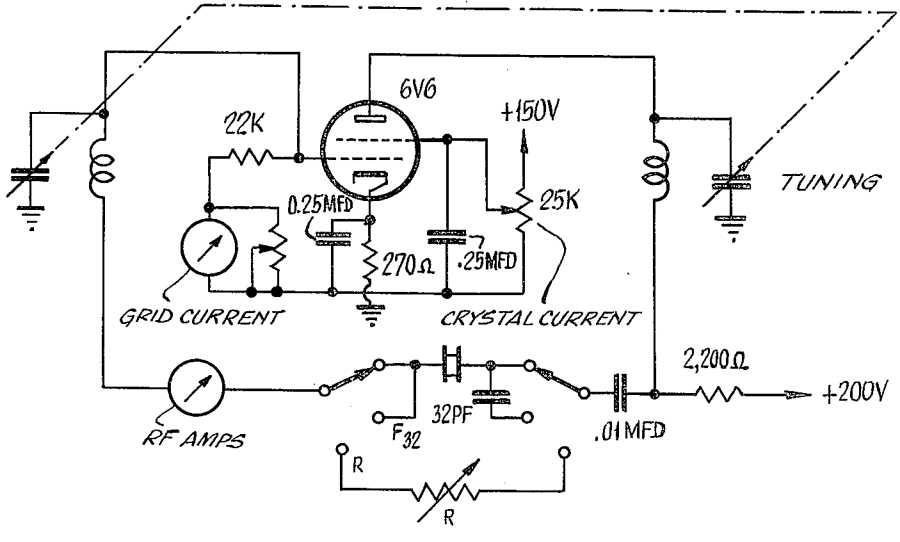


Fig. 3.

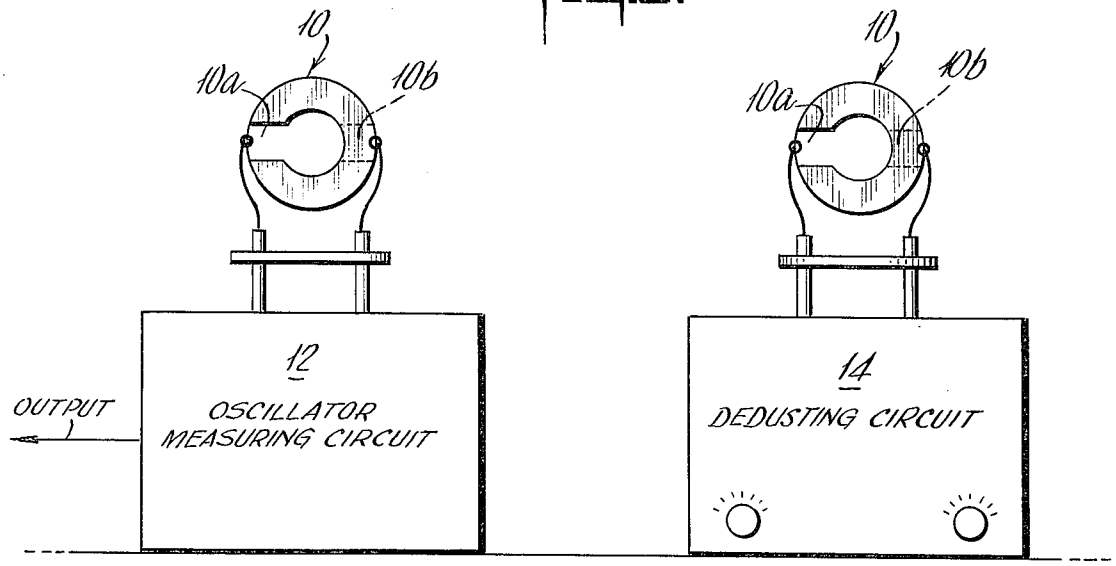
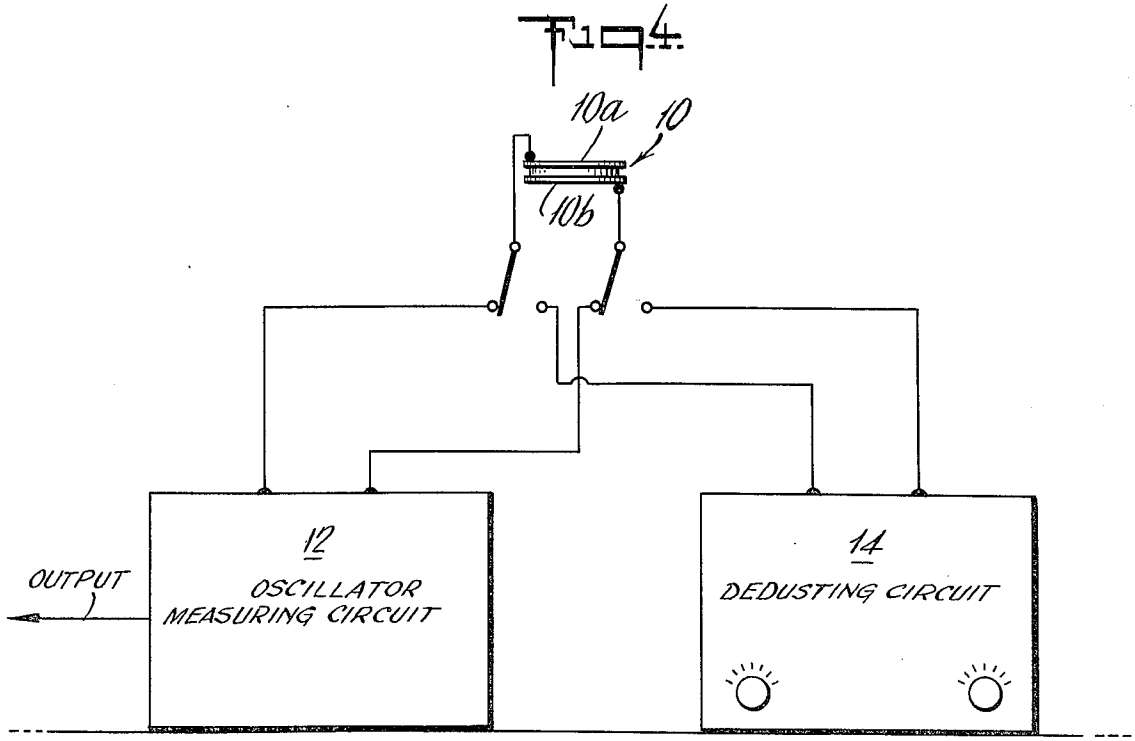

10
10a
10b
12
OSCILLATOR
MEASURING CIRCUIT
OUTPUT
10
10a
10b
14
DEDUSTING CIRCUIT

Fig. 4.


10a
10
10b
12
OSCILLATOR
MEASURING CIRCUIT
OUTPUT
14
DEDUSTING CIRCUIT

METHOD FOR CLEANING A CRYSTAL MICROBALANCE

The invention described herein was made in the performance of work under NASA contract number NAS1-9000/RCO-446067 and is subject to the provisions of Section 305 of the National Aeronautics and Space Act of 1958 (72 Stat. 435; 42 USC 2457).

BACKGROUND OF THE INVENTION

Piezoelectric crystals have been used as microbalances for measuring very small quantities of materials. In U.S. Pat. No. 3,164,004, the use of such crystals is generally discussed, the preferred method being to place a substrate on the crystal which can absorb from a gas stream the material to be measured. The absorbed material increases the effective mass of the crystal, changing its resonant frequency in proportion to the amount absorbed, thus making possible using the crystal as a weighing device.

The crystals have also been used for measuring solid particles, with and without a substrate which causes the particles to adhere to its surface. Disclosure of such applications is given in U.S. Pat. Nos. 3,653,253; 3,561,253; and 3,715,911. When measuring material absorbed from the gas phase, it is possible to desorb the material from the substrate, leaving it clean and ready to receive a new sample. However, measurement of solid particles presents a different cleanup problem. The crystals are insensitive to particles lying loosely on the crystal surface. The vibration is in the direction of the plane of the crystal in the typical AT-cut crystals, which are commonly used for this application. Thus, the solid particles must be securely attached to the crystal surface if they are to increase the crystal mass and so be measured. An adhesive is sometimes used to attach the particles or electrostatic forces may be developed to accomplish the same result without an adhesive. Once the solid particles have adhered, however, it is necessary to remove them if another measurement is to be made with the same crystal. This may be done in a number of ways, as is noted in U.S. Pat. No. 3,653,253 (column 6, lines 4 through 13).

A similar problem arises when the crystal is to be used with an absorptive substrate for measurement of moisture in air. Measurements made in a very dusty atmosphere could be hindered by dust attached to the crystal. The crystal could measure the weight of the dust itself instead of the moisture absorbed on the substrate. Also, moisture absorbed by the dust can be added to that absorbed by the substrate.

Assuring a clean crystal in dusty atmospheres or removing solid particles attached to crystals represents a significant problem, especially in situations where the instrument is to be operated automatically without the attention of an operator over significant periods of time. The present invention has overcome these difficulties and makes possible essentially complete cleaning of piezoelectric crystals which have become intentionally or inadvertently affected by the presence of dust particles.

SUMMARY OF THE INVENTION

While a normal piezoelectric crystal (AT-cut) oscillates parallel to the plane of its surface when using a crystal as a microbalance, it has been found possible to remove dust particles attached to the surface by forcing

such crystals into many different vibrational modes, that is, contrary to the normal use as a microbalance. With sufficient amplitude, such vibrations are sufficient to remove solid particles from the crystal surface without additional assistance. In order to fully clean it, a crystal is forced to vibrate at a series of frequencies spanning its natural resonant frequency and at a relatively high power compared to that at which the crystal vibrates during its measuring mode. When driven at relatively high power, the crystal can be made to oscillate in many vibrational modes. By scanning a range of frequencies, beginning below and ranging to above the principal natural resonant frequency, it has been found possible to excite many of the crystal's minor resonances, the net effect of which is to clear all of the dust particles from the surface without any further physical removal by blowing, sweeping, or other such prior art means. In the preferred embodiment, the scanning process is repeated in a plurality of cleaning steps until the crystal is fully restored to its original clean condition.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 illustrates a typical transistorized oscillator circuit used when the crystal is operated as a microbalance.

FIG. 2 illustrates a crystal impedance meter circuit which may be used to force the crystal to oscillate in its cleaning mode.

FIG. 3 illustrates schematically an embodiment of the invention wherein a single crystal is transferred between a frequency measuring oscillator circuit and the de-dusting circuit.

FIG. 4 shows an alternative embodiment in which the crystal is switched from one circuit to another.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

A piezoelectrical crystal used as a microbalance will normally be operated in an oscillator circuit designed for this purpose. A typical transistorized Pierce oscillator circuit is illustrated in FIG. 1. Other similar oscillators may be used to make such measurements, but the details of such circuits are not considered to be part of the present invention. The function of any such oscillator circuit is generally to apply a voltage to the piezoelectric crystal which causes it to oscillate and thereafter feeds back power into the crystal to maintain the vibration at its natural resonant frequency. This resonant frequency is measured and referred to that of a clean crystal, the difference in frequency being proportional the mass of the material deposited or absorbed on the crystal surface.

It must be appreciated that in this normal operation the amount of power which is applied to the crystal is relatively low and that the crystal is usually cut (AT-cut) so that it vibrates parallel to the plane of its surface. In such a vibration, particles attached to the crystal surface will have a significant effect on the crystal's resonant frequency. However, if the crystal moves independently of particles which are loosely resting on its surface it will fail to indicate their presence. When piezoelectric crystals are used to detect solid particles in atmospheric pollution testing this problem has been solved in two ways. First, adhesive materials are applied to the crystal surface to which the solid particles can be firmly attached; and second, electrostatic charges are generated on the dust particles opposite to

that of the crystal surface. These adhering particles must be removed if the crystal is to be used in repetitive measurements. Various methods may be used as suggested in U.S. Pat. No. 3,653,253, for example, blowing off the solid particles or washing with a cleaning fluid. Another possibility would be to brush away the solid particles. It is also suggested in that same reference that by driving the crystal at a very high current the particles may be dislodged from the surface and then may be blown away by a jet of clean air. It has been found, however, that such semi-manual cleaning is not necessarily required, that by proper imposition of preselected frequecies on the crystal, spanning the range of its principal natural resonant frequency, the essentially complete cleaning can be achieved.

Removal of solid particles from the crystal surface may also be a problem where the crystal is not being used for measuring solid particles but for measuring the absorption of moisture on a special substrate applied to the crystal. In such a situation, the solid particles will interfere with the normal function of the crystal and they must be removed if the crystal is to properly measure the absorbed gas. As has been mentioned, if the dust is tightly bound to the crystal, it will substantially affect its resonant frequency, but if loosely attached to the crystal, the dust may have little or no effect. However, where substantial amounts of dust are present, even without significant attachment of the dust, a mechanical damping effect may cause a shift in the resonant frequency, which could lead to erroneous results. Particularly where the crystal microbalance is to be used unattended in automatic routine operation, it is essential to assure that the crystal is free of any such interfering solid particles. Accordingly, the present invention provides a means by which the crystal may be clean prior to making the measurement to assure that any such interference of solid particles has a negligible effect upon the result. It has been found that if the dust loading is high enough, that it is possible to so completely damp the crystal oscillation that the normal Pierce oscillator circuit of FIG. **1** is unable to force the crystal into resonant vibration. According to the present invention, however, the crystal may be forced into vibration at other than its principal resonant frequency and at relatively high power in order to excite resonances normal to the crystal surface, loosening and effectively removing solid particles.

It has been recognized in the prior art that the surface of a crystal will exhibit various patterns of vibration, depending upon the frequency at which it is excited. In fact, one mode of discovering the patterns of vibration has been to apply a fine dust to the surface of the crystal and then to observe the rearrangement of the dust on the crystal which depends upon the mode of vibration. When interacting with the normal oscillator circuit, an AT-cut piezoelectric crystal vibrates parallel to the surface of the crystal. It has been discovered, however, that by applying a significant amount of power to the crystal at other than its normal resonant frequency, that many minor resonances can be excited. It is this abnormal mode of operation which causes solid particles to be dislodged. If one applied power at only a single frequency, the dust particles would tend to try to arrange themselves so as to collect on the nodal points and to leave the portions of the crystal which are vibrating with a maximum amplitude in much the same way as experimental determinations of the mode of crystal vibration were made. However, it has been found that by beginning at a frequency significantly below the natural resonant frequency of the crystal, scanning over a range of frequencies extending through and beyond the natural resonant frequency and applying sufficient power, it is possible to excite a large number of resonances so that during the scanning procedure all of the crystal surface is subjected to a substantial vibration, not only dislodging the particles, but removing them entirely. This is true even when the crystal is in a horizontal position where it might be expected that the dust particles would simply be dislodged and then fall back onto the crystal again.

In one embodiment of the invention, a crystal impedance meter similar to that shown schematically in FIG. **2**, was used to carry out the method of the invention. In a typical AT-cut crystal used as a microbalance and having a fundamental resonance of about 9 megacycles, a range of frequencies ranging from about 4 kilocycles below the resonant frequency to 6 kilocycles above the resonant frequency was scanned at a rate of about 1 kilocycle per second. A low voltage was applied to the crystal, selected to avoid excessive currents when resonant frequencies were reached and thus avoiding damage to the crystal. The power applied during the frequency scanning varied depending upon the resistance of the crystal, which was continually changing as dust was being removed. The power dissipated also depended upon whether a natural resonance was being excited. It was found that maximum power had to be limited in order to avoid physical damage to the crystals and, accordingly, the power was generally limited with a 9 megacycle crystal to about one-half watt maximum input. This proved to be sufficient power input to completely remove the dust when the scanning of the frequency range was repeated 5 to 10 times and required no other assistance. A test of the crystal's ability to measure absorbed moisture in its clean condition and after having been loaded with dust and then cleaned according to the invention indicated that the response of the crystal was substantially the same after cleaning as before. Thus, the cleaning method of the invention was effective in removing dust and permitted repeatable measurements, which is an essential feature of such a cleaning technique.

FIG. **3** illustrates schematically one mode of applying the invention. The crystals **10** are typically available in a plug-in format and can be inserted into the Pierce oscillator circuit **12** or other oscillator circuit for making measurements of mass deposited on the surface, acquired through absorption of a gas on a substrate or by deposition of solid particles. After the measurements have been made and cleaning is necessary, it would be possible to unplug the crystal and to place it into a de-dusting circuit **14** which would provide the scanning of frequencies at relatively high power compared to the Pierce oscillator circuit **12**, which simply seeks out and maintains the natural vibrating frequency. The crystal impedance meter illustrated in FIG. **2** could be used as the de-dusting circuit into which the dirty crystal is plugged.

In remote use where self-cleaning feature of the crystal would be important, the arrangement of FIG. **4** would be preferred, wherein the crystal **10** is simply switched from the measuring circuit **12** to the cleaning circuit **14**, depending upon the mode in which the crystal is to be operated. In FIG. **4** the crystal **10** is viewed 90° from that shown in FIG. **3**, illustrating that a typical crystal **10** is a flat circular disc onto which electrodes

10*a* and *b* have been secured, one on either side of the crystal. It will be appreciated that a dust particle lying on the surface of such a crystal **10** might have little effect when the crystal is vibrating parallel to the plane of the crystal, but such a particle securely attached to the crystal could vibrate with it, affecting its mass and its resonant frequency. In the present invention the crystal is forced to vibrate in many vibrational modes, not just in the plane of the crystal, so that any particle which rests or is secured to its surface may be dislodged by the vibrations. Such a use of the crystal is completely contrary, not only to its normal use in measuring mass, but to the general conception of the operation of such crystals, which are carefully cut in order to obtain vibration parallel to the surface. Since it is necessary when dislodging solid particles to force the crystal into a mode of operation which substantially departs from the normal method of operating such crystals, the present invention represents a major departure from teachings of the prior art.

The invention has application in situations where crystals are used for measuring of solid particles and must be cleaned prior to a subsequent measurement, but it also may be applied where the interference of dust particles must be avoided. Various electrical circuits may be used in order to provide for both measurement or cleaning according to the invention, without departing from the essence of it. The foregoing description of the preferred embodiments is for illustration of the method only and should not limit the scope of the invention which is defined by the claims which follow.

What is claimed is:

1. A method for removing particles from the surface of a piezoelectric crystal used in its measuring mode as a microbalance, comprising the steps of:

a. applying a variable electrical AC voltage to said crystal, thereby generating variable frequency signals in a frequency range extending from a lower frequency below the natural clean resonant frequency of said crystal to an upper frequency above said natural clean resonant frequency of said crystal, whereby said crystal vibrates in different vibrational modes corresponding to said frequency range spanning said natural clean resonant frequency; and

b. vibrating said crystal as in step (a) at a relatively high power compared to the power at which said crystal vibrates in its measuring mode, whereby said particles are dislodged from and vibrated off of said crystal and thereby said crystal is substantially cleaned of any particles present on the surface thereof.

2. The method of claim **1** including the step of repeating the process of vibrating said crystal in substantially all of its vibrational modes until said crystal is fully restored to its original clean condition, whereby said crystal vibrates in a plurality of directions causing removal of said particles from the surface thereof and wherein the excitation of said crystal is in a direction normal to said crystal surface.

3. The method of claim **2** wherein steps (a) and (b) are repeated from 5–10 times.

4. The method of claim **1** wherein said frequency range is from 4 kc below said natural clean resonant frequency to 6 kc above said natural clean resonant frequency.

5. The method of claim **1** including the step of limiting the power input to said crystal to about 0.5 watts and wherein said crystal has a natural clean resonant frequency of 9 megacycles.

6. The method of claim **1** including the step of scanning said frequency range at a rate of 1 kc/second.

7. The method of claim **1** including the step of transferring said crystal from its modes required for removal of said particles from said surface to its measuring mode.

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