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METHOD FOR TREATING FILAMENTS OF
IONIZATION GAUGES

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This invention is concerned with ionization gauges provided with a filament employed as an electron source. The invention is particularly applicable to the mass spectrometer (which is a type of ionization gauge) and provides means for eliminating gas-sensitivity in filaments employed in spectrometers and other gauges as electron emission elements.

In vacuum tube techniques it is customary to treat metallic filaments, say tungsten filaments, with hydrogen or other reducing gases prior to using the filaments as an electron source. Thus it is customary to heat the filament in an atmosphere of hydrogen to a cherry red prior to evacuation of the vacuum tube containing the filament. This procedure, although satisfactory from many standpoints, causes the filament to become gas-sensitive in the sense that the introduction of gas into the region of the filament brings about (1) a variation in the intensity of the electron emission of the filament (2) a variation in the density of the electron emission, i. e. in the cross section of an emitted electron beam. Both of these phenomena are objectionable in ionization gauges and particularly in mass spectrometers, because each introduction of gas changes the calibration of the instrument.

As a result of our investigations, we have discovered that metallic filaments and particularly tungsten filaments can be rendered free of gas-sensitivity by treating them with a liquid hydrocarbon prior to evacuation and heating. Preferably the hydrocarbon is an aromatic compound. For example, a pure tungsten filament can be treated with a liquid paraffin hydrocarbon or with benzene (C_6H_6) or other liquid ring compound prior to being placed in the envelope of a mass spectrometer. If the filament is then subjected to heating to a red heat after evacuation to a low pressure, say 5×10^{-7} mm. of mercury, it will be free from gas-sensitivity under most conditions.

Our invention will be more thoroughly understood in the light of the following detailed description of the presently preferred practice:

In accordance with this practice, the filament is thoroughly washed in a liquid hydrocarbon, say in benzene. Subsequently it is placed in the envelope of the spectrometer and the envelope is then heated and simultaneously evacuated, the heating serving to drive occluded substances from the wall of the envelope and from the metal and other parts of the spectrometer enclosed in the envelope. When the pressure in the envelope has been reduced to a low value, say 5×10^{-6} mm. Hg,

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heating of the filament itself is begun. The filament is heated slowly over a two hour period until a temperature of 2200° Kelvin is attained.

Under the foregoing conditions, the heating of the filament takes place in the presence of several gases. Thus carbon dioxide, carbon monoxide, water vapor, benzene and perhaps other hydrocarbons are released from glass and metal surfaces within the envelope. In the presence of the oxides of carbon and the hydrocarbon vapors, the filament becomes free from gas-sensitivity and accordingly more useful in mass spectrometry. It may be that the treatment with the gaseous oxides of carbon and hydrocarbon vapors causes a formation of a stable layer of some compound of the metal (say tungsten) with carbon and oxygen on the surface of the filament and that this layer acts as an armor to prevent carbonization of the filament and development of tungsten carbide at depth therein, and that in some manner this prevents gas sensitivity by preventing absorption of gases on the filament. Whatever be the explanation, the fact remains that filaments treated as described above are free from gas-sensitivity.

We have found that leakage of air into the envelope of a mass spectrometer over long periods will cause filaments to attain gas-sensitivity even though they are treated as described hereinbefore. Thus the presence of air within a spectrometer at a pressure of 10^{-6} mm. of mercury for a period of 48 hours may cause the filament to become gas-sensitive. If, however, this filament has been subjected to pre-treatment with a hydrocarbon in accordance with the invention, this gas-sensitivity is merely temporary and may be cured by eliminating the air within the envelope and introducing, say a C_3 , C_4 or C_5 hydrocarbon i. e. a hydrocarbon having as its nucleus either 3, 4 or 5 carbon atoms for a considerable period, say 24 hours.

It is desirable, in order to preserve the freedom from gas-sensitivity attained through the treatment of the invention, to maintain a concentration of a C_3 , C_4 or C_5 hydrocarbon in the spectrometer during idle periods. Thus if the spectrometer is not in use over a week end, the presence of n-butane within the envelope at a pressure of say 10^{-6} mm. of mercury tends to preserve the calibration of the instrument. This treatment with C_3 , C_4 or C_5 hydrocarbons will cure gas-sensitivity of a filament temporarily, even though the filament has not been subjected to the pre-treatment described hereinbefore,

Without the pre-treatment, however, the cure is only temporary.

We claim:

1. In the treatment of metallic filaments adapted for service as electron emitters in ionization gauges, the improvement which comprises treating the filament with a liquid hydrocarbon and subsequently heating it in an evacuated envelope in the presence of a minute amount of the hydrocarbon adhering to the filament as a result of the prior treatment, thereby reducing gas sensitivity of the filament.

2. In the treatment of tungsten filaments adapted for service as electron emitters in ionization gauges, the improvement which comprises treating the filament with a liquid hydrocarbon and subsequently heating it in an evacuated envelope in the presence of a minute amount of the hydrocarbon adhering to the filament as a result of the prior treatment, thereby reducing gas sensitivity of the filament.

3. In the treatment of metallic filaments adapted for service as electron emitters in ionization gauges, the improvement which comprises treating the filament with a liquid aromatic hydrocarbon and subsequently heating it in an evacuated envelope in the presence of a minute amount of the hydrocarbon adhering to the filament as a result of the prior treatment, thereby reducing gas sensitivity of the filament.

4. In the treatment of metallic filaments

adapted for service as electron emitters in ionization gauges, the improvement which comprises treating the filament with a liquid hydrocarbon and subsequently heating it to redness in an evacuated envelope in the presence of a minute proportion of an oxide of carbon plus the hydrocarbon adhering to the filament from the prior treatment, thereby reducing gas sensitivity of the filament.

5. In the treatment of metallic filaments adapted for service as electron emitters in ionization gauges, the improvement which comprises treating the filament with benzene and subsequently heating it in an evacuated envelope in the presence of a minute proportion of the benzene adhering to the filament from the prior treatment, thereby reducing gas sensitivity of the filament.

6. In the treatment of metallic filaments adapted for service as electron emitters in ionization gauges, the improvement which comprises treating the filament with a liquid hydrocarbon and subsequently heating it in an evacuated envelope in the presence of no more than that minute proportion of the hydrocarbon which adheres to the filament in the prior treatment, the pressure in the envelope being below 5×10^{-6} mm. Hg during the heating, thereby reducing gas sensitivity of the filament.

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