[54]	MICROWAY	E DEVICES	
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[52]	U.S. Cl	333/73 R, 333/73 S, 333/ 333/83 R, 333	
		01p 3/08, H01p 7/06, H03 .333/84, 83 R, 84 M, 73 S,	h 7/08
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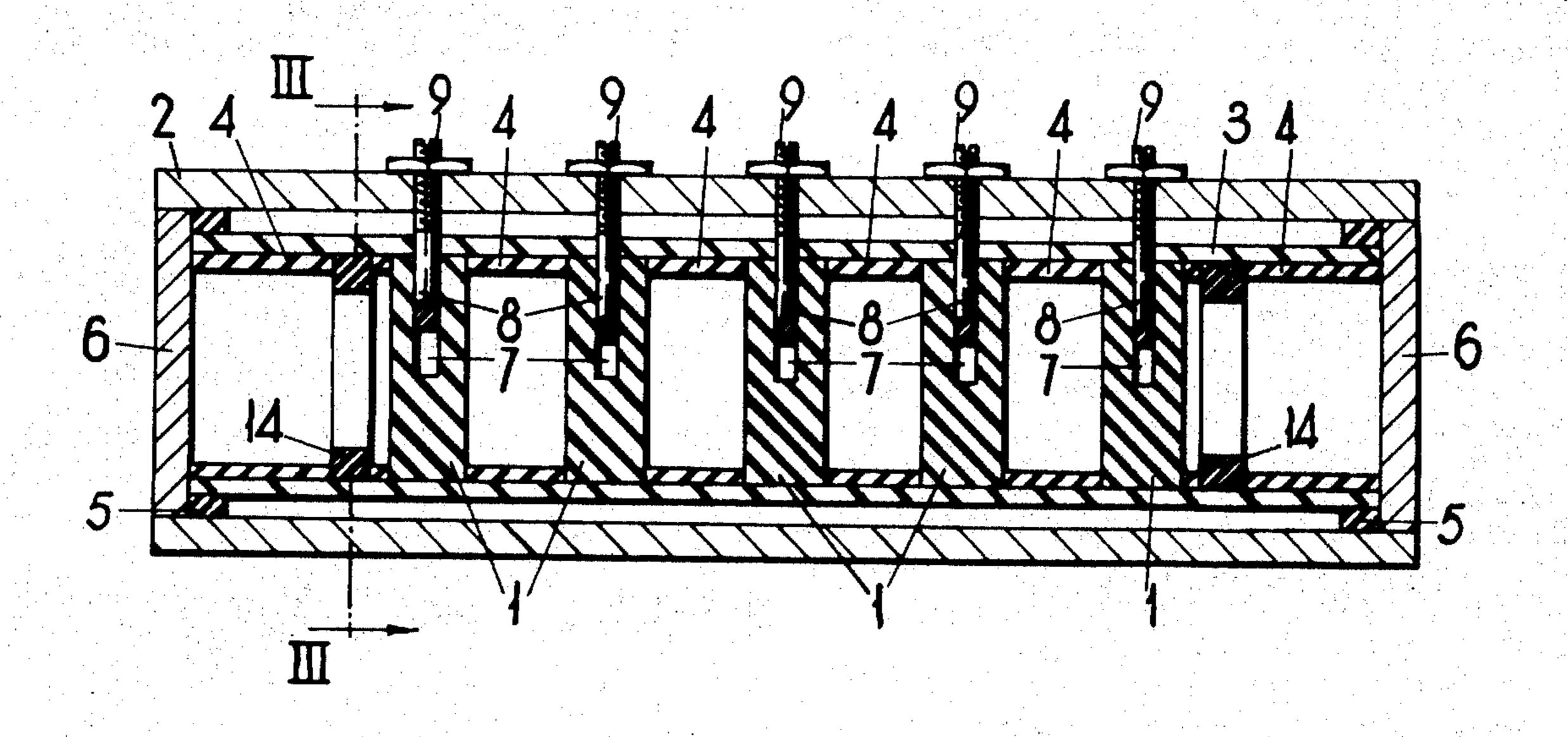
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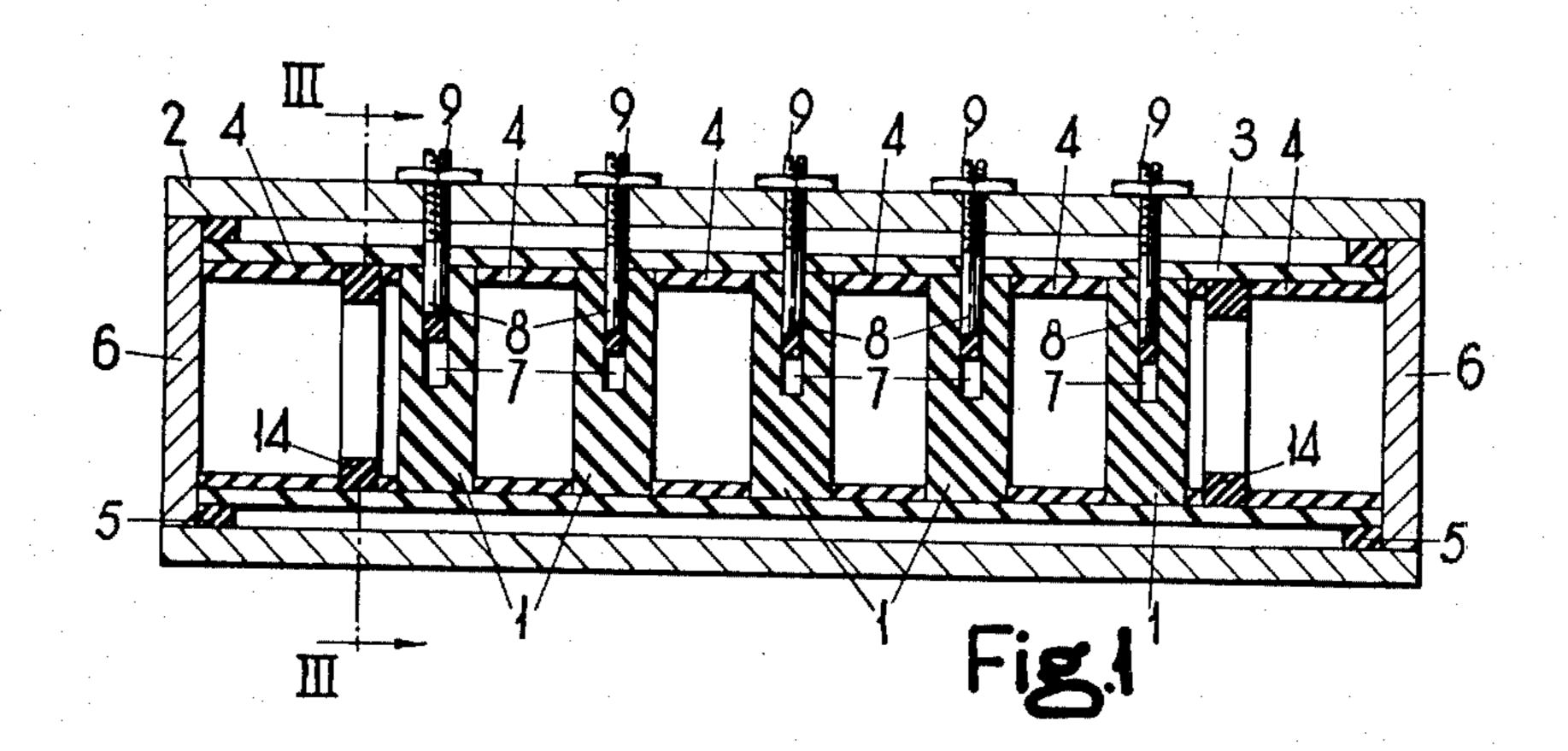
[57] ABSTRACT

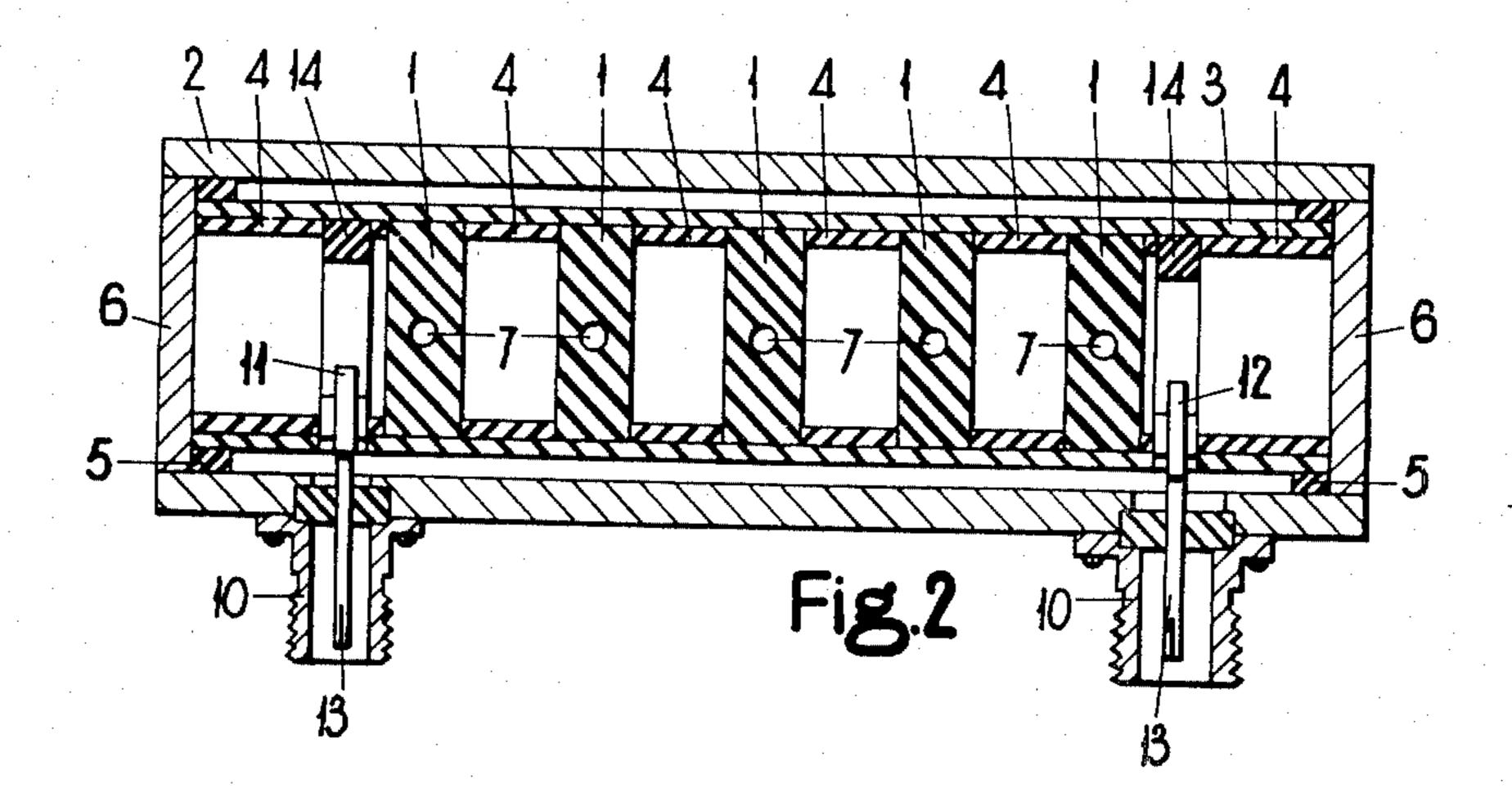
In a microwave device incorporating a component formed of dielectric material, and so designed that the response of the device is dependent on the permittivity of the said material, the component is formed of a ceramic material consisting of one or more alkaline earth metal zirconates, or zirconates and titanates, together with niobium pentoxide and/or tantalum pentoxide, the composition of the material being such that the atomic ratio of zirconium to titanium is not less than 80: 20, that the total proportion of niobium pentoxide/tantalum pentoxide is in the range of 0.1 to 3.0 mole per cent of the total amount of the zirconate/titanate constituent, that it does not contain more than 10 mole per cent of barium titanate, and that the material will have, at microwave frequencies, permittivities in the range of 25 to 75, a substantially constant temperature coefficient of permittivity, which is preferably within the range from +50 to -100p.p.m. per degree Centigrade, and a loss tangent not exceeding 0.001 at 20°C. The dielectric materials are advantageous for use, for example, as resonators for microwave bandpass filters, and as substrates for microwave integrated circuits. In the cases of some of the materials, the inclusion of niobium pentoxide/tantalum pentoxide reduces the microwave losses, as compared with similar materials without such additions.

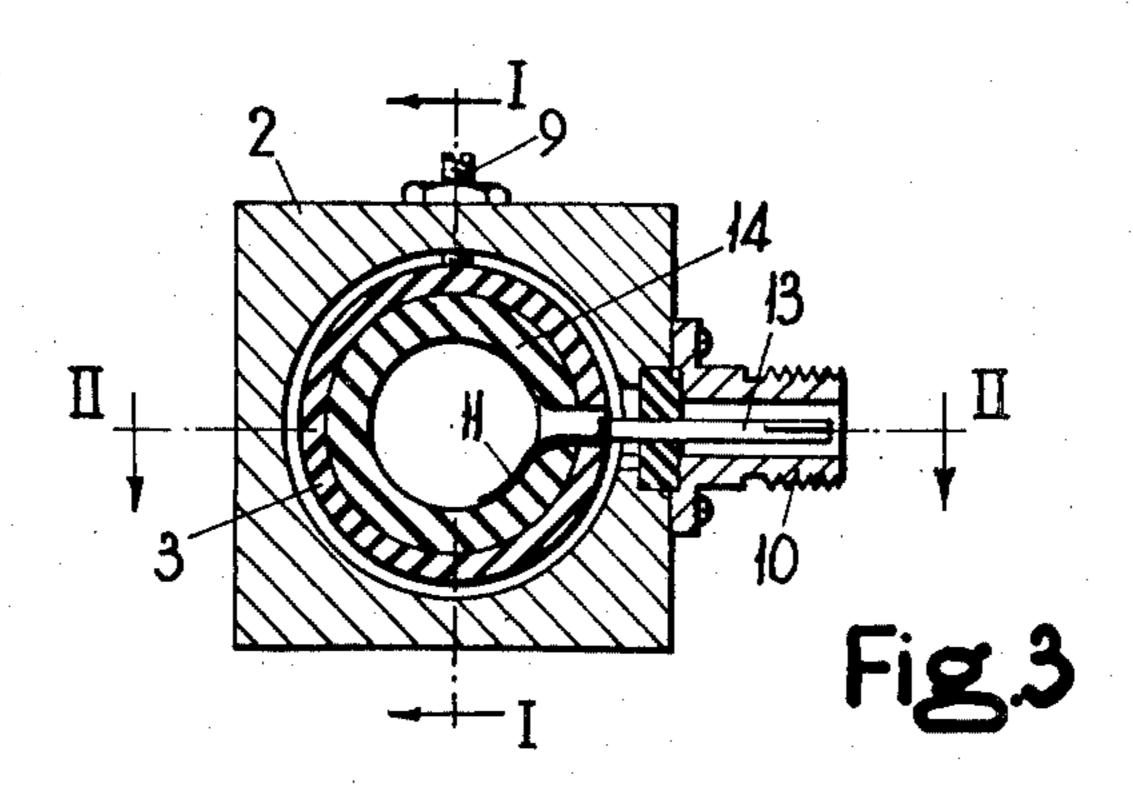
4 Claims, 5 Drawing Figures



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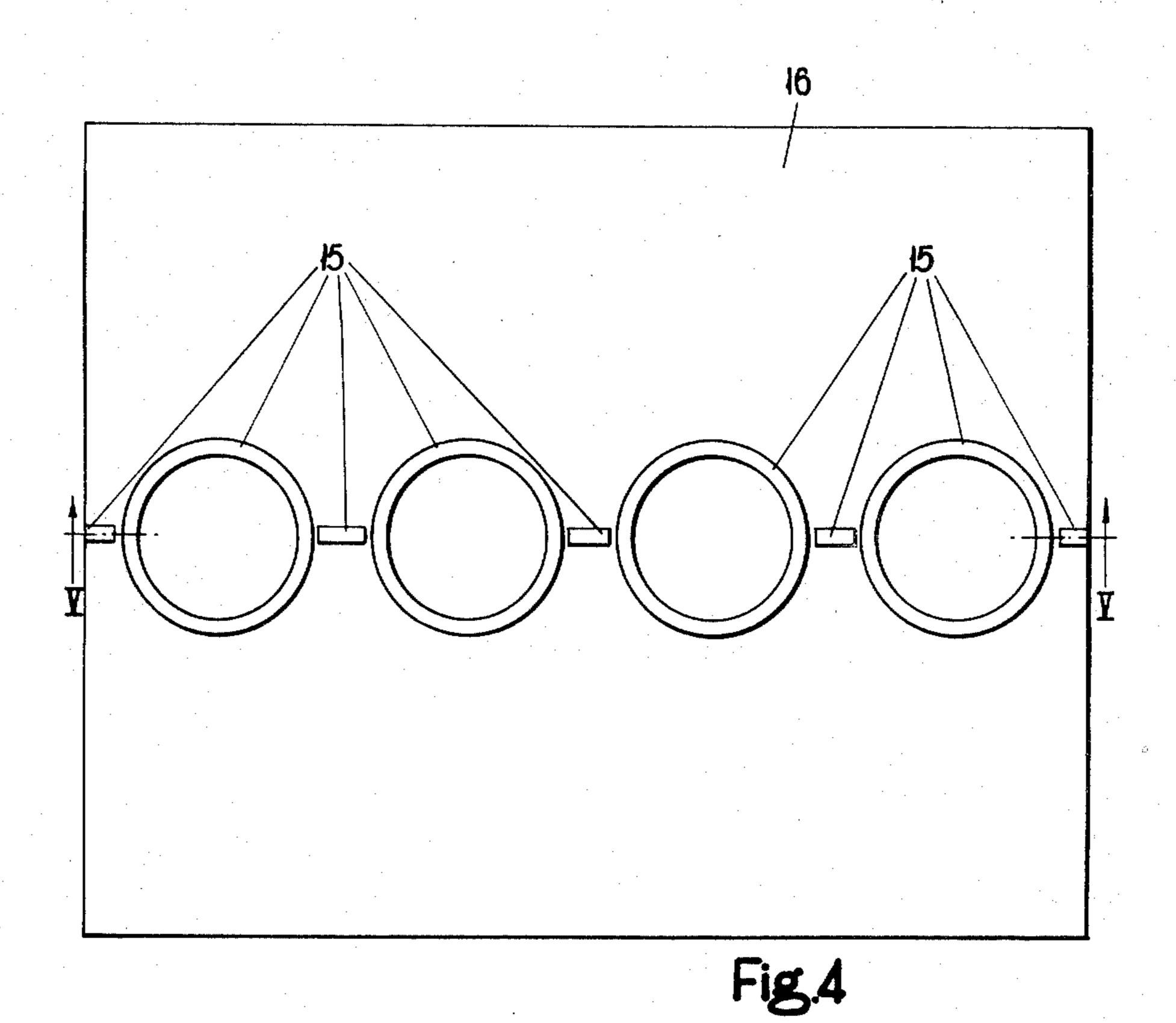


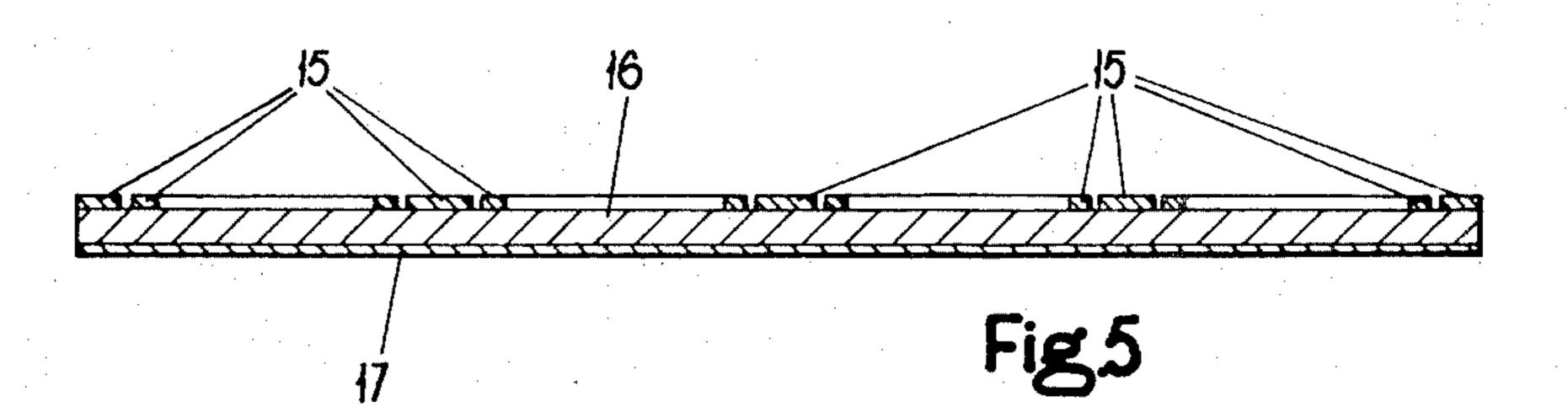
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MICROWAVE DEVICES

This invention relates to electrical devices of the kind designed for operation at microwave frequencies, that is to say frequencies in the range of 400 MHz to 30 5 GHz, for use for example in telecommunications equipment, and incorporating components formed of dielectric materials, wherein the response of the device is dependent upon the permittivity of the dielectric material.

United States Pat. Application Ser. No. 64,301, filed in the names of Robert Christopher Kell, David Forbes Rendle and Eric Edward Riches on Aug. 17, 1970, for Improvements in or relating to Microwave Devices, and assigned to the Assignee of the present application, relates to microwave devices of the aforesaid kind, in which the dielectric component is formed of a ceramic dielectric material consisting of at least one compound of the general formula ABO₃, where A is a metal of the group consisting of barium, strontium and calcium and B is a metal of the group consisting of zirconium and titanium, the composition of the material being so chosen that the atomic ratio of zirconium to titanium is in the range of 80: 20 to 100: 0, that it does not include significant amounts of both barium and titanium and that the material will have, at microwave frequencies, permittivities in the range of 25 to 75, a substantially constant temperature coefficient of permittivity, and a loss tangent not exceeding 0.005 at 20° C.

Microwave devices of the kind referred to in the aforesaid application include, for example, a microwave bandpass filter incorporating a resonator of dielectric material as specified, in replacement for the metal waveguide resonator incorporated in a conventional microwave filter, and an integrated microwave circuit in which the dielectric material is used to form the substrate carrying the conducting strips constituting the circuit elements.

Suitable dielectric materials for use in such devices, 40 in accordance with the aforesaid specification, include calcium zirconate, and combinations of barium zirconate and strontium zirconate, barium zirconate and calcium zirconate, strontium titanate and strontium zirconate, and calcium titanate and calcium zirconate. 45 Whilst all the dielectric materials referred to above have loss tangents, at microwave frequencies, not exceeding 0.005, and in some cases the loss tangents are less than 0.001, at 20°C, it is desirable that, for the applications referred to, the microwave losses should be as low as possible, and some of the materials are less advantageous than others in this respect. For example, the barium strontium zirconates have microwave losses in excess of 0.001: one particular barium strontium zirconate containing barium and strontium in the atomic ratio of 56: 44, which is especially advantageous for some microwave applications since its temperature coefficient of permittivity is near zero, would be even more suitable for use in these applications if its microwave loss tangent were reduced to a value below 0.001.

I have now found that the microwave losses of some of these materials, especially the barium strontium zirconates and to a lesser extent the strontium titanate-zirconates, can be markedly reduced, with little or no modification of the permittivity or of the temperature coefficient of permittivity, by the incorporation of a

small proportion of niobium pentoxide or tantalum pentoxide in the materials.

Thus according to the present invention, in a microwave device incorporating a component formed of dielectric material, and so designed that the response of the device is dependent upon the permittivity of the said material, the said component is formed of a ceramic dielectric material consisting of at least one compound of the general formula ABO₃, where A is a metal of the group consisting of barium, strontium and calcium and B is a metal of the group consisting of titanium and zirconium, together with at least one oxide of the group consisting of niobium pentoxide and tantalum pentoxide, the composition of the material being so chosen that the atomic ratio of zirconium to titanium is in the range of 80: 20 to 100:0, that the total proportion of niobium pentoxide and tantalum pentoxide is in the range of 0.1 to 3.0 mole per cent of the total amount of the compounds ABO₃, that the material does not include significant amounts of both barium and titanium and that it will have, at microwave frequencies, permittivities in the range of 25 to 75, a substantially constant temperature coefficient of permittivity, and a loss tangent not exceeding 0.001 at 20°C.

As explained in the specification of Application No. 64,301, if the dielectric material contains a relatively large proportion of either barium or titanium, there should not be a sufficient amount of the other one of these elements present to make it possible for barium titanate to be formed in a proportion which will cause the specified limits of permittivity and loss tangent of the material as a whole to be exceeded, and in particular the proportions of barium and titanium present should be such that barium titanate does not constitute more than 10 mole per cent of the material.

As also explained in the aforesaid application, the compound or compounds ABO₃ may be so chosen, and where two or more of such compounds are present the relative proportions of the compounds may be so adjusted, that the dielectric material as a whole has a temperature coefficient of permittivity of a desired positive or negative value, a valve within the range of +50 to -100 p.p.m. per degree Centigrade usually being preferred and in some cases the composition of the material being balanced to give a value of, or near, zero.

The dielectric material may consist of a single compound of the type ABO₃ having the requisite properties, such as calcium zirconate, or of a mixture or solid solution of two or more of such compounds, together with a proportion of niobium pentoxide and/or tantalum pentoxide in the range specified. Since a material consisting of a single phase solid solution is more readily reproducible than a material consisting of a mixture of compounds, where two ABO₃ compounds are present it is in general preferred to employ combinations of compounds which form such a single phase, that is to say to use combinations of barium-barium, strontium-strontium, barium-strontium, or calcium-calcium compounds. Particularly preferred combinations of ABO₃ compounds are barium strontium zirconates in which the atomic ratio of barium to strontium is in the range of 40:60 to 80:20, especially 56:44, calcium titanate-zirconates in which the atomic ratio of titanium to zirconium is in the range of 0:100 to 5:95,

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and strontium titanate-zirconates in which the atomic ratio of titanium to zirconium is in the range of 2:98 to 8:92. The calcium-containing materials tend to have increased losses and variable temperature coefficients of permittivity in the presence of moisture: it may 5 therefore be necessary to ensure that moisture is excluded from these materials during use.

One example of a device in accordance with the invention is a microwave bandpass filter incorporating one or more dielectric resonators in the form of bars, 10 cylinders or discs of dielectric material as specified above: resonators of this kind are advantageous in comparison with the conventional metal waveguide resonators, since the use of a dielectric enables the size of the 15 resonator to be reduced. In use, a ceramic dielectric resonator is usually placed within a metal screen, which results in a slight increase in the resonant frequency of the dielectric element. Another type of device in which the aforesaid dielectric materials can be employed with 20 advantage is an integrated microwave circuit, the dielectric material being used to form the substrate carrying the circuit elements. The dielectric materials employed in accordance with the invention are advantageous in this connection, as compared with high 25 density alumina which has hitherto been proposed for this application, since they have higher permittivities, and lower temperature coefficients of permittivity, than those of alumina.

The dielectric materials for use in the devices of the invention can be prepared by techniques conventionally employed for the production of ceramic dielectric materials of this type, that is to say by preparing an intimate mixture of suitable powdered starting materials in the required relative proportions, pressing the mixture, and heating the pressed compacts to effect reaction and sintering. If desired the materials can be prepared from mixtures of the requisite pre-formed compounds of the formula ABO₃, together with niobium pentoxide and/or tantalum pentoxide, but preferably the ABO₃ compounds are prepared from starting mixtures comprising the constituent oxides and/or compounds, such as carbonates or hydroxides, which decompose on heating to give the oxides.

The niobium pentoxide and/or tantalum pentoxide may be initially introduced into the dielectric material either in the free state or in the form of an alkaline earth metal niobate or tantalate of the general formula MR₂O₆ or M₂R₂O₇, where M is barium, strontium or 50 calcium and R is niobium or tantalum. In some cases, when the niobium and/or tantalum is initially introduced as the free oxide, it might be possible, or desirable, to reduce the content of zirconium or titanium in the dielectric material by an amount atomically equivalent to the amount of niobium and/or tantalum introduced, the niobium/tantalum thus replacing part of the zirconium/titanium in the dielectric composition.

A preferred procedure for preparing the dielectric components for use in devices in accordance with the invention, by which ceramic bodies of density approaching the theoretical density, and hence having optimum permittivity, can be obtained, includes the steps of isostatically pressing the powdered starting mixture to form compacts of simple shapes, such as rods, prefiring at a sufficiently high temperature to effect partial sintering so as to form coherent bodies, then crushing

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the prefired compacts to powder, die-pressing the powder to form compacts of the desired shapes of the components to be produced, and firing these compacts at a temperature higher than that employed for the prefiring step to convert them into dense, sintered, ceramic bodies, the niobium pentoxide and/or tantalum pentoxide, in the free state or in the form of compounds as aforesaid being either included in the initial starting mixture or added to a powdered prefired material consisting only of the desired compound or compounds ABO₃, prior to the die-pressing and sintering steps.

The niobium pentoxide/tantalum pentoxide is thus incorporated in the dielectric material composition before or during the final sintering process, and appears to go into solid solution in the ABO₃ material.

The preparation and properties of some dielectric components for use in devices in accordance with the invention, together with the preparation and properties of components of similar materials without niobium pentoxide or tantalum pentoxide, for comparison, will now be described in the following specific examples.

EXAMPLE 1.

For the preparation of a disc (A) of barium strontium zirconate of composition Ba_{0.56}Sr_{0.44}ZrO₃, powdered barium carbonate, strontium carbonate and zirconium dioxide were mixed in the required relative proportions and the powder mixture was milled with water in a porcelain ball mill for 36 hours, then dried and compacted into rods under hydrostatic pressure of 7 tons per square inch, and the rods were prefired in air at 1250°C for 2 hours. The prefired rods were crushed in a disc mill, and the resulting powder was wet milled in a ball mill for 24 hours. The powder was then dried, mixed with a solution of 2 wt. percent camphor in ether, and die-pressed under a pressure of 9 tons per square inch, to form a disc, which was finally sintered by firing in air at 1450°C for 2 hours.

Two further discs (B, C) were prepared in the manner described above, with the addition of powdered niobium pentoxide, in amounts, respectively, of 0.25 and 1.0 mole per cent of the barium strontium zirconate, to the prefired powder before the die-pressing and sintering steps.

EXAMPLE 2.

A disc (A) of calcium zirconate was prepared by the method described in Example 1, using calcium carbonate and zirconium dioxide powders as starting materials. Further discs (B, C) were prepared in the same manner with the addition of, respectively, 0.25 and 1.0 mole per cent of niobium pentoxide to the prefired powder.

EXAMPLE 3.

A disc (a) of strontium zirconate-titanate of composition SrZr_{0.955}Ti_{0.045}O₃ was prepared by the method described in Example 1, using a powdered starting mixture of strontium carbonate, zirconium dioxide and titanium dioxide. Two additional discs (B, C) containing respectively 0.25 and 1.0 mole percent of niobium pentoxide were prepared in the same manner with the addition of the niobium pentoxide to the prefired powder.

Some of the properties of the materials prepared as described in the above Examples are given in the following Table. The properties which have been determined are the permittivity, loss tangent, and tempera-

frequency being determined for such discs 20 mm in diameter and 4 mm thick, resonated in the TE_{011} mode in a closely fitting waveguide reflection cavity cut-off in the air regions.

TABLE										
·		Added	Properties at frequency 1.6 kHz.			Properties at frequency 5 gHz.				
Example	Composition (ABO ₃ compound or compounds)	Nb_2O_5 , $mol.$ percent	10 ⁵ ×TCC per ° C. ±15 error	Permit- tivity at 20° C.	104×loss tangent at 100° C.	10°×TCF per ° C.	tivity at t	10 ⁴ ×loss angent it 20° C.		
1(A) 1(B) 1(C) 2(A) 2(B) 2(C) 3(A) 3(B) 3(B) 3(C)	Ba _{0.56} Sr _{0.44} ZrO ₃ Ba _{0.56} Sr _{0.44} ZrO ₃ Ba _{0.56} Sr _{0.44} ZrO ₃ CaZrO ₃ CaZrO ₃ CaZrO ₃ SrZr _{0.055} Ti _{0.045} O ₃ SrZr _{0.955} Ti _{0.045} O ₃ SrZr _{0.955} Ti _{0.045} O ₃	$\begin{array}{c} 0 \\ 0.25 \\ 1.0 \\ 0 \\ 0.25 \\ 1.0 \\ 0 \\ 0.25 \\ 1.0 \end{array}$	$\begin{array}{c} 0 \\ -11 \\ 0 \\ +33 \\ +32 \\ +91 \\ 0 \\ 0 \\ +20 \end{array}$	38. 1 30. 6 30. 5 32. 0 31. 1 30. 9 36. 1 37. 2 36. 1	11 <4 <0 72 247 <3 <3 <3	-17.6 -14.4 -23.7 -17.3 -15.7 -21.1 -14.4 -23.7	34. 7 31. 3 32. 3 28. 0 27. 2 27. 1 33. 4 33. 4 33. 3	18 4.7 4.4 7.6 5.5 6.3 7.0 6.2 4.9		

ture coefficient of capacitance (TCC) at audio frequency (1.6 kHz), and the permittivity, loss tangent, and temperature coefficient of resonant frequency (TCF) at microwave frequency (5 GHz). Audio frequency measurements were carried out, as well as microwave frequency measurements, because knowledge of the audio frequency properties of a material is of value in giving an indication of the properties the material will possess at microwave frequencies, and audio frequency measurements are more easily made.

The temperature coefficients of permittivity of the materials were not determined directly, but can readily be deduced from the temperature coefficient of capacitance, or from the temperature coefficient of resonant frequency of a microwave cavity containing a disc of the material, which properties are more conveniently measured at audio frequency and microwave 35 frequency respectively. Thus the temperature coefficient of permittivity is derived from the temperature coefficient of capacitance by subtracting from the latter the coefficient of thermal expansion of the material, which for these ceramic materials in only 8 - 40 10 × 10⁻⁶/°C, or is derived from the temperature coefficient of resonant frequency by solving the resonator equations as given by S. B. Cohn and K. C. Kelly in an article published by the Institute of Electrical and Electronics Engineers, in the Transactions on Microwave 45 Theory and Techniques, Volume 14 (1966), page 406. In practice, the important temperature coefficient for microwave applications is that of the resonant frequency (which can be measured) rather than that of the permittivity (which must be calculated). The resonant 50 frequency is related to $E^{-1/2}$, where E is the permittivity, and the temperature coefficient of resonant frequency is related to -1/2 times the temperature coefficient of permittivity. It is therefore expected that if the temperature coefficient of permittivity is small, that of resonant frequency will also be small, and if the temperature coefficient of permittivity is large, that of resonant frequency will be large and of opposite sign.

For carrying out the measurements of the properties referred to, at audio frequency, the major faces of the sintered discs of the materials, prepared as described above, were lapped to produce flat parallel surfaces and silver paste was applied to these surfaces, dried at 120°C for 12 hours and fired at 650°C for one hour. The measurements of microwave properties were carried out on non-metallized discs at a frequency close to 5 GHz, the temperature coefficients of resonant

The above Table shows that the incorporation of niobium pentoxide in the barium strontium zirconate material, while only slightly reducing the loss tangent at audio frequency, effects a considerable reduction in the loss tangent at microwave frequency, at the same time slightly reducing the permittivity but not having a marked effect on the temperature coefficients of capacitance and resonant frequency. However, the addition of niobium pentoxide appears to result in only a slight reduction of the loss tangent, at microwave frequency, in the cases of strontium zirconate-titanates and calcium zirconate, which materials have lower microwave losses than barium strontium zirconates in the absence of niobium pentoxide additions.

The dielectric components in accordance with the invention, prepared as described in the above Examples, and listed in the Table, are suitable for use as resonators for filter elements. Suitably shaped plates of the same materials, of thickness about 1 mm, can also be used as substrates for integrated microwave circuits to be operated at frequencies of 1 to 5 GHz.

It will be appreciated that a device in accordance with the invention may incorporate more than one dielectric component as specified. For example, a microwave filter may comprise a number of dielectric resonators distributed along the axis of a waveguide used below its cut-off frequency.

Two specific microwave devices in accordance with the invention are shown in the accompanying drawings and will now be described by way of example. In the drawings, in which like parts in the different figures are indicated by the same reference numerals,

FIG. 1 shows, in sectional elevation, a bandpass filter incorporating five dielectric resonators;

FIG. 2 is a sectional plan view of the filter shown in FIG. 1;

FIG. 3 is a transverse section of the filter shown in FIGS. 1 and 2, drawn on the line III—III of FIG. 1;

FIG. 4 is a plan view of a microstripline circuit on a dielectric substrate; and

FIG. 5 is a section drawn on the line V—V of FIG. 4. Referring to FIGS. 1, 2 and 3 of the drawings, the relationship between which is indicated by the lines I—I and II—II of FIG. 3 and III—III on FIG. 1, the device shown is a narrow band, high Q, filter designed to operate at a frequency of 4 GHz, comprising five resonator discs 1 formed of a dielectric material of a composition as specified in accordance with the invention, suitably one of the Nb₂O₅ — containing materials

listed in the foregoing Table, each disc having a diameter of 20 mm, a thickness of 4 mm, and being adapted to resonate in the TE₀₁₁ mode. The resonator discs are supported in a copper outer casing 2, suitably 14 cm long and 3.5 cm square in cross-section, by means of a tube 3, cylindrical spacers 4 and rings 5, all formed of a low loss, low permittivity dielectric material, for example the material sold under the Registered Trade Mark "Rexolite" the tube 3 being closed at both ends by copper caps 6. The resonator discs 1 have central holes 7 into which are inserted rods 8 of the same dielectric material as the discs themselves, and tuning screws 9 are inserted through the casing 2 to bear upon the rods 8 for adjusting the position of the rods in the holes 7, in $_{15}$ order to adjust the resonant frequency of the discs as required.

As shown in FIGS. 2 and 3, two 50 ohm Type-N connectors 10 are attached to the casing 2, one at each end of the resonator disc assembly; copper coupling strips 20 11, 12, for signal input and output respectively, are soldered to the center pins 13 of the connectors, which pass through apertures in the casing 2, and the copper strips are supported within the filter cavity by rings 14 of the same dielectric material as the members 3, 4 and 25, referred to above.

FIGS. 4 and 5 of the drawings show a filter circuit in 50 ohm microstripline, 15, carried on a substrate 16 in the form of a rectangular plate of a dielectric material of a composition as specified in accordance with the in- 30 vention. The substrate may be, for example, 15 mm long, 12.5 mm wide and 0.8 mm thick and, as shown in FIG. 5, has a continuous metal coating 17 on the face opposite to that on which the stripline circuit 15 is carried. Both the circuit 15 and the coating 17 suitably 35 consist of a layer of chromium covered with a layer of gold: these layers are formed on both sides of the dielectric plate by evaporating first chromium and then gold on to the faces of the plate and finally increasing the gold layer to the desired thickness by electroplat- 40 ing; part of the coating is then removed from one face of the plate by photo-etching, to leave the desired circuit 15.

I claim:

- 1. A microwave bandpass filter comprising in com- 45 bination
 - a. input means,
 - b. output means, and
 - c. coupling means for coupling the input microwave signal energy to the output means,
 - d. said coupling means comprising at least one resonator in the form of a body of dielectric material arranged to be subjected to the microwave signal energy so that the response of the bandpass filter depends on the permittivity of 55

the dielectric,

- e. the said resonator body being formed of a ceramic dielectric material consisting of
 - i. at least one compound of the general formula ABO₃, wherein
 - A. A is a metal of the group consisting of barium, strontium and calcium and
 - B. B is a metal of the group consisting of zirconium and titanium,
 - ii. together with at least one oxide of the group consisting of niobium pentoxide and tantalum pentoxide,
 - iii. the composition of the material being so chosen A that the atomic ratio of zirconium to titanium is in the range of 80:20 to 100:0,
 - B. that the total proportion of niobium pentoxide and tantalum pentoxide is in the range of 0.1 to 3.0 mole per cent of the total amount of the compounds ABO₃,
 - C. that if both barium and titanium are present the proportions thereof are such that barium titanate does not constitute more than 10 mole per cent of the material, and
 - D. that the material will have at frequencies in the range of 400 MHz to 30 GHz,
 - I. permittivities in the range of 25 to 75,
 - II. a temperature coefficient of permittivity which is substantially constant with changes in temperature, and
 - III. a loss tangent not exceeding 0.001 at 20°C, and
- f. wherein the said resonator body has a hole formed therein, and
- g. there is provided a rod slidable in said hole and tuning means coupled to said rod to adjust the position of said rod in said hole whereby to vary the resonant frequency of said body.
- 2. A microwave bandpass filter according to claim 1, wherein the said compound ABO₃ constituent of the dielectric material forming the said resonator body consists of a barium strontium zirconate in which the atomic ratio of barium to strontium is in the range of 40: 60 to 80: 20.
- 3. A microwave bandpass filter according to claim 1, wherein the said rod slidable in the hole of the resonator body is composed of the same ceramic dielectric material as the resonator body itself.
- 4. A microwave bandpass filter according to claim 1, which includes a housing of low permittivity dielectric material and wherein said resonator body of said ceramic dielectric material is in the form of a disc, said disc being disposed within said housing, and said input means and said output means being disposed on said housing on opposite sides of said disc.

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