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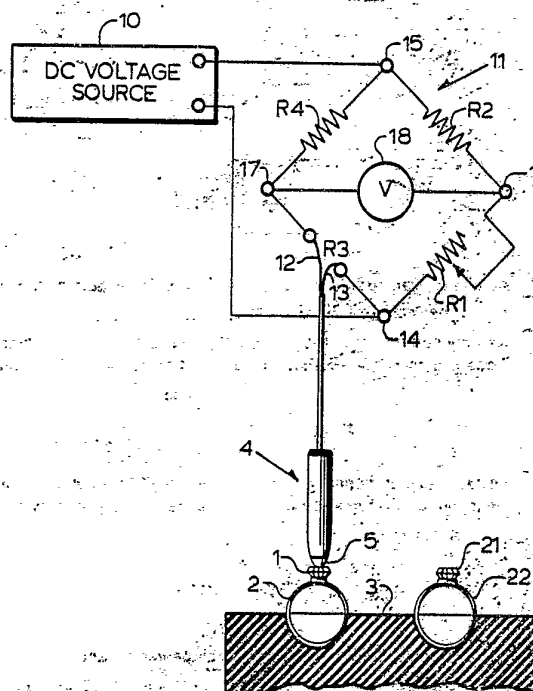
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(54) Title: DISTINGUISHING MATERIAL BY THERMAL CONDUCTIVITY

(57) Abstract

A simulated diamond (1) such as crystalline cubic zirconia, which has optical properties very similar to natural diamond and so is difficult to distinguish optically from natural diamond, is distinguished from natural diamond (21) by measuring its thermal conductivity which is significantly different from the thermal conductivity of natural diamond, by measuring the temperature of a heated probe (4) held against the simulated diamond as an indication of the simulated diamond thermal conductivity. In a preferred embodiment of the present invention, a controlled amount of heat energy is generated at the probe and thereafter, while the probe is held against the simulated diamond, the temperature of the probe is detected as a measure of relative thermal conductivity of the simulated diamond to the natural diamond.



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-1-

METHOD AND APPARATUS FOR DISTINGUISHING
MATERIAL BY THERMAL CONDUCTIVITY

Description

Technical Field

- 5 This invention relates to methods and means of distinguishing material and in particular for distinguishing simulated diamonds made of cubic zirconia from natural diamonds.

Background Art

- 10 Diamond-like gems produced from material other than carbon have found a significant commercial market. Some of these materials such as cubic zirconia, have optical properties sufficiently similar to natural diamonds that experienced
- 15 jewelers have difficulty in distinguishing the gem from natural diamond without removing the gem from its mounting to measure hardness and/or density, or, with the gem in its mounting, making relatively complex x-ray tests that sometimes take
- 20 several hours to perform. Furthermore, the hardness test requires scratching or otherwise marring the gem and this is destructive. It is most commonly performed by removing the gem from its mount and scratching the bottom. For example, crystalline
- 25 cubic zirconia can be cut and faceted so that it has the appearance of a natural diamond and when inspected optically even an experienced jeweler cannot easily distinguish it from natural diamond;



-2-

and so, without removing the gem from its mounting and/or making complex x-ray tests, the jeweler cannot tell within a few minutes time whether the cubic zirconia gem is or is not a natural diamond. This situation can be used by unscrupulous persons who would attempt to pass off an imitation diamond gem, such as a cubic zirconia gem, as a natural diamond.

The x-ray tests mentioned above involves making x-ray pictures of the gem. From these x-ray pictures, some experienced jewelers can distinguish whether the gem is or is not a natural diamond. While this technique is well known and quite reliable, it does take considerable time and skill to perform and most jewelers are not equipped with the x-ray equipment required for the test. It is the principle object of the present invention to provide a method and means of testing such diamond-like gems or imitation diamond gems by which the gem can be distinguished from natural diamond without removing the gem from its mount and in a relatively shorter period of time than by the techniques used heretofor.

Disclosure of the Invention

It is a general object of the present invention to provide an improved method and means for distinguishing imitation diamond gems from natural diamond.

It is another object to provide a method and means for distinguishing one material from another by virtue of the difference in thermal conductivity of the one material relative to the other.



-3-

It is another object to provide a method and means, other than optical, mechanical or x-ray for distinguishing a diamond-like gem or a simulated diamond from a natural diamond.

5 It is another object to provide a method and means for distinguishing diamond-like gems or simulated diamond gems from natural diamonds quickly and without removing the gem from its mount.

10 It is another object to provide a method and means for reliably distinguishing an imitation gem, such as cubic zirconia, from natural diamond within a few seconds time.

It is another object to provide relatively
15 simple apparatus at relatively low cost for accomplishing any of the above enumerated objects.

In accordance with the present invention the thermal conductivity of a test material, and particularly of a gem, is determined by subject-
20 ing the gem to a steady heat flow and then measuring the temperature of the gem. The thermal conductivity may be determined as a function of the change in steady state temperature of a test probe resulting from a change in heat flow from the probe.

25 In accordance with the present invention in another of its aspects, a simulated diamond gem such as cubic zirconia is identified or distinguished from natural diamond by measuring the thermal conductivity of the gem relative to a reference.

30 In as much as the thermal conductivity of natural diamond is at least an order of magnitude greater than any of the imitation diamond materials cur-



-4-

rently used, this physical parameter, thermal conductivity, is a useful and reliable criterion. In all embodiments of the present invention, the thermal conductivity of the simulated diamond gem is represented by the change in temperature of a probe in thermal contact with the gem immediately after applying a predetermined amount of heat to the probe. For this purpose, a thermoresistance device such as a thermistor is placed in intimate thermal contact with the gem after, or at the same time electric current of prescribed magnitude and duration is applied to the thermistor, heating the thermistor to an elevated temperature, and, thereafter, measuring the reduced temperature of the thermistor due to conduction of heat from it by the gem as an indication of the thermal conductivity of the gem. Thus, the temperature of the thermistor, being indicative of the thermal conductivity of the gem, is also an indication of whether or not the gem is a natural diamond. For example, within a certain range it can be concluded that the gem is not a natural diamond.

In a preferred embodiment of the present invention, a second thermistor is provided, very similar to the first one, and it is held in intimate thermal contact with reference material having a known thermal conductivity (the reference material may be a natural diamond) and both of the thermistors are in electrical circuit with a resistance bridge as resistance legs of the bridge. When the bridge is electrically balanced by varying the resistances of other legs of the bridge, the values of those



-5-

other resistances can be used in a simple calculation to make a comparison of the thermal conductivity of the gem to the thermal conductivity of the reference material. This comparison can reveal
5 with a high degree of certainty that an imitation diamond of, for example, cubic zirconia, is not a natural diamond.

Other objects and features of the present invention will become apparent from the following
10 specific description of embodiments of the invention which, at the present time, represent the best known uses of the invention.

Brief Description of the Drawings

15 Figure 1 is a mechanical-electrical diagram illustrating structure including a resistance bridge circuit for carrying out the generic process of the present invention, using a heat generating probe in thermal contact with the gem and
20 measuring the temperature of the probe as it is reduced by heat conduction through the gem;

Figure 2 is an enlarged cross section view of a suitable thermoresistance temperature probe including a thermistor that can be used in the
25 apparatus of Figure 1, showing details of construction of the probe;

Figure 3 is an electrical-mechanical diagram of apparatus similar to the apparatus shown in Figure 1 and including two substantially similar
30 heat generating probes, one in contact with the imitation diamond gem and the other in contact with a reference material that may be a natural diamond; and



-6-

Figure 4 is a representative plot of the resistance bridge unbalance current versus the probe heating current pulse duration using the apparatus in Fig. 3, when comparing natural diamond with glass and when comparing a cubic zirconia gem with another cubic zirconia gem.

Best Mode of Carrying Out the Invention

Two embodiments of the present invention are disclosed herein. Both embodiments incorporate the generic features of the invention and are represented by electrical-mechanical apparatus for detecting and measuring the temperature of a heated probe in thermal contact with a sample material, such as a diamond-like gem, as an indication of the relative thermal conductivity of the material. While the embodiments disclosed herein can have use and application for the general measurement of relative thermal conductivity of just about any material, it should be kept in mind that these embodiments are adapted particularly for distinguishing any of the current diamond-like gems and particularly those that cannot be distinguished by simple optical inspection (such as cubic zirconia) from natural diamond. When the invention is used for this particular purpose the advantages already discussed herein and above are gained most effectively. In order to stress this point, the embodiments are described herein as particularly adapted and used to distin-



-7-

guish cubic zirconia gems from natural diamond using the considerable difference in thermal conductivity of this gem from the thermal conductivity of natural diamond as the determining criteria.

5 Turning first to Figure 1 there is shown ready for testing a cubic zirconia gem 1 held by its usual mounting 2, such as a ring, which is fastened in just about any convenient manner to a holder 3. A temperature probe 4 having a thermoresistance
10 device 5 at the tip thereof is held placing the thermoresistance device which may be a thermistor against a convenient surface of the gem 1. Then, electric current is fed from a suitable source such as source 10 via a bridge circuit 11 and electrical
15 leads 12 and 13 to the thermistor, very quickly raising its temperature above the prevailing ambient temperature. The heating current is controlled so that the thermistor at the tip of the probe in thermal contact with the gem very quickly
20 reaches a steady state temperature. At this steady state temperature, a significant portion of the heat flow from the thermistor is conducted by the gem 1 to the mount 2 and the surrounding air. Clearly, that steady state temperature is determined
25 by many factors, one of which is the thermal conductivity of the gem. Hence, it can be concluded that the steady state temperature which is the temperature of the thermistor is indicative of the thermal conductivity of the gem and so a measure of
30 that temperature for many of the current imitation diamond gems can reveal that the gem is or is not a natural diamond.



-8-

Measurement of the thermistor temperature is accomplished using the resistance bridge 11. This bridge includes four resistance legs, one of which is the thermistor 5. The four legs are denoted R1, 5 R2, R3 and R4 and are connected as shown to define four terminals 14, 15, 16 and 17. The source 10 is connected across 14 and 15 and a voltmeter 18 is connected across terminals 16 and 17. R1 is a variable resistance, R3 is the thermistor 5 and 10 R2 and R4 are fixed equal resistances. This bridge is also known as a Wheatstone bridge.

In accordance with the preferred operation of this embodiment referred to, herein as the steady state measurement embodiment, the first step is 15 to apply a DC voltage from a source 10 across the terminals 14 and 15 of sufficient magnitude to quickly bring the end of the thermistor to a steady temperature, preferably above ambient temperature. This voltage from the source is referred to herein 20 as the heating voltage. Then, after a few seconds, to be sure the thermistor temperature is stabilized and while the voltage is applied, the bridge is balanced by varying R1 until the voltage across terminals 16 and 17 as read by the voltmeter 18 25 is zero. Hence, at this point, the test system is in electrical balance at steady state thermal conditions, heat dissipation from the probe being by radiation to the ambient surroundings and by ambient air convection. At this initial balanced 30 condition the value of R1 is read as R1s.

The next step is to place the probe thermistor in intimate thermal contact with the test gem 1 as



-9-

shown in Figure 1. The probe is preferably placed against a flat surface of the gem under a constant controlled pressure. Then, after a few seconds, without changing the heating voltage, heat flow
5 stabilizes at a steady state flow condition again. At this steady condition a significant portion of the steady flow of heat from the probe is by conduction through the gem 1, in addition to radiation to the ambient surroundings and by ambient air
10 connection. Clearly, heat flow from the probe has been increased by an amount related to the thermal conductivity of the gem. The result of all this is that by placing the heated probe in contact with the gem, the thermistor is cooled by
15 a number of degrees depending upon the thermal conductivity of the gem. At this steady state test condition, the bridge circuit is again balanced by varying R1 until the voltage across terminals 16 and 17 read on voltmeter 18 is again
20 zero and at this test balanced condition, the value of R1 is read as R1t.

When the bridge is in electrical balance, R1, R3 and R3 is indicative of the temperature of the probe thermistor. The change in the thermistor
25 temperature, ΔT , from the steady state initial balanced condition to the steady state test balanced condition is representative of the thermal conductivity of the gem. When the bridge is electrically balanced the resistance values of the legs of the
30 bridge are related as follows:

$$\frac{R_1}{R_2} = \frac{R_3}{R_4}$$



-10-

At the initial balanced condition the resistance values of the legs of the bridge are expressed as follows:

$$5 \quad \frac{R_{1s}}{R_2} = \frac{R_{3s}}{R_4} \quad \text{or} \quad R_{3s} = R_{1s} \frac{R_4}{R_2}$$

and at the test balanced condition, the resistance values of the legs of the bridge are expressed as follows:

$$10 \quad \frac{R_{1t}}{R_2} = \frac{R_{3t}}{R_4} \quad \text{or} \quad R_{3t} = R_{1t} \frac{R_4}{R_2}$$

and, since $\Delta T_{s-t} \propto R_{3t} - R_{3s}$, then

$$\Delta T_{s-t} \propto R_{1t} - R_{1s}$$

and it can be concluded that the thermal conductivity of the gem, K_g , is related as follows:

$$15 \quad K_g \propto R_{1t} - R_{1s}$$

A standard or reference reading of the instrument shown in Figure 1 can be made using a reference material that may be a natural diamond. For this purpose a natural diamond 21 may be provided in setting 22 which is preferably similar to the setting 2 of the gem and also held by holder 3. Since the purpose of the instrument is to distinguish the imitation diamond gem from natural diamond, the thermal conductivity, K_g , of the gem 1 and the thermal conductivity of the reference, K_r , (in this case, the natural diamond 2) are measured by this equipment under the same conditions and then compared. The table below shows the thermal conductivity of several imitation diamond or diamond-like gems and the thermal conductivity of natural diamond.



-11-

TABLE - THERMAL CONDUCTIVITY

K = watts/cm /°C

	<u>Gem</u>	<u>Temp, °Kelvin</u>	<u>K</u>
	Natural Diamond	196	8.7
5	"	273	6.6
	Al ₂ O ₃ Sapphire	373	0.030
	Al ₂ O ₃ Ceramic	273	0.35
	Pyrex	273	0.01
	MgO.Al ₂ O ₃ Spinel	373	0.13
10	SiO ₂ parallel to C axis	273	0.12
	SiO ₂ perpendicular to C axis	273	0.07
	ZrO ₂ Cubic	373	0.02
	3Y ₂ O ₃ 5Al ₂ O ₃ Yttrium aluminum garnet	273	0.11
15	TiO ₂ Rutile C Axis	310	0.09
	TiO ₂ Rutile A Axis	310	0.06
	ZrSiO ₄	310	0.04



-12-

Figure 2 is a cross section showing in detail the tip of the probe containing the thermoresistance device. In this example, the thermoresistive device is a thermistor. A thermistor is a resistance element made of semiconductor material which exhibits a high negative temperature coefficient of resistivity. It consists of a small bead of semiconductor material placed between two wire leads and is commonly used to measure temperature. The probe may consist of a steel tube enclosing a ceramic tube that contains the leads 12 and 13 from the thermistor 33 which is mounted at the tip of the tube encased in epoxy 34 and a tip of solder 35 completes the probe. The solder tip is held against the gem or reference material when making the thermal conductivity measurements.

The steady heating technique described above with reference to the first embodiment is most effective for detecting the thermal conductivity of an imitation diamond gem having a mass of 0.1 gram (0.5 carat) or more. For smaller gems, heat conduction from the thermistor to the probe and radiation to the surroundings so dominate heat flow from the thermistor, where the heat is generated, that the thermal conductivity of the gem is obscured and so the reading is less reliable. For smaller gems, even as small as .01 grams, if the heating energy is pulsed so that heat is applied for only a few seconds, a reliable reading of thermal conductivity can be obtained even on these very small gems. A pulsed heating system is illustrated in Figure 3 and consists of a DC voltage source that provides a relatively



-13-

high voltage for the heating pulse and a relatively much lower voltage to energize the bridge for making a measurement. In addition to the pulsed heating feature, the embodiment shown in Figure 3 also includes a reference leg in the bridge so that the thermal conductivity of the sample gem can be directly compared with the thermal conductivity of a reference. In view of the preferred use of the present invention, to distinguish diamond-like gems from natural diamond, the reference material can be a natural diamond.

The test system shown in Figure 3 includes a DC voltage source 40 providing two voltage levels the heating voltage at 20 volts from terminal 40A and the measuring voltage at 5 volts from terminal 40B, both with reference to the base voltage at terminal 40C. A timer switch 42 selects either the heating voltage or the measuring voltage and applies it to the resistance bridge 41. This bridge essentially is a Wheatstone bridge having four resistance legs denoted generally R_1 to R_4 . Resistance leg R_3 of this bridge is the thermistor 5 at the tip of test probe 4 that is held against the sample gem 1 that is under test. Resistance leg R_1 is derived from a second probe 44 that may be constructed exactly as test probe 4 and includes a thermistor 45 at the tip and two electrical leads 46 and 47 extending through the probe to the bridge.

The reference body 48 may be any suitable material having known thermal conductivity against which the thermal conductivity of the test gem is



-14-

to be compared. For example, 48 may be a natural diamond in a setting mounted to the same holder 3 as the test gem; or it may be a block of glass or copper of known thermal conductivity.

5 Legs R_2 and R_4 are each made up of a fixed impedance and a portion of a variable impedance that is variable to balance the bridge. For example, R_2 is made up of fixed impedance 52 and one side of variable impedance 53, while R_4 is
10 made up of fixed impedance 54 and the other side of variable impedance 53. The four legs of the bridge are connected together as shown defining bridge terminals 55 to 58 and voltage from the source is applied across terminals 55 and 56
15 while an ammeter 59 is connected between terminals 57 and 58.

Operation of this embodiment is versatile, because it has the added feature of a reference of known thermal conductivity for dynamic simultane-
20 eous comparison with the test gem. There are several sequences of operation of this embodiment by which the thermal conductivities of two materials can be compared as a basis for distinguishing one from the other. In any of these sequences, the
25 probes 4 and 44 are heated simultaneously by activating switch 42 and applying 20 volts across the bridge 41 while variable resistance 53 is set at its center position so that R_2 equals R_4 . As a result, equal pulses of current are fed through R_1
30 and R_3 causing equal amounts of heat energy to be delivered to the test and reference thermistors



-15-

5 and 45. This current pulse is a few seconds in duration as determined by the timer switch 42, whereupon it is turned off and immediately thereafter the measuring voltage, five volts, from the 5 supply is applied across the bridge. Immediately after that, variable impedance 53 is varied to produce a zero reading of the ammeter 59. At that point, the bridge is balanced and the position of the variable arm 53a of variable resistance 53 is indicative of the rate of heat dissipation or conduction from the test probe as compared to the reference probe.

The purpose of the reference and reference probe is to directly compare the test gem with 15 a known reference and the balanced resistance bridge is used very advantageously to do this. The bridge can be balanced before either probe is heated (except by the relatively small measuring current). Following that, one is assured that 20 each probe draws the same heating current. Ideally, if the two probes are in contact with materials having the same thermal conductivity and both exceed a minimum size, the bridge should remain balanced following the heating pulse. The 25 measuring voltage, five volts, is so small that it causes very little heating of the test and reference thermistors, and, since it produces substantially the same current in each test probe, when the bridge is balanced, its effects are balanced.

30 When the bridge is balanced, the resistance legs are related as follows:



-16-

$$\frac{R_3}{R_1} = \frac{R_4}{R_2}$$

When the resistances 52, 53 and 54 are each 1500 ohms as shown in the figure and the portion of variable resistance 53 that becomes part of R_4 is denoted X, then the above relationship between the legs of the bridge can be expressed as follows:

$$\frac{R_3}{R_1} = \frac{R_4}{R_2} = \frac{1500 + X}{3000 - X}$$

10 clearly this bridge circuit can be calibrated by a calibration plot of X vs R_3/R_1 and, it should be clear this ratio is the ratio of the temperature of the test thermistor 5 to the reference thermistor 45.

15 The duration of the heating pulse that yields a reliable result using the measuring system shown in Figure 2 will depend on the size of the test gem. A series of charts can be constructed for determining the necessary heating interval to realize a
20 reliable test result. For this purpose, a plot of ammeter 59 reading for the bridge unbalanced condition versus the heating interval will indicate how long that interval must be in order to achieve the maximum unbalanced and the magnitude of the
25 unbalance. Then, when the bridge is balanced, the value of X will be most indicative of the ratio of thermal conductivities of the test gem and the reference. This sort of calibration plot is illustrated in Figure 4 which is a plot of micro-
30 amperes read on ammeter 59 when the bridge is unbalanced ($2X=1500$ ohms), versus the heating interval

-17-

in seconds. This plot shows two curves, one for a 1-carat natural diamond as the test gem compared to a block of glass as the reference and another for a 1-carat cubic zirconia imitation diamond gem as the test gem as compared to another cubic zirconia gem as the reference. Clearly, these curves level off over fifty microamperes apart after a two second heating interval. Hence, the heating interval should be at least two seconds long, but need not be much longer than that in order to produce the maximum or most reliable indication of the ratio of test and reference thermal conductivities, and, particularly, the ratio of the thermal conductivity of a test cubic zirconia gem to the thermal conductivity of a natural diamond.

A useful sequence of operation of the pulsed embodiment illustrated by Figure 3 is as follows:

Step A: While both probes 4 and 44 are subject to the same exposure for example, exposed only to air, set switch 42 at the measuring voltage (terminal 40a) applying the measuring voltage across the bridge.

Step B: Balance the bridge by varying 53 until voltmeter 59 reads zero volts across terminals 57 and 58.

Step C: Place the probes 4 and 44 in thermal contact with the gem 1 and reference 48 respectively, each under the same constant force.

Step D: Switch 42 to terminal 40b applying heating voltage across the bridge and, after about two seconds of heating, switch 42 back to terminal 40a, applying measuring voltage across the bridge.



-18-

Step E: Within a few seconds after Step D, repeat Step B and record the value of 53b as X.

Having completed the above sequence, and
5 recorded the value X, the ratio of thermal conductivity of the test gem to the reference material is related as expressed by the equation from above.

$$\frac{\text{Thermal Conductivity Test Gem}}{\text{Thermal Conductivity Reference}} = \frac{R_3}{R_1} = \frac{1500+x}{3000-x}$$

10 Clearly, the above sequence provides a numerical ratio of the thermal conductivity of the test material to the known reference material. A useful variation of the above sequence is the following sequence:

Step A: (as above)
15 Step B: (as above)
Step D: (as above)
Step B: (as above)
Step C: (as above)
Step D: (as above)

20 Another sequence is as follows:

Step C: (as above)
Step F: Set switch 42 at terminal 40a applying
measuring voltage across the bridge
Step B: (as above)
25 Step D: (as above)
Step E: (as above)

Another sequence, particularly useful for quickly distinguishing an imitation diamond gem from natural diamond, provides as the reference, a mat-
30 erial that has substantially the same thermal con-



-19-

ductivity as natural diamond, and, of course, the reference can be a natural diamond. For this purpose the preferred sequence is as follows:

- 5 Step F: Set 53 so that $R_2 = R_4$. This insures
 that equal heating pulses are applied
 to the probe thermistors.
- Step C: (just as above)
- Step D: (just as above)
- 10 Step G: Within a few seconds after Step D,
 read the voltmeter and if the read-
 ing exceeds zero by a predetermined
 amount it is concluded that the gem
 is not a natural diamond.

Any of the above sequences can be carried out,
15 step by step by a human operator using the apparatus
shown in one or the other of the embodiments, and
so the apparatus is used to measure the thermal
conductivity of a material or to distinguish
between two materials by their difference in thermal
20 conductivity. The above sequences and other se-
quences derived from them, (or at least part of the
sequence) could be carried out automatically once
initiated by an operator and for that purpose, addi-
tional structure and circuits could be provided by
25 those skilled in the art to simplify the actions
carried out by the operator. For example, the
timer switch 42, once set at the heating voltage
position (terminal 40a) could be timed automati-
cally to dwell at that position the prescribed
30 interval (about two seconds), and then switch back
to the measuring voltage terminal 40b.



-20-

Further mechanization could include a feedback drive mechanism in place of voltmeter 59 for driving the variable resistance 53 to balance the bridge automatically.

5 The embodiments of the present invention described hereinabove incorporate the process and structure of the present invention. More particularly, the process is intrinsic in the structure, however, other structures and equipment could be
10 used to perform the process. The embodiments herein represent the best known uses of the invention at the present time, including use to distinguish imitation diamond gems from natural diamond, particularly those gems that cannot be readily
15 distinguished by eye. It should be understood that some changes in the process could be made and/or some changes in the apparatus could be made by one skilled in the art without departing from the spirit and scope of the present invention
20 set forth by the appended claims.

Industrial Applicability

This invention can be applied wherever the thermal conductivity of a material is to be determined. It has particular application in distinguish-
25 ing between natural and simulated gems.



-21-

CLAIMS

1. In a process of distinguishing a test material from another material, the following combination of steps:
 - 5 (a) placing a heated probe in intimate thermal contact with the test material and
 - (b) thereafter, measuring the temperature of the probe,
 - 10 (c) said temperature being indicative of the rate of conduction of heat from the probe by the test material.
2. A process as in Claim 1 wherein:
 - (a) the temperature of the probe is measured while heat is being applied thereto.
- 15 3. A process as in Claim 1, wherein:
 - (a) the temperature of the probe is measured after heat has been applied thereto and while substantially negligible heat is being applied thereto.
- 20 4. A process as in Claim 1, further including:
 - (a) the same combination of steps performed with another probe and the other material in the same sequence and at the same time as the combination of steps set forth by Claim 1,
 - 25 (b) the difference between said measured temperature of the test material and said measured temperature of the other material being indicative of the difference between the



-22-

thermal conductivity of the test material and the other material.

5. A process as in Claim 1 wherein:
- (a) the probe includes a thermoresistance device,
 - 5 (b) heat is applied to the probe by feeding an electric current to the device and
 - (c) the measure of temperature is the electrical resistance of the thermoresistance device.
6. A process as in Claim 4 wherein:
- 10 (a) each probe includes a thermoresistance device,
 - (b) heat is applied to both of the probes by feeding an electric current to the thermoresistance device in the probe and
 - 15 (c) said difference between said measured temperatures is represented by the difference between the resistances of said probes,
 - (d) whereby the difference between the resistances of said probes is representative of the difference between the thermal conductivity
 - 20 of the test material and the other material.
7. A process as in Claim 1, further including:
- (a) the same combination of steps performed with another probe and the other material in the
 - 25 same sequence and at the same time as the combination of steps set forth by Claim 1,
 - (b) the ratio of the measured temperature of the test material to the measured temperature of the other material being indicative



-23-

of the ratio of the thermal conductivity of the test material to the thermal conductivity of the other material.

8. A process as in Claim 5 wherein:

- 5 (a) the thermoresistance device is heated by electric current conducted by the device, the rate of heat so produced not exceeding the combined rate of conduction of heat from the probe by surroundings and the test materials, whereby the probe is at a temperature indicative of the thermal conductivity of the material.
- 10

9. A process as in Claim 8 wherein:

- (a) the electric current is a pulse of current and
- 15 (b) the duration and magnitude of said pulse of current is such that the thermoresistance device reaches a steady temperature greater than ambient temperature within a few seconds, said steady temperature being indicative of the thermal conductivity of the material.
- 20

10. A process as in Claim 5 wherein:

- (a) the thermoresistance device is in electrical circuit with a resistance bridge circuit as one of the resistance legs thereof.
- 25



-24-

11. A process as in Claim 6 wherein:
- (a) each thermoresistance device in thermal conduction with one of the materials is in electrical circuit with a resistance bridge circuit as one of the resistance legs thereof.
12. A process as in Claim 11 wherein:
- (a) the bridge circuit has four resistance legs, R_1 , R_2 , R_3 and R_4 ,
- (b) R_2 and R_4 are known variable resistances,
- (c) R_1 is the thermoresistance device in contact with the test material,
- (d) R_3 is the other thermoresistance device in thermal contact with the other material and,
- (e) the bridge is balanced by varying R_2 or R_4 or both R_2 and R_4 ,
- (f) whereby upon balancing the bridge circuit $R_2/R_4 = R_1/R_3$ indicating that the ratio of the thermal conductivity of the test material to the thermal conductivity of the other material varies as R_2/R_4 .
13. In a process of distinguishing a simulated diamond from a natural diamond, the step of:
- (a) measuring a parameter indicative of the thermal conductivity of the simulated diamond.
14. A process as in Claim 13 wherein:
- (a) the parameter is the temperature of a heated probe held against the simulated diamond.



-25-

15. A process as in Claim 14 wherein:
- (a) the temperature of the heated probe is measured while heat is applied to the probe and after the probe is held against the simulated diamond.
- 5
16. A process as in Claim 15 wherein:
- (a) the temperature is detected by a thermo-resistance device in the probe and
 - (b) the electrical resistance of the thermo-resistance device is measured as an indication of said temperature.
- 10
17. A process as in Claim 16 wherein:
- (a) the probe is held against the simulated diamond while at the same time,
 - (b) the heat is applied to the probe by feeding an electrical current to the thermoresistance device of sufficient magnitude to heat both the device and the simulated diamond.
- 15
18. A process as in Claim 17 wherein:
- (a) the electrical current fed to the thermo-resistance device causes heat flow to the probe at a rate not exceeding the combined rate of heat flow from the probe to the surroundings and the simulated diamond so that the probe temperature is indicative of the thermal conductivity of the simulated diamond.
- 20
- 25



-26-

19. A process as in Claim 18 wherein:
(a) the electric current is a pulse of current.
20. A process as in Claim 19 wherein:
(a) the duty cycle and magnitude of said pulse
5 of current is such that the synthetic diamond
reaches a steady state temperature greater
than ambient temperature within a few minutes,
said steady state temperature being indica-
tive of the thermal conductivity of the syn-
10 thetic diamond.
21. A process as in Claim 16 wherein:
(a) a similar thermoresistance device is in
thermal contact with a reference material,
and
15 (b) both thermoresistance devices are in elec-
trical circuit with a resistance bridge
circuit as different resistance legs thereof.
22. A process as in Claim 21 wherein:
(a) the bridge circuit has four resistance legs,
20 R1, R2, R3 and R4,
(b) R2 and R4 are known variable resistances,
(c) R1 is the thermoresistance device in contact
with the material
(d) R3 is the similar thermoresistance device in
25 thermocontact with the reference material,
and
(e) the bridge is balanced by varying R2 or R4
or both R2 and R4,



-27-

(f) whereby upon balancing the bridge circuit,
R2/R4=R1/R3 indicating that the ratio of
the thermal conductivity of the simulated
diamond to the thermal conductivity of the
reference material varies as R2/R4.

23. Apparatus for distinguishing a simulated diamond
from natural diamond comprising:

(a) means for measuring the temperature change
of a body in intimate thermal contact with
the simulated diamond as an indication of
the relative thermal conductivity of the
simulated diamond to natural diamond.

24. Apparatus as in Claim 23 wherein:

(a) the body is a heated probe that is in intimate
thermal contact with the simulated diamond.

25. Apparatus as in Claim 24 wherein:

(a) means are provided for applying heat to
said probe, sufficient to raise the temper-
ature of the probe above ambient temperature,
and

(b) the temperature of the probe is measured
after the heat is applied thereto and while
the probe is in intimate thermal contact with
the simulated diamond.

26. Apparatus as in Claim 25 wherein:

(a) a thermoresistance device is provided in the
probe for detecting said temperature, and



-28-

- (b) means are provided for measuring the electrical resistance of the thermoresistance device as an indication of said temperature.

27. A process as in Claim 26 wherein:

- 5 (a) the probe is so constructed that the thermoresistance device is held against the simulated diamond, and
- (b) means are provided for applying electrical current to the thermoresistance device of
- 10 sufficient magnitude to heat the probe.

28. Apparatus as in Claim 27 wherein:

- (a) said means for feeding electric current to the thermoresistance device includes a source of a pulse of electric current.

15 29. Apparatus as in Claim 28 wherein:

- (a) the duty cycle and magnitude of said pulse of current is such that the thermoresistance device reaches a steady temperature greater than ambient within a few seconds, said
- 20 temperature being indicative of the thermal conductivity of the simulated diamond.

30. Apparatus as in Claim 29 wherein:

- (a) an electrical resistance bridge circuit is provided,
- 25 (b) one of the resistance legs of said bridge circuit is the thermoresistance device, and
- (c) means are provided for balancing the bridge by varying one or more of the other resistance legs thereof,



-29-

(d) whereby the resistance values of said other legs are indicative of the thermal conductivity of the simulated diamond.

31. Apparatus as in Claim 30 wherein:

5 (a) another thermoresistance device is provided in thermal contact with a reference material of known thermal conductivity and in electrical circuit with said resistance bridge circuit as one of the resistance legs
10 thereof.

32. Apparatus as in Claim 31 wherein:

(a) the bridge circuit has four resistance legs R1, R2, R3 and R4,
(b) R2 and R4 are both variable resistances,
15 (c) R1 is the thermoresistance device in contact with the simulated diamond,
(d) R3 is the similar thermoresistance device in thermal contact with the reference material, and
20 (e) the bridge is balanced by varying R2 or R4 or both R2 and R4,
(f) whereby upon balancing the bridge circuit $R2/R4=R1/R3$ indicating that the ratio of the thermal conductivity of the simulated
25 diamond to the thermal conductivity of the reference material varies as $R2/R4$.

33. A method of determining the thermal conductivity of a test material comprising the steps of:



-30-

- (a) measuring a first steady temperature of a test probe by means of a temperature sensor,
 - (b) subjecting the test material to a steady heat flow relative to the test probe;
 - 5 (c) sensing a second steady temperature of the test probe by means of said temperature sensor, and
 - (d) providing an indication of the change in steady temperature of the probe at the
 - 10 temperature sensor.
34. A method of determining the thermal conductivity of a test material comprising the steps of:
- 15 (a) holding a test probe in thermal contact with the test material until the test material reaches a steady thermal condition,
 - (b) subjecting the test material to a pre-determined level of heat flow relative to the test probe until the test material reaches another steady thermal condition,
 - 20 and
 - (c) comparing the temperatures of the test probe during the two steady conditions to provide an indication of the thermal conductivity of the test material.
- 25 35. A method of distinguishing a simulated gem from a natural gem comprising the steps of:
- (a) subjecting the gem to steady heat flow, and
 - (b) measuring the temperature of the gem as an indication of thermal conductivity.



-31-

36. Apparatus for measuring the thermal conductivity of a test material comprising:
- (a) a test probe,
 - 5 (b) means for subjecting the test material to a change in heat flow relative to the test probe,
 - (c) a temperature sensor for sensing the temperature of the test probe, and
 - 10 (d) an electrical circuit for determining the thermal conductivity of the material as a function of the change in steady state temperature of the test probe resulting from said change in heat flow.



- 32 -

AMENDED CLAIMS

(received by the International Bureau on 13 August 1979 (13.08.79))

1. Apparatus for distinguishing materials comprising,
 - a probe having a small electric heat flow device in close thermal contact to a material contacting tip;
 - a pulse source for applying a pulse of electric current to the heat flow device; and
 - means for measuring the temperature change of the probe in intimate thermal contact with the material as an indication of the relative thermal conductivity of the material.
2. Apparatus as in Claim 1 wherein,
 - a thermoresistance device is provided in the probe for detecting said temperature, and
 - means are provided for measuring the electrical resistance of the thermoresistance device as an indication of said temperature.
3. Apparatus for distinguishing materials as claimed in Claim 1 wherein the duty cycle and magnitude of said pulse of current is such that, when the tip is placed against a gem, the probe approaches a steady temperature greater than ambient within a few seconds.



- 33. -

4. Apparatus for measuring the thermal conductivity of a test material comprising:

a test probe having a small test material contacting surface at a probe tip;

a small heat flow element in close thermal contact with the contacting surface for subjecting the test material to a change in heat flow relative to the test probe; and

means for sensing the temperature of the test probe near the contacting surface.

5. Apparatus as in Claim 4 further including:

an electrical circuit for determining the thermal conductivity of the material as a function of the change in steady state temperature of the test probe resulting from said change in heat flow.

6. Apparatus for distinguishing a simulated diamond from natural diamond materials comprising:

a probe having a thermoresistance device, the probe constructed to hold the thermoresistance device in intimate thermal contact with a simulated diamond,

means for providing a pulse of electric current to the thermoresistance device of sufficient magnitude to heat the probe sufficiently to raise the temperature of the probe above ambient temperature and,

means for measuring the electrical resistance of the thermoresistance device as an indication of the temperature of the probe.



- 34 -

7. Apparatus as in Claim 6 wherein,
the duty cycle and magnitude of said pulse of current is such that the thermoresistance device reaches a steady temperature greater than ambient within a few seconds, said temperature being indicative of the thermal conductivity of the simulated diamond.
8. An apparatus for measuring the thermal conductivity of a solid test material and of the type having a heating element and a temperature sensing element in a test material contacting probe, the improvement of:
a thermoresistance device mounted to the probe with means for applying electric current to the device for heating the probe and means for sensing the electrical resistance of the same device as a measure of probe temperature.
9. A process of distinguishing a simulated gem from a natural gem comprising the steps of:
subjecting the gem to a change in steady heat flow from a heat flow element in a probe through a probe tip; and
measuring the change in temperature of the probe as an indication of thermal conductivity of the gem.



- 35 -

10. A process as in Claim 9 wherein,
the temperature of the heated probe is measured while heat is applied to the probe and after the probe is held against the gem.
11. A process as in Claim 10 wherein,
the temperature is detected by a thermoresistance device in the probe and
the electrical resistance of the thermoresistance device is measured as an indication of said temperature.
12. A process as in Claim 11 wherein,
the probe is held against the gem while at the same time,
the heat is applied to the probe by feeding an electrical current to the thermoresistance device of sufficient magnitude to heat both the device and the gem.
13. A process as in Claim 12 wherein,
the electric current fed to the thermoresistance device causes heat flow to the probe at a rate not exceeding the combined rate of heat flow from the probe to the surroundings and the gem so that the probe temperature is indicative of the thermal conductivity of the gem.
14. A process as in Claim 11 wherein,
a similar thermoresistance device is in thermal contact with a reference material, and
both thermoresistance devices are in electrical circuit with a resistance bridge circuit as different resistance legs thereof.



- 36 -

15. A process as in Claim 9 wherein,
the heat flow element is heated by a pulse
of electric current.
16. A process as in Claim 15 wherein,
the duty cycle and magnitude of said
pulse of current is such that the synthetic
gem reaches a steady state temperature
greater than ambient temperature within a
few seconds, said steady state temperature
being indicative of the thermal conductivity
of the gem.
17. In a process of distinguishing a test material
from another material, the following combination
of steps:
placing a probe in intimate contact with
the test material,
applying a pulse of electric current to
an electric heat flow device in the probe
to cause the flow of heat between the probe
and the test material, and
measuring the temperature of the probe,
said temperature being indicative of the
rate of conduction of heat relative to the
probe by the test material.
18. A process as in Claim 17 wherein,
the temperature of the probe is measured
while heat is being applied thereto.
19. A process as in Claim 17 wherein,
the temperature of the probe is measured
after heat has been applied thereto and while
substantially negligible heat is being ap-
plied thereto.



- 37 -

20. A process as in Claim 17 further including,
the same combination of steps performed
with another probe and the other material
in the same sequence and at the same time
as the combination of steps set forth by
Claim 17.
21. A process as in Claim 17 wherein,
the probe includes a thermoresistance
device,
heat is applied to the probe by feeding
an electric current to the device, and
the measure of temperature is the elec-
trical resistance of the thermoresistance
device.
22. A process as in Claim 21 wherein,
the rate of heat flow so produced does
not exceed the combined rate of conduction
of heat from the probe by surroundings and
the test material, whereby the probe is at
a temperature indicative of the thermal
conductivity of the material.
23. A process as in Claim 21 wherein,
the duration and magnitude of said pulse
of current is such that the thermoresistance
device reaches a steady temperature greater
than ambient temperature within a few seconds,
said steady temperature being indicative of
the thermal conductivity of the material.



- 38 -

24. A method of determining the thermal conductivity of a test material comprising the steps of:

measuring a first steady temperature of a test probe;

subjecting the test material to a steady heat flow relative to the test probe through a probe tip by means of a small element positioned in close thermal contact with the test material;

sensing a second temperature of the test probe; and

providing an indication of the change in temperature of the probe.

25. A method of determining the thermal conductivity of a test material such as a mounted diamond comprising the steps of:

holding the tip of a test probe in thermal contact with the test material until the test material reaches a steady thermal condition;

subjecting the test material to a pre-determined level of heat flow relative to the test probe by means of a small heat flow element positioned in close thermal contact with the test material, the magnitude of the heat flow being such that the difference in temperatures of the probe in contact with a mounted diamond, with and without heat flow, is dominated by the conductivity of the diamond; and

providing an indication of the thermal conductivity of the test material based on the change in temperature of the test probe.



- 39 -

26. In a process of distinguishing a test material from another material, the following combination of steps:

placing a probe having a thermoresistance device in intimate thermal contact with the test material;

applying heat to the probe by feeding a pulse of current to the thermoresistance device, the duration and magnitude of said pulse of current being such that the rate of heat so produced does not exceed the combined rate of heat from the probe by surroundings and the test material, and such that the thermoresistance device reaches a steady temperature greater than ambient temperature within a few seconds, said steady temperature being indicative of the thermal conductivity of the material, and

thereafter, measuring the temperature of the probe by the electrical resistance of the thermoresistance device, said temperature being indicative of the rate of conduction of heat from the probe by the test material.



- 40 -

27. A process of distinguishing a simulated diamond from a natural diamond comprising:

holding a probe against the simulated diamond,

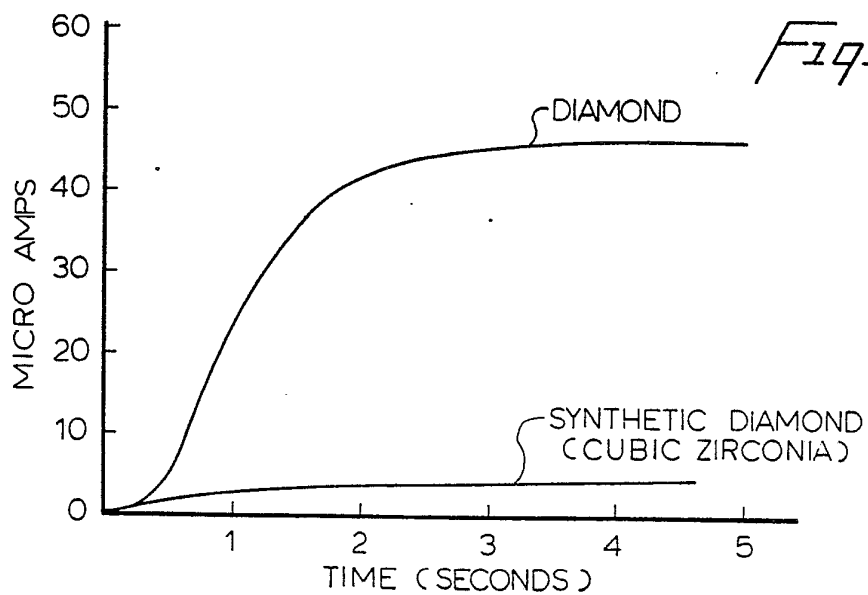
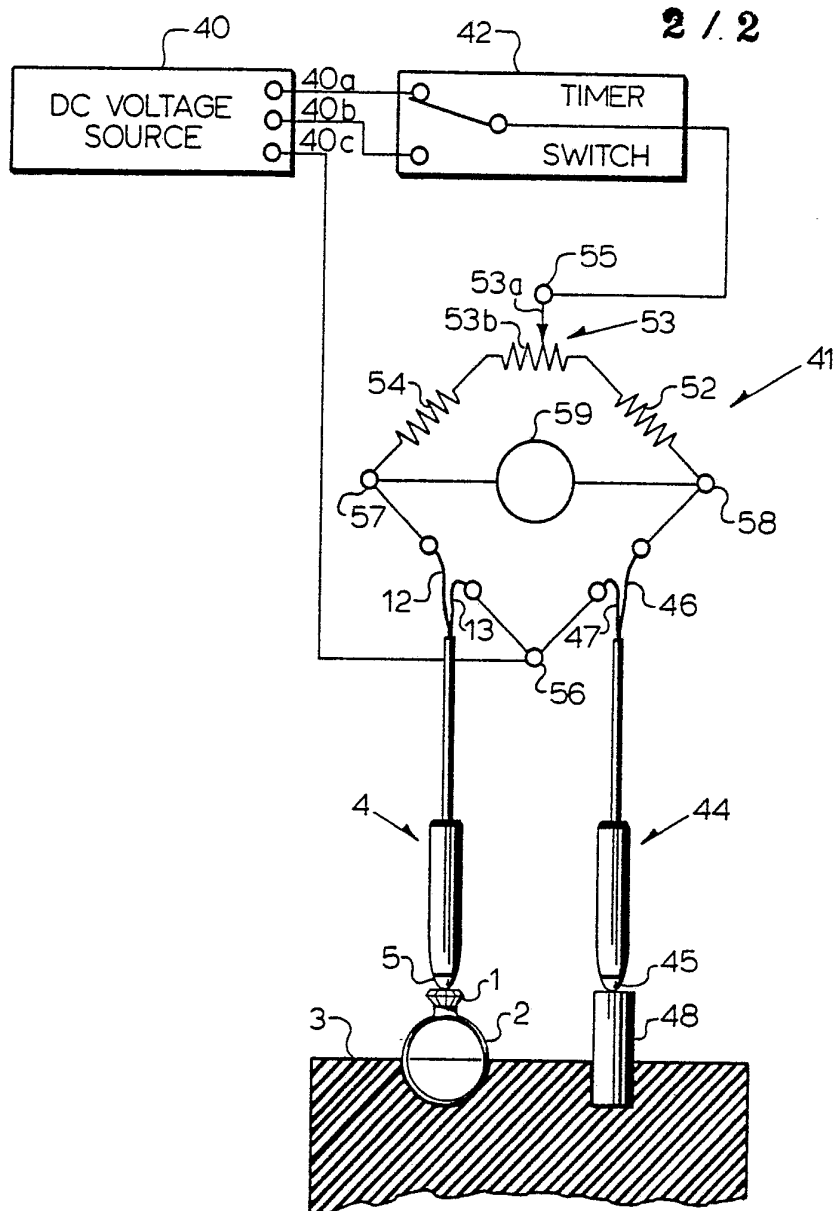
applying a pulse of current to a thermoresistance device in the probe to cause heat flow from the device to the probe at a rate not exceeding the combined rate of heat flow from the probe to the surroundings and to the simulated diamond so that the probe is heated to a temperature indicative of the thermal conductivity of the simulated diamond, and

detecting the temperature of the probe by measuring the electrical resistance of the thermoresistance device.

28. A process as in Claim 26 wherein:

the duty cycle and magnitude of said pulse of current is such that the simulated diamond reaches a steady state temperature greater than ambient temperature within a few seconds, said steady state temperature being indicative of the thermal conductivity of the simulated diamond.





INTERNATIONAL SEARCH REPORT

International Application No PCT/US79/00220

I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all) ³ According to International Patent Classification (IPC) or to both National Classification and IPC INT CL. G01N25/18 U.S. CL. 73/15A 25/00911																										
II. FIELDS SEARCHED Minimum Documentation Searched ⁴ <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th style="width: 20%;">Classification System</th> <th style="width: 80%;">Classification Symbols</th> </tr> <tr> <td style="text-align: center; padding: 10px;">U.S.</td> <td style="padding: 10px;">73/15R, 15A</td> </tr> </table> Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁵			Classification System	Classification Symbols	U.S.	73/15R, 15A																				
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III. DOCUMENTS CONSIDERED TO BE RELEVANT ¹⁴ <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th style="width: 10%;">Category [*]</th> <th style="width: 60%;">Citation of Document, ¹⁶ with indication, where appropriate, of the relevant passages ¹⁷</th> <th style="width: 30%;">Relevant to Claim No. ¹⁸</th> </tr> <tr> <td style="text-align: center;">X</td> <td>U.S., A, 2,323,715 Published 06 July 1943, Kuehni</td> <td>1-36</td> </tr> <tr> <td style="text-align: center;">X</td> <td>U.S., A, 2,951,360 Published 06 September 1960, Sampson et al</td> <td>1-36</td> </tr> <tr> <td style="text-align: center;">X</td> <td>U.S., A, 2,264,968 Published 02 December 1941 DeForest</td> <td>2,4-8, 10-12</td> </tr> <tr> <td style="text-align: center;">X</td> <td>U.S., A, 3,611,786 Published 12 October 1971, Schorr</td> <td>13-17, 35</td> </tr> <tr> <td style="text-align: center;">X</td> <td>U.S., A, 3,981,175 Published 21 September 1976, Hammond, III et al</td> <td>13-16</td> </tr> <tr> <td style="text-align: center;">X</td> <td>GB, A, 855,658 Published 07 December 1960, Powell</td> <td>1-36</td> </tr> <tr> <td style="text-align: center;">X,P</td> <td>U.S., A 4,138,878 Published 13 February 1979 Holmes</td> <td>4,6,21,22</td> </tr> </table>			Category [*]	Citation of Document, ¹⁶ with indication, where appropriate, of the relevant passages ¹⁷	Relevant to Claim No. ¹⁸	X	U.S., A, 2,323,715 Published 06 July 1943, Kuehni	1-36	X	U.S., A, 2,951,360 Published 06 September 1960, Sampson et al	1-36	X	U.S., A, 2,264,968 Published 02 December 1941 DeForest	2,4-8, 10-12	X	U.S., A, 3,611,786 Published 12 October 1971, Schorr	13-17, 35	X	U.S., A, 3,981,175 Published 21 September 1976, Hammond, III et al	13-16	X	GB, A, 855,658 Published 07 December 1960, Powell	1-36	X,P	U.S., A 4,138,878 Published 13 February 1979 Holmes	4,6,21,22
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* Special categories of cited documents: ¹⁵ "A" document defining the general state of the art "E" earlier document but published on or after the international filing date "L" document cited for special reason other than those referred to in the other categories "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but on or after the priority date claimed "T" later document published on or after the international filing date or priority date and not in conflict with the application, but cited to understand the principle or theory underlying the invention "X" document of particular relevance																										
IV. CERTIFICATION <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 50%; padding: 5px;"> Date of the Actual Completion of the International Search ¹ 25 MAY 1979 </td> <td style="width: 50%; padding: 5px;"> Date of Mailing of this International Search Report ² 19 JUN 1979 </td> </tr> <tr> <td style="padding: 5px;"> International Searching Authority ¹ ISA/US </td> <td style="padding: 5px;"> Signature of Authorized Officer ²⁰ <i>Herbert Goldstein</i> Herbert Goldstein </td> </tr> </table>			Date of the Actual Completion of the International Search ¹ 25 MAY 1979	Date of Mailing of this International Search Report ² 19 JUN 1979	International Searching Authority ¹ ISA/US	Signature of Authorized Officer ²⁰ <i>Herbert Goldstein</i> Herbert Goldstein																				
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FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

A	U.S., A, 3,810,009 Published 07 May 1974, Hausler et al	34
A	U.S., A, 2,924,771 Published 09 February 1960, Greenberg et al	1-36
A	U.S., A, 3,016,732 Published 16 January 1962, Hanyasz et al	1-36
A	U.S., A, 3,075,377 Published 29 January 1963, Lang	1-36

V. ☐ OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE ¹⁰

This international search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1. ☐ Claim numbers because they relate to subject matter ¹² not required to be searched by this Authority, namely:

2. ☐ Claim numbers because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out ¹³, specifically:

VI. ☐ OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING ¹¹

This International Searching Authority found multiple inventions in this international application as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.

2. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

Remark on Protest

☐ The additional search fees were accompanied by applicant's protest.

☐ No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

A	U.S., A, 3,084,534 Published 09 April 1963 Goton	1-36
A	U.S., A, 3,279,239 Published 18 October 1966, Arends et al	1-36

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