

METHOD AND APPARATUS FOR IDENTIFYING MATERIALS

(13)

This invention relates to a method and apparatus for identifying materials by obtaining a measure of their thermal conductivities and more more particularly, but not exclusively, for identifying gemstones which may only
5 be supplied in very small samples.

Methods and apparatus for identifying materials such as gemstones by means of their thermal conductivities have been previously proposed. Such apparatus has been either large and expensive or somewhat crude and cumbersome, and
10 not particularly well adapted for use in identifying such materials as gemstones where the sample size is likely to be particularly small.

Although certain of the prior patent specifications in this field make suggestions in relation to adapting the
15 specifically disclosed apparatus for use in relation to small sample sizes, none of these proposals is particularly well adapted for the purpose and it is an object of the present invention to provide improvements in this respect.

According to the invention there is provided a method
20 and apparatus for use in identifying materials such as gemstones, as defined in the accompanying claims.

During use of apparatus as defined in the claims, as an

approximation, the maximum temperature difference between the thermocouple junctions is determined by the ratio of the thermal resistance of the branch or element of the thermocouple between the junctions to the thermal resistance of the interface between the contacting junction and the material to be assessed or distinguished. The latter thermal resistance is a function of the thermal conductivity of the material under test provided that the area of contact is substantially constant in all tests made with the apparatus.

In an embodiment of the apparatus or comparator of the invention, the short branch or element of the thermocouple has been a piece of copper wire of about 0.3 mm diameter and about 1 cm in length, the rest of the thermocouple has consisted of constantan wires of about the same diameter. The constantan wires have been coiled to provide a spring inside a glass tube of about 3 mm internal diameter. The copper wire has been allowed to project beyond the constantan at the junction held against the material to be assessed so that the interface has always been between copper and the test material. It has been arranged that the amount of movement against the spring on making contact always has the same value. In one arrangement the whole device has been heated and removed from the source of heat before placing the junction in contact

with the material. The resultant e.m.f. depends on the thermal conductivity of the test material.

Additionally, where the contacting junction has a low thermal capacitance the comparator is particularly suited for assessing small samples of material. In another arrangement the junction remote from the test material has been continuously heated by means of a small electrical heater.

Use of apparatus of this invention is most beneficial in distinguishing diamond from other gemstones, which other gems have a similarly cubic structure and a closely approximating refractive index to that of diamond.

Other values of the present invention reside in that it entails the non-destructive assessment of samples which may only be available as very small samples.

An embodiment of the invention will now be described by way of example with reference to the accompanying drawings, in which:-

Fig. 1 is a schematic sectional view of thermal comparator apparatus in accordance with the present invention; and

Fig. 2 is a graph showing the variation of thermoelectric e.m.f. with time in relation to various gemstones as sensed by the comparator of Fig. 1.

In the comparator apparatus as shown in Fig. 1,

thermocouple junctions are formed between a pair of primary thermocouple elements in the form of two lengths of enamelled constantan wire 10 of 0.46 mm diameter, and a secondary thermocouple element in the form of a short (10 mm) piece of copper wire 11 of 0.22 mm diameter connected between elements 10. A contact in the form of a conical copper head 12, of somewhat less than 1 cubic mm volume, is soldered to the lower thermocouple junction.

The copper wire 11 and the two thermocouple junctions are held in a PVC slide tube or sleeve 13, which can slide in a glass mounting tube 14. The constantan wires 10 are cemented at 15 to the inside of one end of the glass tube and take a spiral form over part of their length. The point of the copper head 12 protrudes from the other end of the glass tube 14 by about 1 mm, but retracts by this amount when the device is pressed against a hard surface, such as a gemstone.

During test work the free ends 10' of the constantan wires 10 were connected (via copper junctions at the same temperature) to a digital millivoltmeter (not shown); an instrument having a limit of resolution of not more than about 100 μ V was used.

The comparator apparatus was first placed in an environment at a specific elevated temperature. In

one example a test tube resting in a beaker of boiling water was used, and it was found that the comparator reached equilibrium with its surroundings after two minutes. The apparatus was then lifted from the test tube and the copper contact 12 was pressed against a gemstone and the maximum e.m.f. registered by the millivoltmeter was recorded. The rate of change of e.m.f. with time is perhaps a more sensitive measure of the thermal conductivity, but the maximum e.m.f. is easier to determine. The table below summarises the values that were obtained with various different gemstones. Invariably diamond gave the largest e.m.f. and cubic zirconia invariably gave the smallest.

The variation of e.m.f. with time was observed using a Tektronix Type 7623A storage oscilloscope with a plug-in amplifier Type 7A22. The behaviour of some of the gemstones and of a piece of glass is shown in Fig. 2. It is noted that there is very little development of the e.m.f. while the comparator is being transferred from the boiling water to the material to be tested. Furthermore, the rapid rise of e.m.f. when contact is made with the sample is usually followed by a rather slow decrease. It is thought that the maximum e.m.f. should correspond quite closely to the value that could be expected if the bulk of the gemstone were continually maintained at room temperature and the remote thermocouple junction

were maintained at 100°C. Using Clark and Powell's equation 1962 J.Sci. Instrum. 39, 545-51 for the thermal resistance of the constriction, it was found that the results were consistent with a reasonable value for the contact radius of about 0.01mm.

TABLE. Maximum e.m.f. from Thermal Comparator

	<u>Sample</u>	<u>Thermal</u> Conductivity *W/mK	<u>Maximum e.m.f. mV</u>	
			Mean	Range of 5 readings
	Diamond	990-2320	1.56	1.47 - 1.63
10	Corundum (synthetic)	32-35	1.19	1.07 - 1.27
	Topaz	19	1.01	0.97 - 1.06
	Zircon	4.1 - 4.2	0.99	0.97 - 1.04
	Spinel	12	0.83	0.74 - 0.96
15	(synthetic) YAG ⁺		0.75	0.70 - 0.80
	Quartz	5.9 - 11	0.75	0.67 - 0.82
	GGG ⁺⁺		0.62	0.58 - 0.65
	Cubic zirconia		0.50	0.45 - 0.53

20 * Values near room temperature from CRC "Handbook of Chemistry and Physics" 57th Edition 1976-7.

+ Yttrium aluminium garnet

++ Gadolinium germanium garnet

25 The heat source for the thermocouple wires may take the form of an electrical resistance element

cemented to the wires. A resistive heater may be employed in addition to or instead of indirect heating as described in the embodiment.

5 Further, the heating of a branch or element of the thermocouple may be provided at the centre of the branch rather than at the junction. If the heater circuit is provided it may incorporate a boosting device so that the comparator reaches its operating condition almost immediately.

10 The contact head 12 of the comparator may be of materials other than copper and may, for example, be of gold, platinum or other materials which may be more suited to particular applications of the invention.



CLAIMS:

1 Apparatus for use in identifying materials such as
gemstones, said apparatus comprising a thermocouple having
thermocouple junctions between dissimilar metals, one of
the junctions of the thermocouple having a contact
5 associated therewith for engagement with a material to be
tested whereby monitoring the thermoelectric e.m.f. between
the junctions of the thermocouple after making such
engagement with a material to be tested following heating
the thermocouple provides identification data relating to
10 the material under test, said contact being in the form of
a head secured to said one of the thermocouple junctions,
the head having a volume of less than one cubic millimetre,
resilient means and a stop being associated with said
contact whereby the contact can be pressed against a material
15 to be tested and caused to retract a predetermined
distance defined by said stop against the action of the
resilient means.

2 Apparatus according to claim 1 wherein said
resilient means is provided by a coiled electrical conductor
20 which acts as a spring.

3 Apparatus according to claim 2 wherein resilient
means in the form of a coiled electrical conductor is
provided in both of the electrical connections to the
thermocouple.



4 Apparatus according to any one of the preceding claims wherein said thermocouple junctions are mounted on a slide member which is itself slidably received in an outer mounting member.

5 5 Apparatus according to claim 4 wherein said slide member and said mounting member are both in the form of tubes.

6 Apparatus according to claim 5 wherein said slide member comprises a tube of PVC and said mounting member
10 comprises a tube of glass.

7 Apparatus according to any one of the preceding claims wherein said two dissimilar metals are copper and constantan.

8 Apparatus according to any one of the preceding
15 claims comprising an electrical resistor associated with the thermocouple to act as a heat source.

9 Apparatus according to any one of the preceding claims in combination with voltage detecting apparatus capable of detecting thermoelectric e.m.f.'s.

20 10 Apparatus for identifying materials such as gemstones substantially as described herein with reference

to the accompanying drawings.

11 A method of identifying materials such as gemstones
by use of apparatus according to claim 1, the method comprising
the step of heating the thermocouple, bringing the contact
5 into engagement with a material to be tested, pressing the
contact against the material so as to cause the contact to
retract a predetermined distance defined by said stop
against the action of the resilient means, and monitoring
the thermoelectric e.m.f. between the junctions of the
10 thermocouple.

The text of the specification has been reproduced by photocopying the applicants original typescript. It may contain a few amendments which are difficult to read. The original typescript containing these amendments may be inspected on the premises of the Patent Office

