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(54) Title
DIAMOND MAPPING

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(56) Prior Art Documents
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US 4030827

(57) Claim

1. A method for producing a record of a diamond, the method comprising the steps of (a) placing the diamond in a beam of monochromatic laser radiation capable of causing Raman radiation to be scattered from the diamond, (b) passing the scattered Raman radiation from the diamond through a filter adapted to pass only scattered Raman radiation characteristic of diamond, (c) measuring the intensity of the filtered Raman radiation, and (d) recording the intensity of the filtered Raman radiation at one or more different orientations of the diamond.

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(57) Abstract

A method for mapping the crystal structure of a diamond comprises placing the diamond in a beam of monochromatic laser radiation, filtering the resultant scattered Raman radiation, and measuring the intensity of the filtered radiation at one or more different orientations of the diamond. The intensity may be recorded to produce a record of the diamond which may be compared with records of known diamonds to identify the diamond.

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Complete Specification for the invention entitled:

"DIAMOND MAPPING"

The following statement is a full description of this invention including the best method of performing it known to us.

The present invention relates to a method for producing a record of a diamond and a method for identifying a diamond.

Diamonds have long been recognised as being of great value not only for decorative and industrial purposes but also as an investment. Their increasing value has presented problems of security for owners, insurance companies and police authorities. Identification of a recovered diamond which has been lost or stolen is made difficult because the superficial appearance of the diamond may have been changed by cutting, repolishing and the like. Conventionally, the recognition of diamond has been achieved on the basis of a record of characteristics comprising carat weight, cut, colour type, clarity etc.

More recently, X-ray topography has been used for identifying diamonds, as is disclosed in UK Patent No. 1,547,371. In this technique a set of records is produced by X-ray topography to provide an overall point-by-point three-dimensional representation of the diamond. The set of records of a recovered diamond that has been lost or stolen may be compared with the sets of records of known diamonds as a means of identifying the recovered diamond. However, this technique may be slow and requires interpretation of the diffraction patterns to produce the sets of records of defects.

The Raman signal of diamond is much stronger than that of other materials because diamond only contains carbon to carbon bonding and

its Raman signal occurs at a position well separated from those of other minerals. Also, as diamond only contains one type of carbon to carbon bond, there is only a single Raman signal which can be readily distinguished from associated broad band fluorescence. Thus the Raman signal is highly specific for diamond. The intensity of the Raman signal is affected by the crystal structure of the diamond and hence by the presence or otherwise of imperfections or inclusions in the diamond. For example, it has been found that imperfections cause a broadening of the diamond Raman signal and inclusions do not give a diamond Raman signal.

Thus according to the present invention there is provided a method for producing a record of a diamond, the method comprising the steps of (a) placing the diamond in a beam of monochromatic laser radiation capable of causing Raman radiation to be scattered from the diamond, (b) passing the scattered Raman radiation from the diamond through a filter adapted to pass only scattered Raman radiation characteristic of diamond, (c) measuring the intensity of the filtered Raman radiation, and (d) recording the intensity of the filtered Raman radiation at one or more different orientations of the diamond.

The invention also includes records whenever produced by the method as hereinbefore described.

According to the present invention there is also provided an apparatus for producing a record of a diamond, the apparatus comprising in combination (a) means for holding the diamond in a beam of monochromatic laser radiation capable of causing Raman radiation to be scattered from the diamond, (b) means for filtering the resultant scattered Raman radiation, said filter being adapted to pass only scattered Raman radiation characteristic of diamond, (c) means for measuring the intensity of the filtered Raman radiation, and (d) means for recording the intensity of the filtered Raman radiation at one or more different orientations of the diamond to produce a record of the diamond.

Preferably, the Raman intensities are recorded at three mutually perpendicular orientations of the diamond.

The record of the diamond may be used for identification purposes. Thus the record of a recovered diamond that has been lost or stolen may be compared with records of known diamonds so that it may be identified. Thus, also, according to the present invention there is provided a method for identifying a diamond, the method comprising the steps of (a) placing the diamond to be identified in a beam of monochromatic laser radiation capable of causing Raman radiation to be scattered from the diamond, (b) passing the scattered Raman radiation from the diamond through a filter adapted to pass only scattered Raman radiation characteristic of diamond, (c) measuring the intensity of the filtered Raman radiation, (d) recording the intensity of the filtered Raman radiation at one or more different orientations of the diamond, and (e) comparing the recorded intensities with records of known diamonds whereby the diamond may be identified.

Preferably the monochromatic laser radiation has a wavelength in the range 450 to 650 nanometers. The filtering means may be a suitable optical arrangement such as a collection optic and monochromator. Preferably, the scattered Raman radiation is focused by a suitable lens arrangement with a long depth of focus so that the scattered Raman radiation from throughout the diamond is in focus at the detector.

The record may be a point-by-point record of the Raman intensities or may be a record of the Raman intensities from the whole of the diamond simultaneously. Thus in the former case the record may be in the form of values stored on magnetic tape or in a computer etc. and in the latter case the record may be in the form of a photograph.

In one embodiment of the present invention a video or television camera may be adapted to display the scattered Raman intensities on a monitor or television screen. It is envisaged that in this embodiment the invention may enable a record to be made in the form of images stored by conventional means e.g. video tape, for different orientations of the diamond, which may be used for future identification purposes.

In another embodiment of the present invention, a photomultiplier or multichannel detector (e.g. diode array detector) may be adapted to scan the scattered Raman radiation. It is also envisaged that the means for holding the diamond in the laser radiation may be adapted so that the orientation of the diamond may be changed under automatic, microprocessor or computer control. A record of the diamond may be made in the form of stored Raman intensities which may be stored by conventional means, for example in a computer or on magnetic tape etc. In this embodiment it may be possible for a computer to control the orientation of the diamond and the production of the record so that a three-dimensional record of the diamond may be produced. This may be stored by conventional means such as in a computer or on magnetic tape or as a hologram, produced under the control of a computer.

In another embodiment of the present invention the filtered Raman radiation may be measured and recorded by a camera with a photographic plate or film sensitive to the filtered Raman radiation. The plate or film is suitably processed to produce a two-dimensional image of the scattered Raman intensity of the diamond in the selected orientation. The record of the diamond may take the form of several such photographic images at different orientations of the diamond and may be used for future identification purposes.

The invention will now be described by way of example only, and with reference to the accompanying drawings. Figures 1 to 4 show graphically, the Raman intensities from various parts of a diamond with an imperfection. Figure 5 shows, in schematic form, an apparatus which may be used to map the crystal structure of a diamond and to produce a record of the diamond which may be used for future identification purposes.

To show that Raman spectroscopy may be used to map the crystal structure of a diamond to show an imperfection, a series of Raman spectrograms were recorded in the region of an imperfection in a diamond. An argon ion laser was used to produce a beam of radiation

having a wavelength of 514.5 nanometres. The beam was used to irradiate a 2 micron diameter spot on the diamond. Resultant scattered Raman radiation was measured using a Jobin-Yvon 3000S spectrometer which measured the intensity of the Raman radiation and recorded it as the spectrograms shown in Figures 1 to 4 as graphs of Raman intensity against Raman shift. The diamond had an imperfection which was a pit, 8 microns in diameter, on its surface. Figure 1 shows the Raman spectrogram from a region of the diamond distant from the pit. The spectral peak in Figure 1 is sharp. Figure 2 shows a spectrogram from the centre of the pit and this shows that the Raman spectral peak is broader due to the imperfection. Figures 3 and 4 show spectrograms from the the sides of the pit. They show the peak being broader but the broadening is asymmetric. It is envisaged that this peak broadening would, for example, show up as a reduction in the Raman intensity on a photographic record of the diamond produced by the method according to the present invention.

To show that Raman spectroscopy may be used to map the crystal structure of a diamond to show an inclusion the method according to the present invention was used to make a record of a diamond with a simulated inclusion. The diamond had a piece of potassium nitrate on it.

The diamond was held in a holder and irradiated with laser radiation of wavelength 514.5 nanometres from a Spectra Physics 165 argon ion laser. The laser power at the laser head was about 300 mW which reduced to about 25 to 50 mW at the sample. The laser radiation was rotated using spinning mirrors and passed through an annular condenser surrounding a 50 times magnification microscope objective to irradiate an area of the diamond 200 microns in diameter. Resultant scattered Raman radiation was collected by the microscope objective and passed to a Jobin-Yvon Raman spectrometer which was operated in its imaging mode (very wide slits and some lenses retracted) to ensure that the image was transmitted undistorted to a 2-dimensional intensified silicon intensified target (ISIT) camera for detection. By focusing the objective, different planes of the diamond may be mapped. It is envisaged that

by using an objective with a large depth of focus the whole diamond may be mapped. The spectrometer passed only radiation characteristic of diamond to the detector. The detector had a 2.5cm square target with a variable integration time from 1 second to a few minutes. The image on the detector was recorded photographically which showed a bright white image in the regions which were pure diamond and dark regions in the area of the non-diamond inclusion. It is envisaged that a record of the inclusions in the diamond may be produced by recording images from the detector for several orientations of the diamond.

Figure 5 shows, in schematic form, an apparatus which may be used to map the crystal structure of a diamond and to produce a record of the diamond which may be used for identification purposes.

A diamond (1) is placed in a holder (2) in a beam of monochromatic laser radiation (3). The radiation (3) is provided by a laser (4). It has a wavelength in the range 450 to 650 nanometres and is capable of causing Raman radiation (5) to be scattered from the diamond (1). The scattered Raman radiation (5) is collected by a lens (6), passed through a beam splitter (7) and to a filter (8) which only passes Raman radiation characteristic of diamond. The intensity of the filtered Raman radiation (9) is measured by a video camera (10) which produces an image on a video monitor (11). The image shows a bright white intensity corresponding to diamond with grey regions indicative of imperfections and black regions indicative of inclusions.

A record of the image on the video monitor is made by photographing it with a camera (12). Several such photographs may be made for different orientations of the diamond to produce a record of the diamond. The record thus produced may be used for future identification purposes.

The claims defining the invention are as follows:

1. A method for producing a record of a diamond, the method comprising the steps of (a) placing the diamond in a beam of monochromatic laser radiation capable of causing Raman radiation to be scattered from the diamond, (b) passing the scattered Raman radiation from the diamond through a filter adapted to pass only scattered Raman radiation characteristic of diamond, (c) measuring the intensity of the filtered Raman radiation, and (d) recording the intensity of the filtered Raman radiation at one or more different orientations of the diamond.

2. A method for producing a record of a diamond according to claim 1 in which the intensity of the filtered Raman radiation is recorded by electronic, photographic, magnetic or holographic means.

3. A method for producing a record of a diamond according to claim 1 or claim 2 in which the intensity of the filtered Raman radiation is recorded at three mutually perpendicular orientations of the diamond.

4. A record of a diamond whenever produced by a method according to any of claims 1 to 3.

5. A method for identifying a diamond, the method comprising the steps of (a) placing the diamond to be identified in a beam of monochromatic laser radiation capable of causing Raman radiation to be scattered from the diamond, (b) passing the scattered Raman radiation from the diamond through a filter adapted to pass only scattered Raman radiation characteristic of diamond, (c) measuring the intensity of the filtered Raman radiation, (d) recording the intensity of the filtered Raman radiation at one or more different orientations of the diamond, and (e) comparing the recorded intensities with records of known diamonds whereby the diamond may be identified.

6. A method for identifying a diamond according to claim 5 in which the intensity of the filtered Raman radiation is recorded at three mutually perpendicular orientations of the diamond.

7. An apparatus for producing a record of a diamond, the apparatus comprising in combination (a) means for holding the diamond in a beam of monochromatic laser radiation capable of causing Raman radiation to be scattered from the diamond, (b) means for filtering the resultant scattered Raman radiation, said filter being adapted to pass only scattered Raman radiation characteristic of diamond, (c) means for measuring the intensity of the filtered Raman radiation, and (d) means for recording the intensity of the filtered Raman radiation at one or more different orientations of the diamond to produce a record of the diamond.

8. A method for producing a record of a diamond as hereinbefore described and with reference to the drawings.

9. A method for identifying a diamond as hereinbefore described and with reference to the drawings.

DATED this 25th day of September, 1990

THE BRITISH PETROLEUM COMPANY p.l.c.,
By its Patent Attorneys,
E. F. WELLINGTON & CO.,
By:

Bruce Wellington

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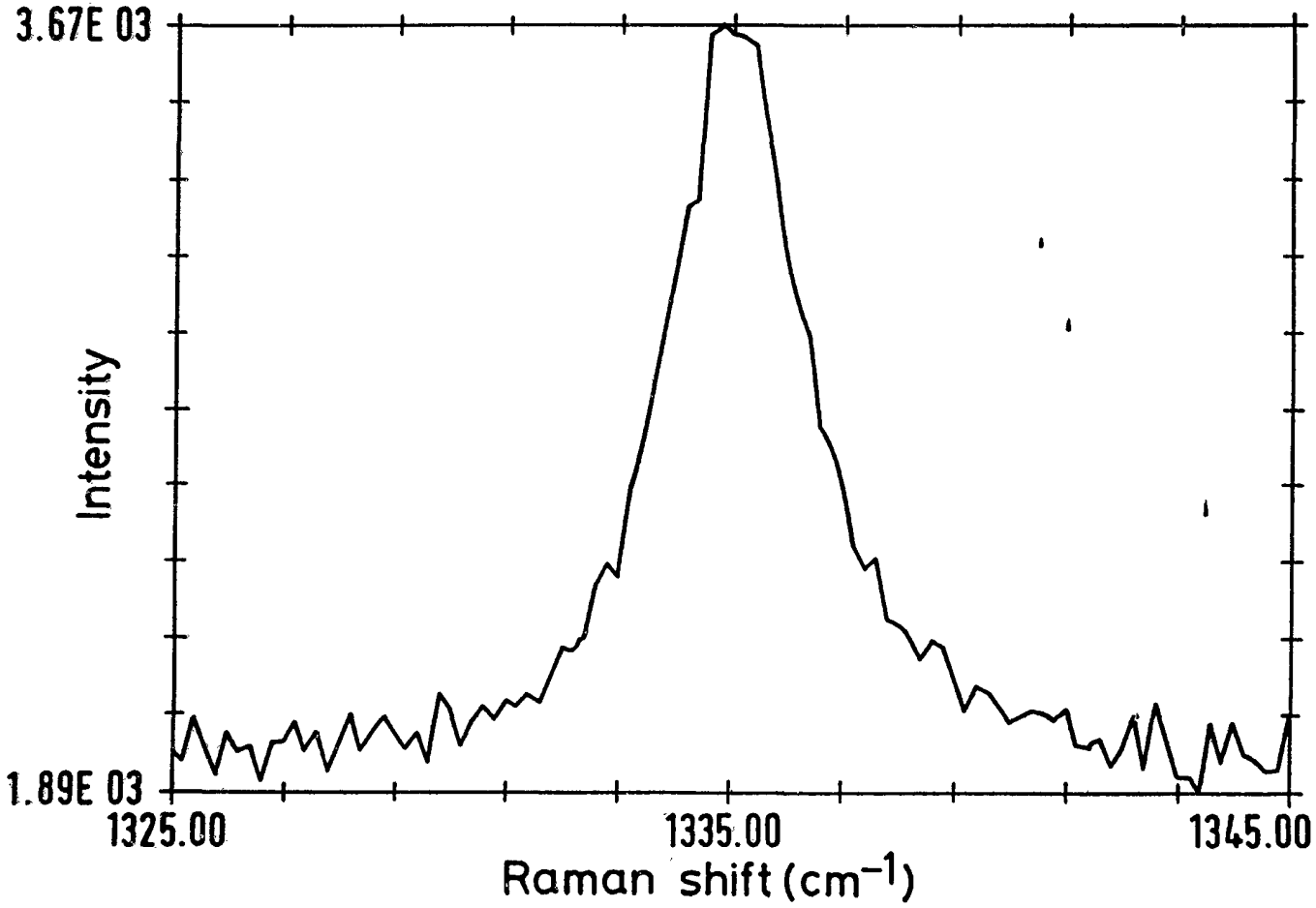
BRUCE S. WELLINGTON

FIG.1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1.89E 03
Intensity
1325.00
1335.00
1345.00
Raman shift (cm^{-1})

FIG. 2

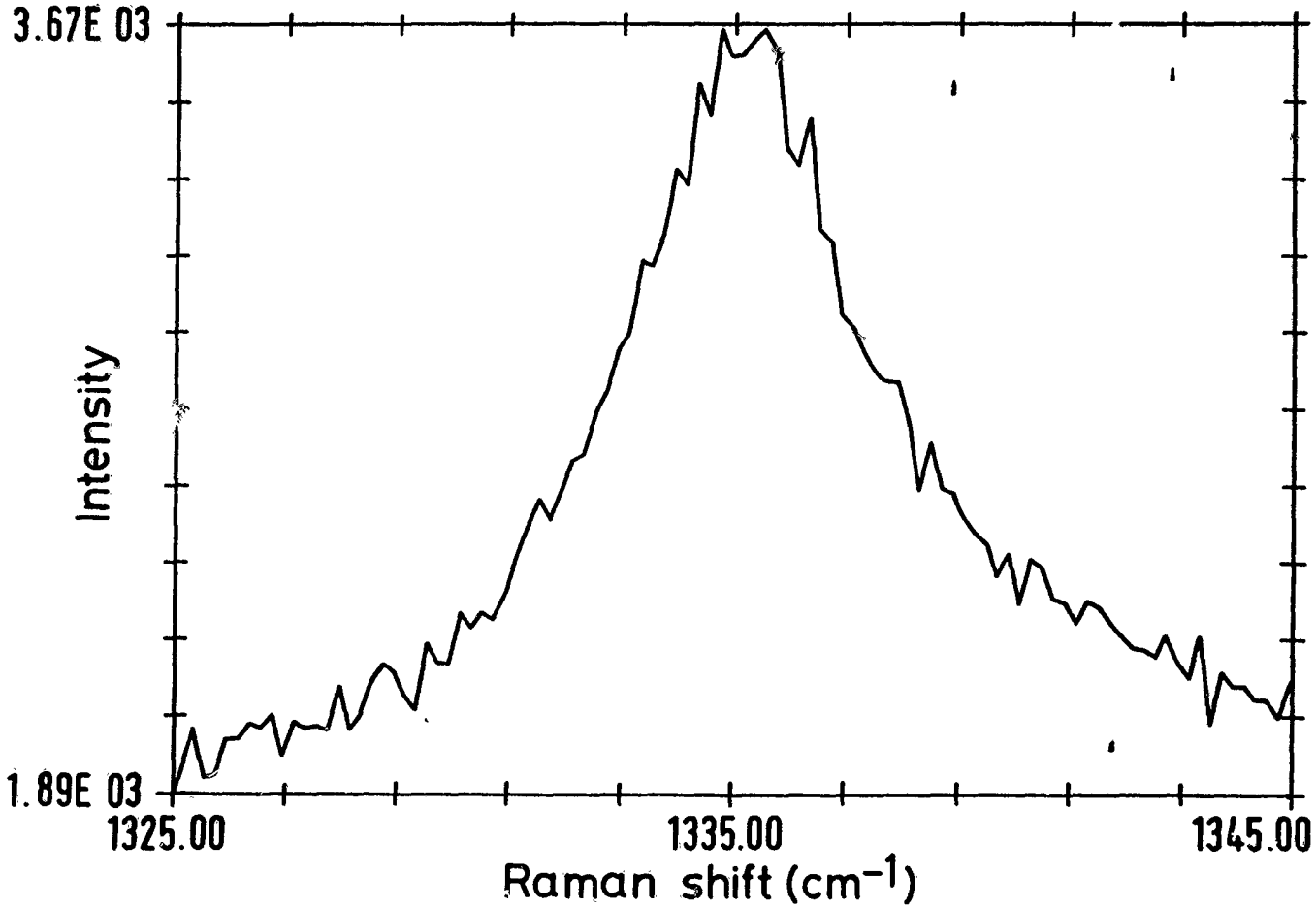
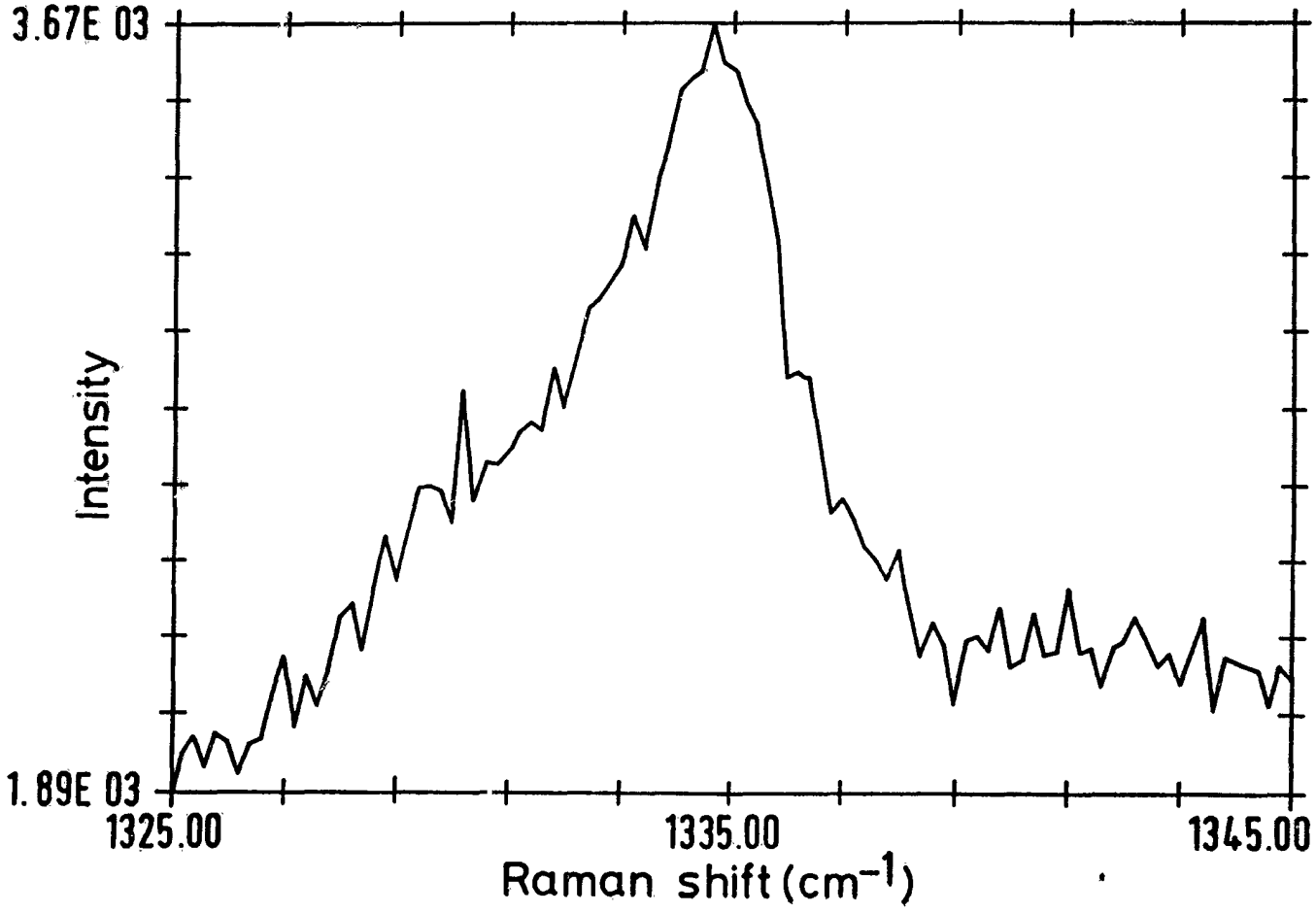

3.67E 03
1.89E 03
Intensity
1325.00
1335.00
1345.00
Raman shift (cm^{-1})

FIG. 3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1.89E 03
Intensity
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1335.00
1345.00
Raman shift (cm^{-1})

FIG. 4

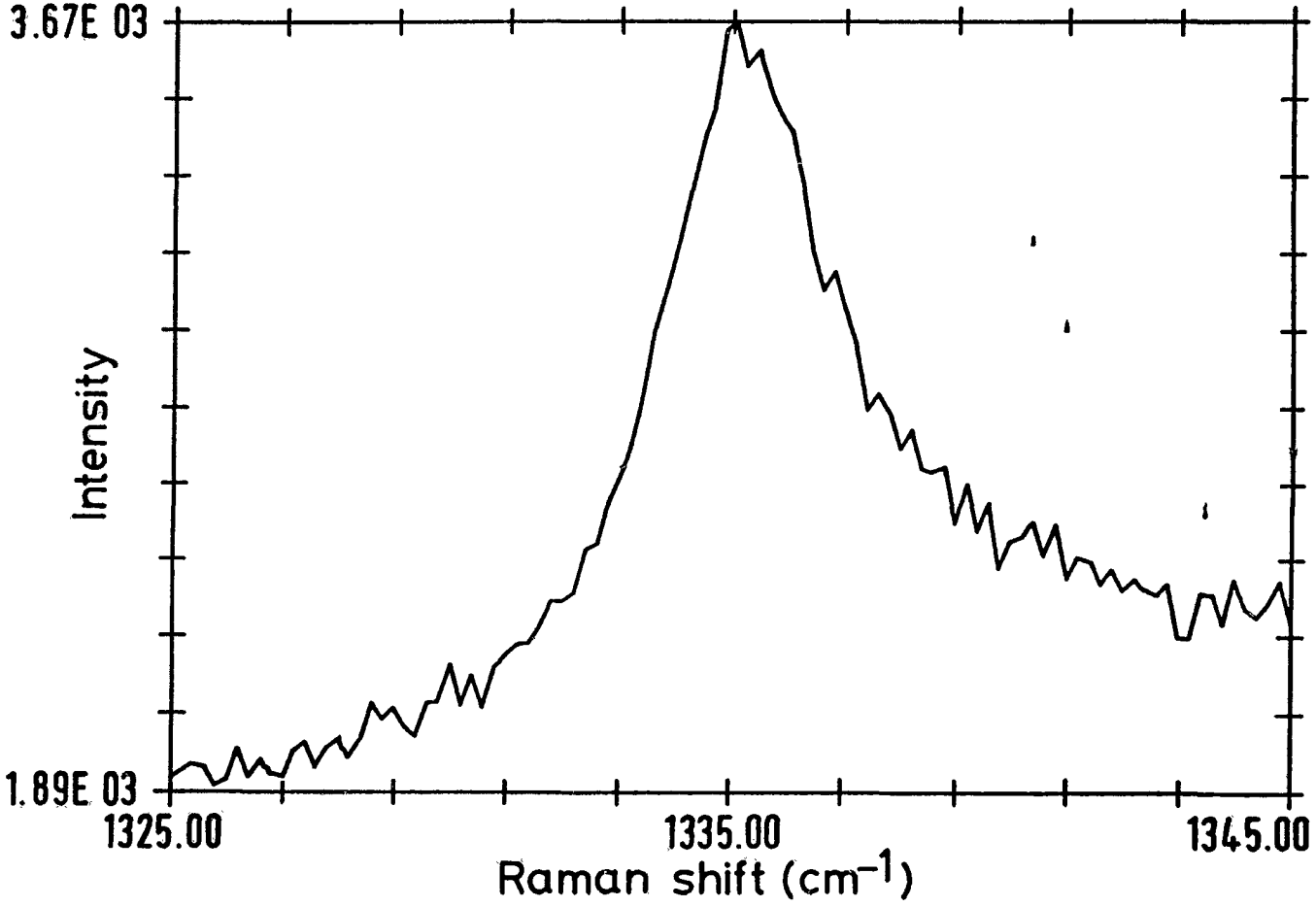

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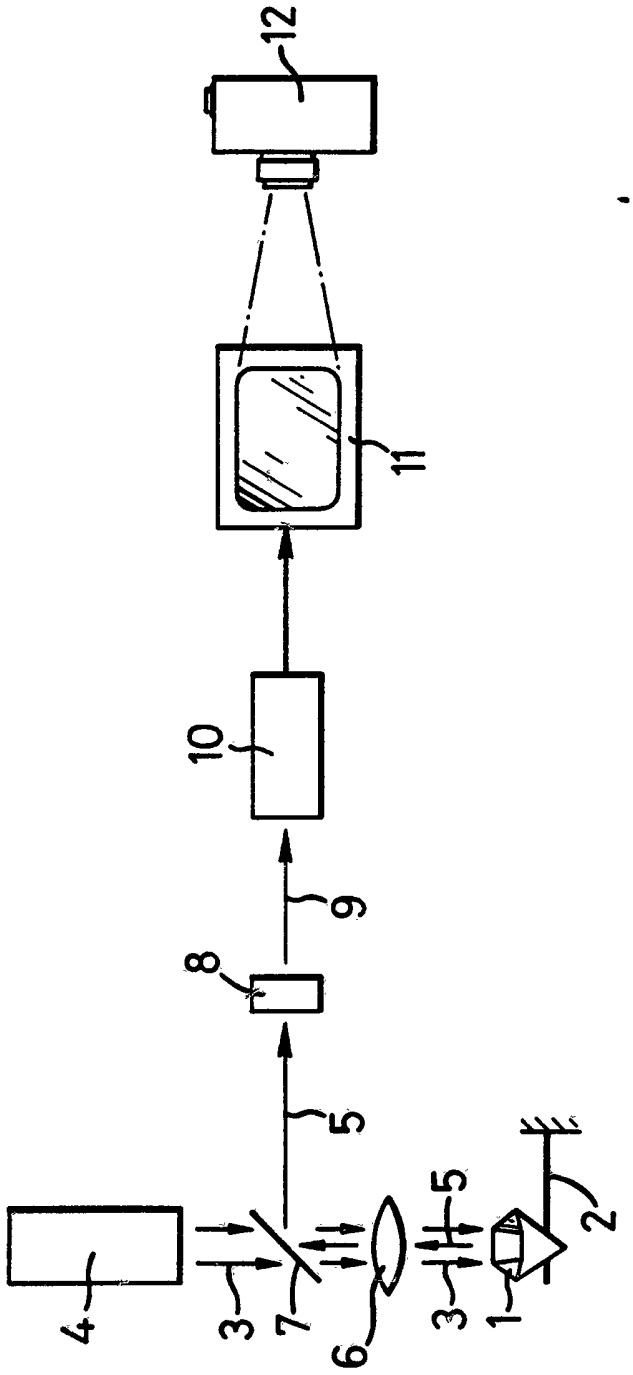
FIG. 5



INTERNATIONAL SEARCH REPORT

International Application No PCT/GB 88/00188

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [6]

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC[4]: G 01 N 21/65, G 01 N 21/87

II. FIELDS SEARCHED

Minimum Documentation Searched [7]

Classification System	Classification Symbols
IPC[4]	G 01 N, G 01 J

Documentation Searched other than Minimum Documentation to the Extent that such Documents are included in the Fields Searched [8]

III. DOCUMENTS CONSIDERED TO BE RELEVANT [9]

Category *	Citation of Document, [11] with indication, where appropriate, of the relevant passages [12]	Relevant to Claim No. [13]
P,X	WO, A, 87/03963 (THE BRITISH PETROLEUM CO.) 2 July 1987, see claims 1-14 --	1-3
A	FR, A, 2496888 (GEMOLOGICAL LABORATORY OF ANTWERP) 25 June 1982, see claims 1-4; page 8, lines 17-22 --	1,8,10
A	Physical Review B, vol. 1, no. 4, 15 February 1970, (New York, US) S.A. Solin et al.: "Raman spectrum of diamond" pages 1687-1698 --	
A	US, A, 3947120 (C. BAR-ISSAC) 30 March 1976, see columns 5,6 --	1,4,8,10
A	WO, A, 86/07457 (THE BRITISH PETROLEUM CO.) 18 December 1986, see claims 1-13 --	1,8
A	US, A, 4030827 (M. DELHAYE et al.) 21 June 1977	

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* Special categories of cited documents: [10]

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published 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; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
27th May 1988	- 8 JUL 1988
International Searching Authority	**Signature of Authorized Officer**
EUROPEAN PATENT OFFICE	P.C.G. VAN DER PUTTEN

Form PCT/ISA/210 (second sheet) (January 1985)

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO. GB 8800188

SA 21148

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
The members are as contained in the European Patent Office EDP file on 20/06/88
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

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WO-A- 8703963	02-07-87	AU-A-	6778987	15-07-87
		EP-A-	0250527	07-01-88
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