Available online at www.sciencedirect.com
ScienceDirect
Int. J. Miner. Process. 84 (2007) 262–273
INTERNATIONAL JOURNAL OF MINERAL PROCESSING
www.elsevier.com/locate/ijminpro

Detection of diamond in ore using pulsed laser Raman spectroscopy

G.H. Lamprecht [a,*], H.G.C. Human [b], L.W. Snyman [c]

[a] De Beers Consolidated Mines, P.O. Box 82851, Southdale 2135, South Africa
[b] National Laser Centre, Council for Scientific and Industrial Research, P.O. Box 395, Pretoria 0001, South Africa
[c] Department Electrical Engineering, Tshwane University of Technology, Private Bag X680, Pretoria 0001, South Africa

Received 14 September 2006; received in revised form 20 June 2007; accepted 20 June 2007
Available online 29 June 2007

Abstract

The viability of using pulsed laser excited Raman spectroscopy as a method for diamond detection from ore, has been investigated. In this method the spontaneous Stokes Raman signal is used as indicator of diamond, and a dual channel system is necessary for correcting for fluorescence of minerals and diamond itself. Various pulsed laser wavelengths from 266 to 1064nm were used, as well as cw lasers for comparison. Wavelength scans of the regions of interest, indicated that pulsed lasers at 532, 355 and 308nm may be used with confidence for this purpose. Mineral fluorescence did not appear to pose a threat to the method, but rather own fluorescence of some types of diamonds. In this respect, pulsed lasers offer a decided advantage above cw, due to non-linear increase of fluorescence with laser power, resulting in superior Raman to fluorescence signal ratios. An apparatus constructed for discriminating diamond from ore was evaluated, and using minerals commonly occurring in diamond carrying ore as well as a wide variety of diamonds, it proved to function effectively. A significant improvement in the capability for diamond detection was found when pulsed lasers at 532 and 308nm were used, in comparison to the 532nm cw laser.

Keywords: Diamonds; Sorting methods; Mineral processing; Raman spectroscopy; Laser excitation

1. Introduction

The method employing the use of the spontaneous Raman signal of diamond (when irradiated with a laser) for extraction of diamond from the diamond carrying ore, seems to offer increased efficiency of extraction over existing methods like the X-ray luminescence method. Various patents have been registered covering the use of lasers for this purpose (see e.g. Bowley, 1990; Donald, 1989; Venter, 1996), while a single research paper has been published (Gudaev et al., 1998), describing the use of a He–Ne laser at 632.8nm for detecting a wide variety of diamonds from a selection of minerals typically present in diamondiferous ore. In all cases the principle of detection employed is the measurement of the spontaneous Raman signal through a narrowband interference filter (typically 0.5 to 1nm) centred on the Raman wavelength, and the simultaneous measurement, on a separate channel, of the emitted radiation through a wide band filter, centred on the Raman wavelength or adjacent to it. With illumination of a diamond by the laser, the channel measuring the Raman signal through the narrowband filter should show an increase relative to the other channel. With illumination of a mineral, no relative increase of one channel above the other will be registered if the luminescence of the mineral is of

* Corresponding author. Tel.: +27 11 3746033; fax: +27 11 3093429.
E-mail address: gert.lamprecht@debeersgroup.com (G.H. Lamprecht).

0301-7516/$ - see front matter © 2007 Published by Elsevier B.V.
doi:10.1016/j.minpro.2007.06.002

continuum nature over the wavelength interval of the wideband filter. Regarding the diamond's own fluorescence, it is required that it also is a continuum, and not too strong in relation to the Raman emission.

The purpose of the work reported here was to investigate, over the wavelength range 266 to 1064nm, the Raman signal strength in relation to the luminescence signals of typical minerals found in diamondiferous ore, as well as it's strength in comparison with the luminescence of diamond itself. Pulsed lasers at 266, 308, 355, 532 and 1064nm were used, as well as cw lasers at 488, 532 and 632nm. Aspects of the spectroscopy that could possibly aid in the discriminating system were investigated, such as the temporal characteristics of the signals, and the linearity of the signals with laser power. The relative signal to noise ratios expected with pulsed and cw systems was also investigated theoretically and experimentally. The measurements were done in order to arrive at an informed decision regarding the potential of pulsed lasers as sources for diamond detection, the best wavelength for use, and its relative merits compared to cw sources, thus the inclusion of cw sources in the investigation. Paragraphs 3 and 4 give these results.

The following part of this report (Section 5) describes the evaluation of an experimental apparatus constructed for discriminating diamond from ore, using the more promising wavelengths indicated in the preliminary part, also including a cw wavelength for comparison. As for the first part, a large variety of diamonds were used, with particular attention paid to the statistical variations involved.

2. Experimental

2.1. Wavelength scanning measurements

For scanning the luminescence and Raman spectra of the diamonds and minerals, a Kratos 0.25m double monochromator was used to minimize stray light falling on the detector as a result of large amounts of laser reflected light entering the first monochromator. Samples were positioned on the optical axis so that the luminescence could be focused on the entrance slit (0.3mm wide, resulting in a spectral bandwidth of approximately 1nm) of the monochromator by a fused silica lens of 100mm focal length. The laser beam was made incident on the samples with an angle of approximately 30° with respect to the optical axis. The radiaton detector used for all wavelengths except for that exceeding 1064nm, was a Hamamatsu R1477 photomultiplier tube, the output of which was fed directly to a digital oscilloscope (Tektronix 360) in the case of cw measurements. The oscilloscope was connected to a PC with the aid of a GPIB to USB interfacing cable and its associated software, allowing measurements to be registered at a selectable interval. For the pulsed laser measurements, it was necessary to use a modified PMT base to handle the high instantaneous flux of photons, together with a fast response amplifier (Burr Brown model 3554) built into the PMT housing to minimize capacitance between source and amplifier. The modification to the base entailed a resistor chain with 270kΩ across the first dynode and cathode to ensure at least a 150V supply between them. Capacitors of 10nF were placed across each of the last four dynodes (out of a total of 9). The last three resistors of the resistor chain were 150kΩ, 180kΩ and 220kΩ respectively, in comparison with the 100kΩ resistor between the rest of the dynodes, to prevent saturation of the detector and maintain a linear response. Calibrated neutral density filters were used to check linearity of response. For detection of the luminescent radiation above 1064nm, a Hamamatsu G8370 InGaAs PIN photodiode was used, and the gratings of the monochromator replaced with gratings suitable for this region.

For the pulsed laser wavelengths 1064, 532, 355 and 266nm, a flashlamp pumped Nd:YAG (Continuum, Powerlite 9010, with maximum pulse energy of 1.4J at 1064nm and with a fixed repetition rate of 10Hz and pulse length ~ 7–9ns) with the appropriate frequency conversion crystals was used. For the pulsed wavelength 308nm, an excimer laser from Lambda Physik, model 203 MSC, with 300mJ pulse energy, using XeCl gas, with a maximum repetition rate of 250Hz (pulse length ~ 25ns) was used.

The 532nm cw laser was a diode pumped 5Watt water cooled laser from Spectra Physics (model Milennia). The 488nm cw laser was a line tunable argon ion laser from Spectra Physics (model 2020), supplying approximately 2W. The cw 632.8nm wavelength was supplied by a Spectra Physics model 127 He–Ne laser at approximately 25mW.

A selection of 18 minerals, listed in Table 1, commonly occurring in diamondiferous ore were used to compare its luminescence spectra with that of diamond. The ceramic is included since it is often present in the ore as an added impurity, due to its use in the processing system as liners to prevent wear.

For the wide wavelength region scans initially performed, four diamonds of type Ia, Ib, IIa and IIb were used. For the scans across a narrow wavelength region adjacent to both sides of the Raman shifted wavelength for diamond, a set of 203 diamonds was available, including a wide variety of types. Each of these stones is individually classified according to colour,