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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 : G01N 21/71

A1

(11) International Publication Number: WO 99/45368

(43) International Publication Date: 10 September 1999 (10.09.99)

(21) International Application Number: PCT/US99/04756

(22) International Filing Date: 3 March 1999 (03.03.99)

(30) Priority Data:
60/076,639 3 March 1998 (03.03.98) US

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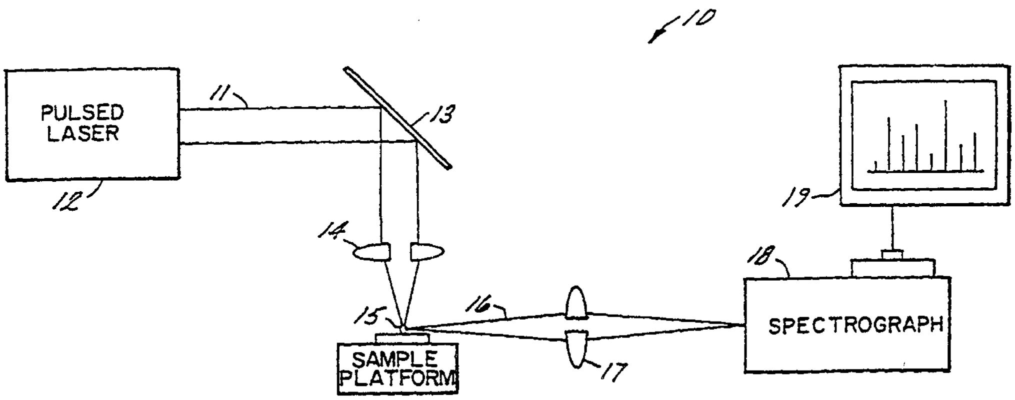
(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

Published
With international search report.

(54) Title: IMPROVED LASER SPECTRAL ANALYZER WITH SAMPLE LOCATION DETECTOR



(57) Abstract

A laser spectral analyzer is presented for analyzing the composition characteristics of a stream of flowable material. The analyzer includes a laser spectroscopy sensor which emits a laser beam at a predetermined focal area to excite the electrons of the material and a detector for receiving and a processor for identifying peaks from the spectra given off by the excited electrons. The analyzer further includes a sample location detection system for determining the position of the sample relative to the laser spectroscopy sensor. The detection system controls the triggering of the light source to only when the sample surface is within predetermined limits about the focal area of the sensor. In alternative embodiments the detection system includes circuitry which directs the detector to disregard electrons or directs the processor not to identify spectra from a sample determined to outside of the predetermined limits of the sensor. In other alternative embodiments the analyzer is included in an industrial manufacturing process having a continuous flow of material to detect the position of an upper surface of the material and control the system.

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IMPROVED LASER SPECTRAL ANALYZER WITH SAMPLE LOCATION DETECTOR

Background of the Invention:

1. Field of the Invention

This invention relates generally to sensors used in connection with equipment (i.e., froth flotation machines) for the separation of particles from a liquid slurry or pulp. More particularly, this invention relates to improved methods and apparatus for automatically monitoring, operating, and controlling continuous feed process equipment using remote sensing devices, particularly laser spectroscopy type sensing devices.

2. Brief Description of the Prior Art

Flotation machines are used in many industrial applications for separation of particulate materials from suspensions in a liquid, usually water. The particles to be removed from the suspension are treated with reagents to render them hydrophobic or water repellant, and a gas, usually air, is admitted to the suspension in the form of small bubbles. The hydrophobic particles come into contact with the bubbles and adhere to them, rising with them to the surface of the liquid to form a froth. The froth containing the floated particles is then removed as the concentrate or product, while any hydrophilic particles are left behind in the liquid phase and pass out as the tailings. Flotation machines find particular utility in the metals recovery industry, providing superior recovery of metallic minerals from a solid/liquid mixture known as a "pulp," "slurry," or "feed". The flotation process can also be applied to the removal of oil droplets or emulsified oil particles, as well as to fibrous or vegetable matter such as paper fibers, bacterial cells, and the like.

Efficient and effective operation of flotation machines requires monitoring and controlling a multitude of process parameters such as slurry levels and bubble size, as described above. Other process parameters include but are not limited to the density of the pulp in the chamber of the flotation machine, bubble concentration and distribution, product and tailings removal rates, reagent addition and consumption rates, air flow, solids concentration, froth mass and volume, froth level, pulp level, feed rate, and the like. These classifications and examples are for convenience and example only.

Simple controllers have been used to measure and adjust the density of feed or pulp. For example, U.S. Patent No. 5,368,166 to Chumak, et al., discloses a control device to measure the level and density of pulp and to control the flow rate of water and frothing agent. A differential densitometer for continuously measuring total undissolved solids in a liquid in U.S. Patient No. 5,417,102 to Prevost.

Another application of a prior art control system is disclosed in U.S. Patent application 756,713 commonly assigned to applicant and is fully incorporated herein by reference in this application, the sensor or sensors comprise a means for determining input stream composition and particle size. Preferably, the sensor provides the data regarding the input stream without the necessity of removing samples from the process flow for analysis at a separate location.

It will be appreciated that it is often difficult to sense and communicate certain parameters in real time within flotation and similar machines. Thus, a variety of technologies including ultrasonic absorption and reflection, laser-heated cavity spectroscopy, laser-induced breakdown spectroscopy (LIBS), laser-induced mass spectroscopy (LIMS), X-ray fluorescence spectroscopy, neutron activation spectroscopy, pressure measurement, microwave or millimeter wave radar reflectance or absorption, and other optical and acoustic methods have been used in the prior art. An example of a suitable apparatus for sensing using LIBS is disclosed in U.S. Patent No. 5,379,103 (Ziegler), all of the contents of which are incorporated herein by reference. An example of a specific apparatus for sensing LIMS is the LASMA Laser Mass Analyzer available from Advanced Power Technologies, Inc., of Washington, D.C.

An example of a LIBS system for analysis of a multiple constituent sample is shown schematically in FIGURE 1, labeled as prior art, wherein the system is shown generally as 10. A beam 11 from a pulsed laser 12 is directed by a system of optical components, which may include a mirror 13 and a focusing lens 14, at a sample 15. The pulsed laser beam 12 may be directed at the sample b movement of the laser or of one or more of the optical components. The beam may impinge on the sample at a single point, or it may move in any required path to provide a profile of the process stream without the necessity of removing individual samples from the process stream. The sample is converted to a plasma state by the energy of the beam which gives off atomic emissions 16 in the ultraviolet and visible range. These emissions are focused by lens 17 to a spectrograph 18. Spectrograph 18 separates the emissions into wavelengths and their corresponding intensities which are in turn plotted on a display 19. The wavelengths of the emissions are known to correspond to specific elements which makes the identification of the constituents of sample 15 straightforward. The LIBS system described typically utilizes a neodymium-YAG laser, which heats a small volume of sample within a volume of a few cubic millimeters to temperatures as high as 100,000 degrees Kelvin. The analysis of the sample is completed very quickly, typically within one to five seconds, depending on the application. Analysis of samples using the LIBS system yields high sensitivities as depicted in FIGURE 2, labeled prior art, wherein the detection of the element beryllium included in sample of water is represented by spike 20 wherein the concentration of beryllium is only one part per million.

Control systems of the prior art which include the use of one or more LIBS or LIMS sensors provide for the determination of elemental composition *in situ*, that is, without the need for removal of a sample for analysis at a separate location. The use of LIBS and LIMS sensors in this manner allows fast, discrete, real-time analysis. When used in conjunction with a controller a control device is actuated in response to the data received from the LIBS sensor and an internal process model to affect changes to the operational parameters of the processing system containing a multi-component mixture.

LIBS sensors are utilized in the prior art as described above for determining elemental composition in essentially dry or dewatered solids or froths. Other prior art processing systems also include using LIBS sensors in processing systems which have sample streams which do not need to be dried or dewatered, including, but not limited to, thickeners, filters, centrifuges, analysis of the molten metal or slag streams of smelting furnaces, chemical process solutions, and the like, for example, crushed ore which is conditioned by adding reagents and then is subjected to froth flotation in at least one flotation machine for separation. A LIBS sensor analyzes the composition of one or more constituents of the crushed ore and communicates these data to an intelligent controller. The controller then send signals to a grinding apparatus or the reagent addition system to make adjustments affecting the operational parameters of the froth flotation machine.

In still other prior art applications, the LIBS sensor performs an analysis of a few key elements in dry or dewatered samples, for example the concentration of copper, molybdenum, iron, silica, and magnesium in copper flotation concentrates. The LIBS sensor is positioned close to the froth overflow of a conventional froth flotation machine to avoid errors. There is a need for a LIBS sensor which incorporates ruggedized optics to allow operation on or near process streams, providing tolerance for vibration, dust, and moisture.

One of the challenges in utilizing a laser spectral analyzer in the prior art is the caused by the difficulty of positioning a sample within the relatively short focal plane of the laser. A typical laser spectral analyzer has a focal plane of +/- 0.5 mm. As described herein above a laser spectral analyzer is used frequently to characterize the constituents of a stream of flowing slurry. The stream surface is typically comprised of turbulent flow and as such the surface of the stream fluctuates in and out of the focal plane of the laser. Numerous attempts to eliminate the turbulent nature of the stream, variations in sample port design for example, have proven to be ineffective. In fact, in current configurations a suitable sample is only acquired in one out of every three attempts.

There are several other industrial processes wherein laser spectral analysis is advantageous for determining the composition, or other characteristics, of a moving sample. Examples of these processes include a sheet metal rolling mill, a continuous casting machine, a textile fiber extrusion machine, etc. These processes have shortcomings similar to those described herein above with respect to sample presentation.

Summary of the Invention:

The above-discussed and other problems and deficiencies of the prior art are overcome or alleviated by using a laser spectral analyzer with a sample positioning detection system in accordance with the present invention.

In accordance with the present invention a detection system is provided to accurately determine the position of an outer surface of a slurry sample relative to a sensor of a laser spectral analyzer. In a particular embodiment of the present invention the laser and sensor of a laser spectral analyzer are focused about a focal area and may accurately characterize a material only within a small limit about the focal area. Samples are presented in a flowable slurry stream having a turbulent, and therefore undulating, outer surface causing the sample to move in and out of the limits of the analyzer. When the slurry stream is within the limits of the analyzer a laser beam excites the slurry producing a sample of excited electrons, a detector receives a spectrum given off by the electrons, and a processor identifies peaks from the spectra which are associated with compositional characteristics of the slurry.

In accordance with the present invention a range finding detector is provided to accurately determine when the outer surface of the slurry is within the focal range of the analyzer. In a particularly preferred embodiment a range-finding laser is utilized wherein a beam is directed from the laser to the stream and is reflected back to the range-finding laser. The range-finding laser compares the detected distance to predetermined limits and provides a triggering signal to either a separate controller or directly to the laser spectral analyzer to fire the laser and obtain a sample from the stream.

In an embodiment of the present invention the time constant related to the outer surface of the slurry stream due to turbulence is on the order of 10^{-3} seconds and the time constant of the firing control of the laser spectral analyzer is on the order of 10^{-6}. The difference in the time constants ensures that the laser's firing can be triggered quickly enough to fire while the slurry stream is within the focal range of the analyzer, which for example is approximately +/- 0.5 mm about a focal plane. In an alternative embodiment the range finder directs the detector of the analyzer to disregard spectra produced by electrons which are excited by the firing of the laser while the stream is determined to be outside of the limits. In yet another embodiment the range finder directs the processor not to identify spectra form a sample when it is determined that the sample was taken while the outer surface of the slurry stream was outside of the limits of the analyzer.

Brief Description of the Drawings:

Referring now to the drawings wherein like elements are numbered alike in the several FIGURES:

FIGURE 1 is a schematic representation of a laser analysis system of the prior art;

FIGURE 2 is a graphical representation of an output plot of a laser analysis system of the prior art;

FIGURE 3 is a schematic diagram of a laser spectral analyzer incorporating a sample detection system in accordance with the present invention;

FIGURE 4 is an enlarged schematic showing the slurry stream and the sample detection system of detail 4 in FIGURE 3; and

FIGURE 5 is a schematic representation of a laser spectral analyzer incorporating a sample detection system in accordance with the present invention for a continuous manufacturing process.

Description of the Preferred Embodiment:

Referring to FIGURE 3 a laser spectral analyzing system is shown generally at 30 incorporating a laser firing control of the present invention. A high-energy laser 35 controlled by controller 36 to emit laser beam 37 toward upper surface of a sample 32, which emits a spectrum 33 that is captured by a spectrograph 34 and further receives emissions given off from the energized sample (not shown). Analyzer 30 further includes range-finding laser 38 which produces an output signal 39 directed toward upper surface of sample 32 and upon contacting upper surface feedback signal 40 is reflected back to range finder 38. In an embodiment, sample

32 may be a slurry stream. Range finder 38 compares output signal 39 to feedback signal 40 to accurately determine the distance of upper surface 33 relative to sensor system 35. Range finder 38 produces an electrical signal to controller 36 triggering laser 41 to emit laser beam 37 when upper surface of sample 32 is ascertained to be within a predetermined distance from analyzer system 35.

With reference to FIGURE 4 the turbulence of slurry is shown by the variation in height of upper surface 33 from, for example a high of point represented by dot 42 and a low point represented by dot 43. As described herein above laser 41 is focused upon a focal plane, represented by line 44, and is able to accurately characterize slurry 32 within an upper limit, represented by dashed line 45, and a lower limit, represented by dashed line 46. In an embodiment the upper limit 45 and the lower limit 46 are each 0.5 mm from focal plane 44. Range finder 38 is capable of accurately determining when the surface 33 of the slurry is within the upper and lower limits by emitting output signal 39 which strikes surface 33 and is reflected back as feedback signal 40. Slurry 32 moves in the direction shown by arrow 47 and upper surface 33 is shown as out of limit, above the upper limit 45. As slurry stream 32 progresses it will be within limits 45, 46 when the portion between points represented by dots 48, 49 pass under signal 39. When the upper surface 33 is within the upper and lower limits 45, 46 a signal is sent to controller to trigger laser 41 to fire and produce beam 37. Beam 37 turns the upper surface 33 of the slurry into a plasma which allows for analysis of the slurry. In an alternative embodiment range finder 38 sends a triggering signal directly to laser 41 to produce beam 37. Laser beam 37 and range finder output signal 39 are shown as separated by a distance in FIGURES 3 and 4 for illustration purposes only. An embodiment of the present invention positions the two signals within a very small distance, 0.1 to 0.25 mm. Another embodiment allows the two signals to be co-linear, thus impinging on the sample surface at the same location.

The range finder may be any one of several devices known in the art including a range-finding laser, ultrasound, radar or other suitable range finding technique. The advantage of the present invention over the prior art is the ability of range finder 38 to quickly and accurately establish that the upper surface 33 is positioned within the limits 45, 46 of the laser spectral analyzer 35. The slurry stream turbulence for a particular embodiment undulates in and out of the laser spectral analyzer 35 focal limits 45, 46 at a frequency of about 10^{-3} seconds. An embodiment of the present invention includes a helium-neon laser range finder which can establish the position of the upper surface 33 at a frequency of about 10^{-6} seconds. The rapid acquisition of the position of upper surface 33 by range finder 38 ensures that laser 41 will only be triggered when the upper surface is within the limits 45, 46 of the laser spectral analyzer leading to a more accurate analysis of slurry 32 and less acquisition errors for the laser spectral analyzer.

While the present invention has been described in conjunction with froth flotation machines, it will be appreciated that many of the sensing, monitoring and control techniques and instrumentation may be used in connection with any processing system for a multi component mixture.

With reference to FIGURE 6 there is shown an embodiment of the present invention directed toward a number industrial manufacturing processes where characterization of a moving sample is particularly advantageous. The Basic laser spectral analyzing system is similar to that described herein above with reference to FIGURE 3. The embodiment of FIGURE 6 is a rolling mill of sheet metal production process including rollers 60, 61 and producing sheet steel 62. Steel sheet 62 flows in a continuous fashion from a rolling mill in the direction indicated by arrow 63 as is well known. It is advantageous to sample certain characteristics of sheet 62 in real time while the process continues. Surface 64 of sheet 62 may vary by more than the tolerance of laser spectral analyzer 30. As described herein above range finding system 38 works in conjunction with computer controller 36 to control the firing of laser 35 and/or data acquisition of system 30 to points in time where surface 64 is within the operating limits of the system. It is contemplated by the present invention that system 30 is included in other types of manufacturing processes similar to those described herein above. Examples of these processes include, but are not limited to, a continuous casting machine, a textile fiber extrusion machine, and the like.

While preferred embodiments have been shown and described, various modifications and substitutions may be made thereto without departing from the spirit and scope of the invention. Accordingly, it is to be understood that the present invention has been described by way of illustrations and not limitation.

What is claimed is:

CLAIM 1 A laser spectral analyzer analyzing composition characteristics of a stream of material moving past the analyzer, the analyzer comprising:

a laser spectroscopy sensor having a light source emitting flashes of high energy light to a predetermined focal space and exciting electrons of material illuminated generally in the focal space;

a detector detecting the spectra of the excited electrons;

a processor receiving data from the detector and identifying peaks from the spectra associated with composition characteristics of the material;

a sample location detection system determining the position of an outer surface of the sample, and

circuitry receiving data relative to the position of the sample surface and controlling the operation of the analyzer to eliminate errors in detection arising when the outer surface of the sample is outside of the focal space.

CLAIM 2 The analyzer of claim 1 wherein the circuitry controls the light source so as not to emit a flash of light when the outer surface of the sample is outside of the focal space.

CLAIM 3 The analyzer of claim 1 wherein the circuitry directs the detector to disregard excited electrons from the sample when the outer surface of the sample is outside of the focal space.

CLAIM 4 The analyzer of claim 1 wherein the circuitry directs the processor not to identify spectra from the sample when the outer surface of the sample is outside of the focal space.

CLAIM 5. The analyzer of claim 1 wherein the sample detection is comprised of a range-finding laser, an infrared range finder or an ultrasonic range finder.

CLAIM 6. A sample location detection system for use with a laser spectral analyzer having a predetermined operating range and analyzing composition characteristics of a material, the sample location detection system comprising a range finder having a sensor sensing a position of an outer surface the material.

CLAIM 7. The sample location system in claim 6 wherein the range finder produces data relative to the position of the material, the sample location system further comprises circuitry analyzing the sensed position and a predetermined operating range of the analyzer.

CLAIM 8. The sample location system in claim 6 wherein the range finder produces data relative to the position of the material, wherein the circuitry produces signals representative of in range and out of range determinations; and

a controller receiving the signals and controlling the operation of the analyzer with the controller enabling the analyzer in response to the in range condition and disabling the analyzer in response to an out of range condition.

CLAIM 9. The sample location system 6 wherein the range finder comprises a range finding laser, an infrared range finder, an ultrasonic range finder, or a radar range finder.

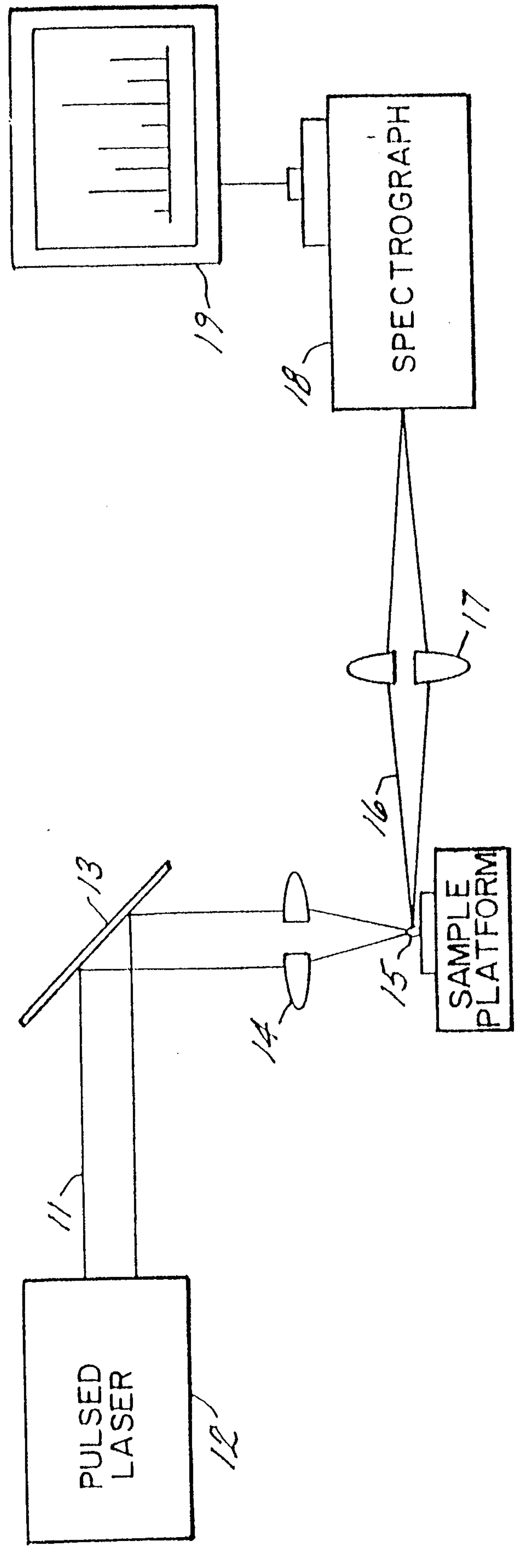


FIG. 1
(PRIOR ART)

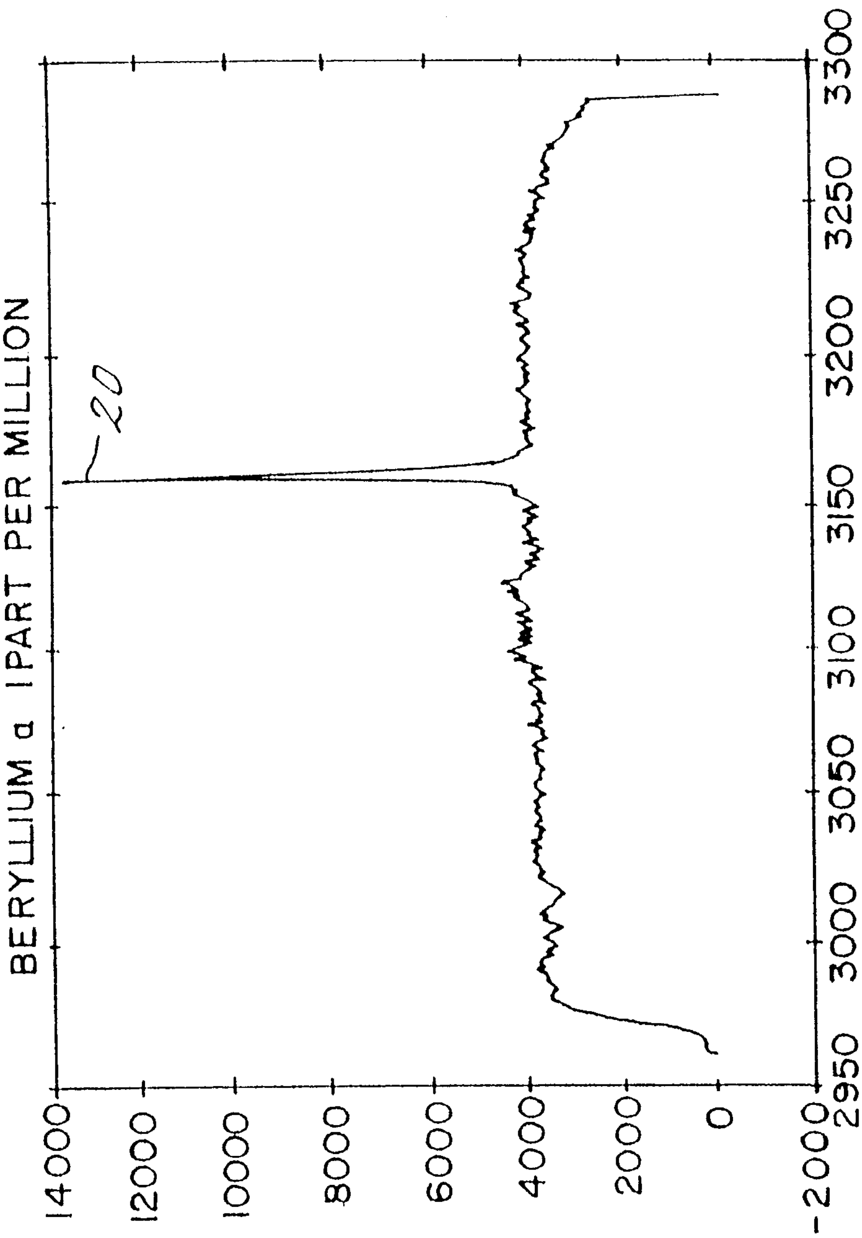
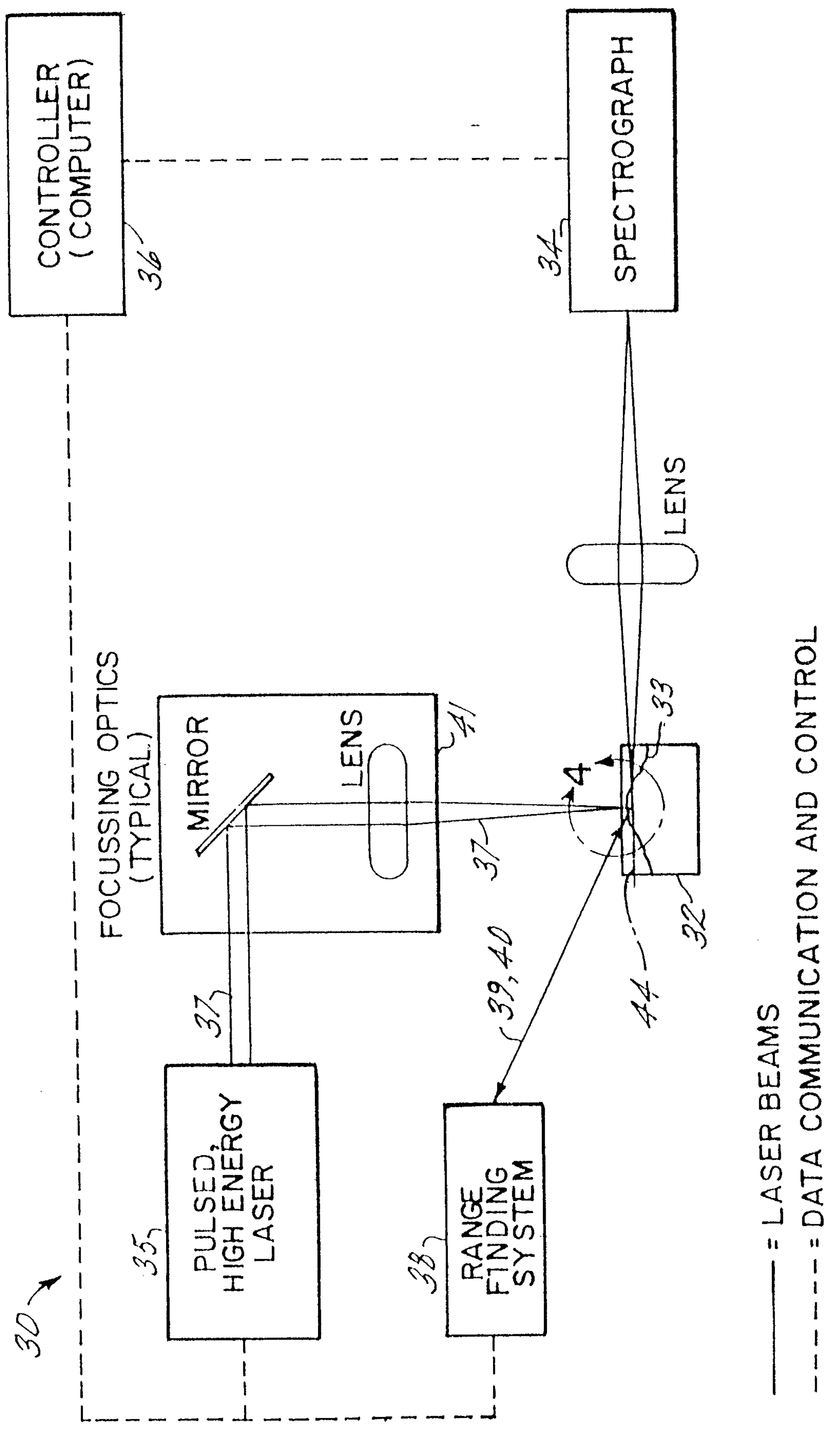


FIG. 2
(PRIOR ART)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LENS
FOCUSSING OPTICS (TYPICAL)
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LENS
41
33
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37
32
44
39, 40
37
PULSED, HIGH ENERGY LASER
35
RANGE FINDING SYSTEM
38
30
= LASER BEAMS
= DATA COMMUNICATION AND CONTROL

FIG. 3

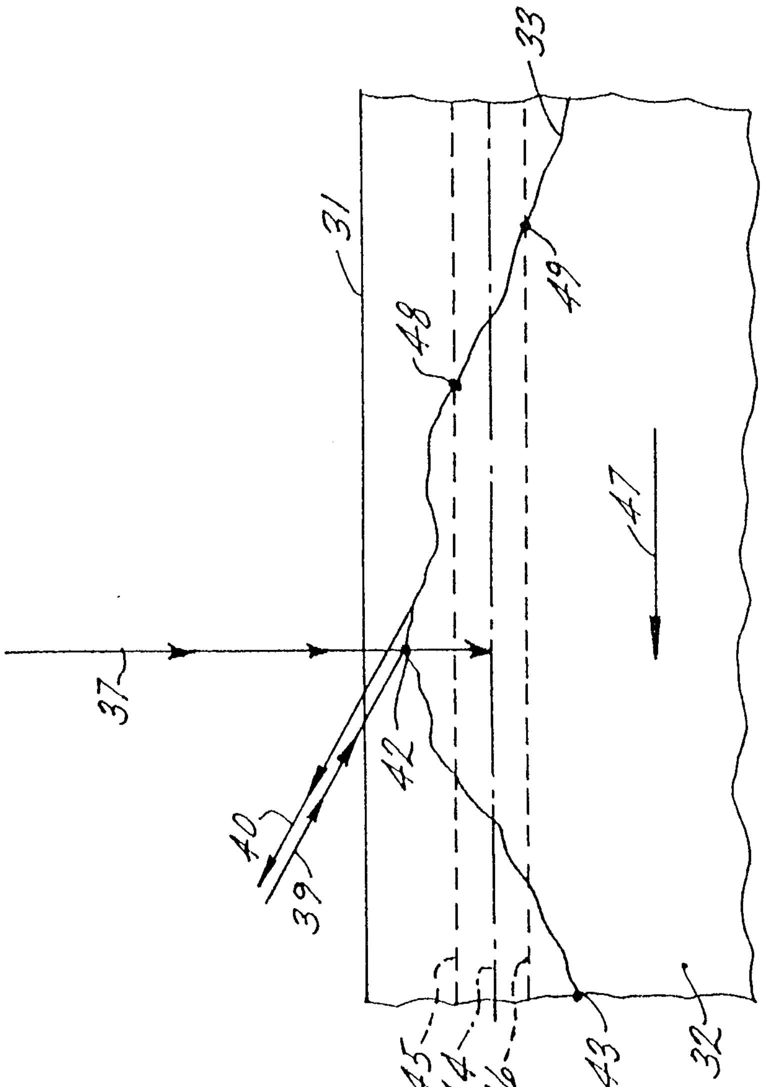
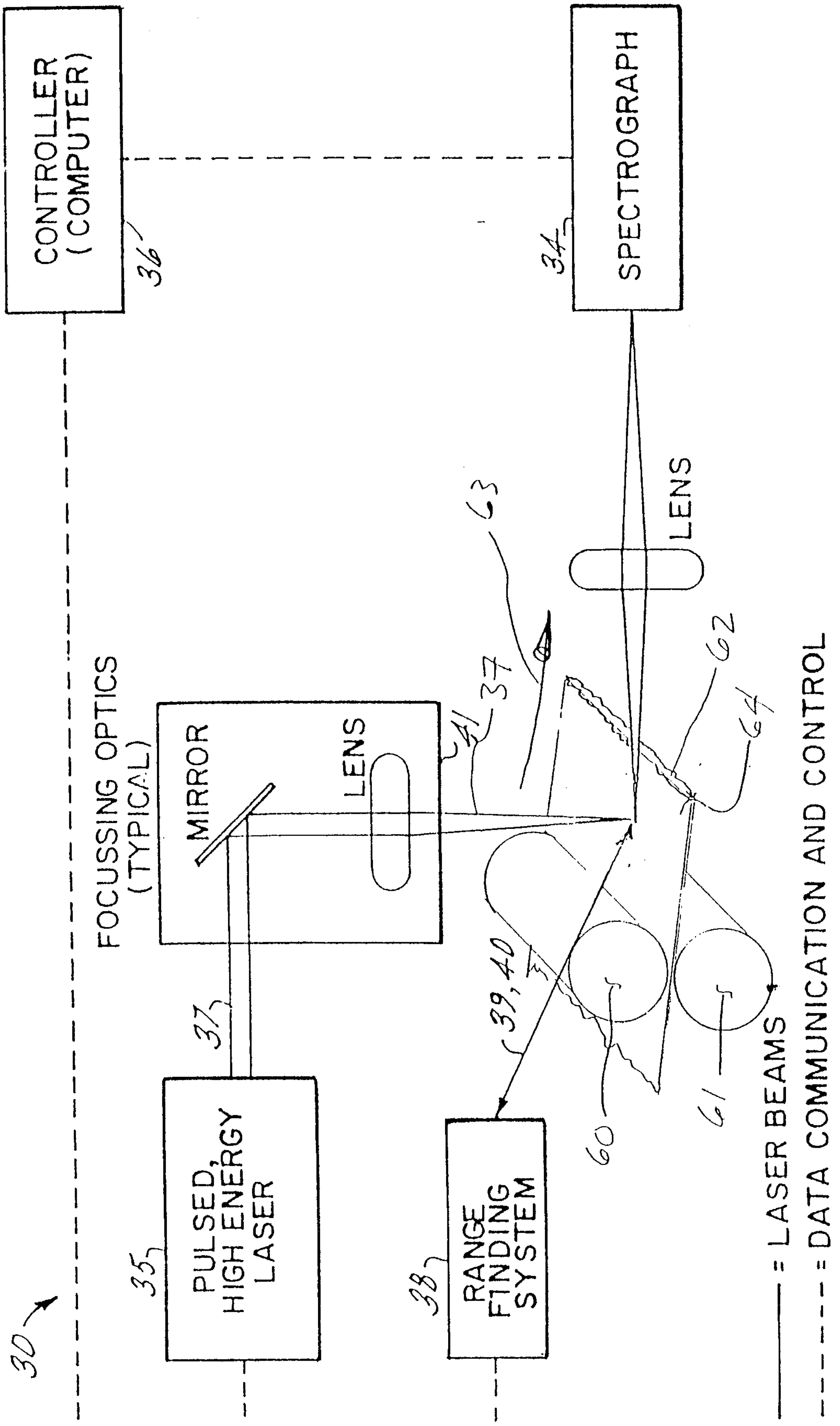


FIG. 4


CONTROLLER (COMPUTER)
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LENS
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64
FOCUSSING OPTICS (TYPICAL)
MIRROR
LENS
41
37
39, 40
PULSED, HIGH ENERGY LASER
35
RANGE FINDING SYSTEM
38
60
61
30
= LASER BEAMS
= DATA COMMUNICATION AND CONTROL

FIG. 5

INTERNATIONAL SEARCH REPORT

In :ional Application No
PCT/US 99/04756

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 G01N21/71

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

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Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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X	US 4 652 128 A (TSUNOYAMA KOUZOU ET AL) 24 March 1987 see figure 1	1-9
A	EP 0 362 577 A (KRUPP GMBH) 11 April 1990 see figure 1	1-9

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
25 May 1999	17/06/1999

Name and mailing address of the ISA	Authorized officer
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INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 99/04756

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
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2

Form PCT/ISA/210 (continuation of second sheet) (July 1992)

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Information on patent family members

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