

This document was produced
by scanning the original publication.

Ce document est le produit d'une
numérisation par balayage
de la publication originale.

No. _____

CANADA

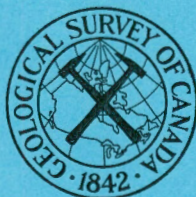
DEPARTMENT OF MINES AND TECHNICAL SURVEYS

GEOLOGICAL SURVEY OF CANADA
TOPICAL REPORT NO. 74

74
74

THE USE OF THE OPTICAL MASER (LASER)
AS A TOOL IN THE STUDY OF
GEOLOGICAL MATERIALS

BY
J. A. MAXWELL



OTTAWA
1963

CANADA
DEPARTMENT OF MINES AND TECHNICAL SURVEYS

GEOLOGICAL SURVEY OF CANADA

TOPICAL REPORT NO. 74

THE USE OF THE OPTICAL MASER (LASER) AS A
TOOL IN THE STUDY OF GEOLOGICAL MATERIALS

by
J. A. Maxwell

OTTAWA
1963

TABLE OF CONTENTS

	Page
Summary	1
Introduction	3
Laser Theory	4
Operational Details	7
Procedure	8
Accuracy of Aim of the Laser Beam	8
Nature of Sample Surface	9
Thickness of the Sample	9
Sensitivity and Reproducibility	10
Nature of the Spectra	10
Experimental Results	11
Polished Sections	11
Rock Slices	13
Thin Section	15
Conclusions	15
Selected Bibliography	19
Appendix	21
Description of Equipment	21
The Laser Head	21
The Microscope	22
The Cross-excitation Electrodes	23
The Spectrograph	24
The Control Console	25
General Comments	25

SUMMARY

1. A two-step process, in which a ruby laser source is used to vaporize a microgram quantity of selected sample followed by further excitation of the vapour by spark discharge across carbon electrodes, can be used for the qualitative and semiquantitative microanalysis of geological material in situ.
2. The limit of detection of certain elements is reported to be 0.001% for a single shot but a sensitivity of 1 p.p.m. is obtainable. The reproducibility is reported to be +5%.
3. The laser action produces a hemispherical pit in the sample. In routine operation the pit is 100+20 microns in diameter and about 50 microns deep, but pits as small as 50 microns in diameter have been achieved.
4. The equipment is easily attached to the optical bar of any spectrograph or it may be placed at right angles to the bar and the light focused on the slit of the spectrograph by means of a mirror. A fast spectrograph is desirable.
5. A metallurgical microscope, with travelling stage, is used to position the sample and to focus the laser pulse. The sample may be viewed before and after impact.
6. Size and shape of the specimen are not critical but it must be small enough to be accommodated on the microscope stage at a proper distance from the electrodes and it must be more than 100 microns thick.

The roughness of the surface is limited by the fixed distance between the sample surface and the electrodes. A smooth surface facilitates selection of the desired target.

7. Standard thin sections are too thin for use. The laser pulse will burn through the section and vaporize the cement and glass slide.

8. Satisfactory qualitative results have been obtained with polished sections of ore minerals in which good pits were usually formed. The results obtained on non-opaque minerals in rock slices are not as good but show much promise. Shattering and fusion of the area adjacent to the pit (or irregular scar) is particularly noticeable in the silicates.

9. It is reported that satisfactory results have been obtained with alloys, ceramics, glass and powders. Organic material is carbonized and no usable vaporization is obtained with liquids.

10. One advantage of the laser as a source over conventional arc-spark spectrography is that the sample does not have to be electrically conducting. Thus, non-metals do not have to be removed from their matrix.

11. An advantage of the laser source over the electron microprobe is that the optical spectra are not subject to the limitations imposed upon the use of X-rays. The electron microprobe can, however, achieve a much narrower beam, of the order of 1 micron, and better quantitative reproducibility.

12. Further study is necessary of the effect of such factors as the degree of smoothness of surface, hardness of material, crystallographic orientation and the nature of the vapour on the efficiency and reliability of the laser source as an analytical tool for the study of geological materials.

INTRODUCTION

Although in practical application the use of an optical maser (laser) source in emission spectroscopy is less than one year old, it has been hailed as a truly revolutionary new development in this field. By focusing the high energy beam of a ruby laser on a selected small portion of a sample, vaporization of a few micrograms of the material is achieved. Further excitation of this vapour by a conventional spark discharge excites the characteristic spectra of the constituent elements of the vapour and identification of these constituents can be made. It is thus possible to achieve qualitative and semiquantitative microanalysis without the necessity of separating the sample from the matrix prior to the analysis. The name "micro-emission spectroscopy" has been coined to describe this new technique.

The writer was requested to investigate this new technique and this report is a summary of available information on the use of the laser source in emission spectroscopy, based upon published material, discussions with individuals working in this field, upon lectures given at the Eleventh Annual Seminar on Spectroscopy, held at the University of Florida, in Gainesville, January 21-24, 1963, and upon a visit to the laboratories of the Jarrell-Ash Company in Newtonville, Mass., February 6-8, 1963, where selected samples were analyzed with the laser source. Emphasis is placed upon the application of this technique to studies in mineralogy, geochemistry and economic geology, but it has, of course, much wider application.

The writer has benefited from discussions with Dr. B.P. Stoicheff, Pure Physics Division, National Research Council and with Mr. Frederick Brech, Director of Research of the Jarrell-Ash Company. Lectures at the Seminar were given by Professor W.F. Fagen, Department of Electrical Engineering, University of Florida; Dr. T.A. Welton, Oak Ridge National Laboratory, and Mr. Brech. Miss Mary Healy and Mr. John Devaney of the Jarrell-Ash Company analyzed the samples with the laser source and supplied the writer with much practical information. Drs. J.A. Chamberlain, G. Pouliot, T.N. Irvine, and K.R. Dawson of the Geological Survey of Canada provided the samples for analysis.

LASER THEORY

Discussion of laser theory has become something of a popular indoor sport, and simplified accounts can be found in recent issues of TIME, LIFE, THE AMERICAN SCIENTIST, READER'S DIGEST and the TORONTO STAR WEEKLY. For a thorough and detailed exposition of the theory and nature of optical masers, reference should be made to the 142-page supplement on Optical Masers in Applied Optics, 1962¹. A significant indication of the newness of the application of the laser as a source in emission spectrography is provided by the fact that such an application is not mentioned in the thorough coverage given by this supplement.

¹A selected bibliography is given at the end of this report.

No attempt will be made here to discuss the quantum mechanics behind laser operation and those interested are referred to the selected bibliography for sources of this information.

A distinction should be made first between the terms "maser" and "laser". The maser (microwave amplification by stimulated emission of radiation) is a generic term for devices involving the amplification of electromagnetic radiation by passage of an external source of such radiation through an active medium and resulting in stimulated emission of radiation. The laser (or optical maser) is a special case of the maser in which the active medium is pumped or activated by light in the commonly accepted sense and results in light amplification by stimulated emission of radiation.

The basic principles were first elucidated by C.H. Townes in 1952 but of more direct concern to practical spectroscopy was the proposal of the solid state laser in 1958 by Townes, and its practical application by Schawlow in 1960. The most popular of operating lasers is the pink ruby rod and the following discussion is based on its operation.

Stored energy from a large capacitor produces, through an electrical discharge, an extremely intense burst of incoherent (irregular and unpredictable) light which is made to impinge on a special ruby rod. An optical cavity is set up along the long axis of the ruby and certain ions are stimulated to emit electromagnetic radiation of essentially a single frequency and with a planar wave front. In the case of the pink ruby, these ions are those of chromium which is present to the extent of 0.05%, and they are pumped or excited by the stimulus of the impinging light,

commonly provided by a xenon flashlamp would around, or arranged parallel to, the ruby rod.

The chromium ions absorb the green light of the xenon flashlamp, are pumped or activated to a higher energy level and then revert to the ground state, emitting energy in the form of red light. The device thus becomes a light amplifier and is made to oscillate by means of a special optical system consisting of a pair of optical flats, one of which is placed at each end of the long axis of the rod. One flat is fully silvered, the other semi-silvered, and the stimulated radiation is made to bounce back and forth between these mirrors, resulting in further stimulation of chromium ions and an increase in its intensity. It finally is allowed to emerge and does so as a nearly monochromatic and directional beam of red light (about 6940\AA) which rivals radio waves in regularity or coherence. Because it is parallel and coherent it can be focused with considerable accuracy. Many variants of this basic system are being employed. A major practical problem was the existence of a threshold level for operation of the laser and early units required peak power levels in the pumping system in order of tens or hundreds of kilowatts. This required specially constructed pump lamps whose life was short and the heat generated by these large bursts of energy created further problems. The development in recent months of specially grown ruby boules has greatly increased pumping efficiency and the threshold level has been reduced to less than one kilowatt.

Workers are actively investigating the use of many other laser materials, among which are corundum (containing nickel, vanadium, or iron), calcium tungstate (containing rare earths), calcium fluoride (with uranium or samarium), barium fluoride (with uranium), neodmium glass and gallium arsenide. The use of a liquid as the active medium is also being considered, and much work has been done with the gas laser since it was first proposed by Javan in 1959. The gas laser is activated by low radio frequencies rather than by light and a helium-neon unit has been made to operate continuously. The light produced is the most nearly monochromatic of all the maser-laser family.

OPERATIONAL DETAILS

The writer was afforded an opportunity to test the application of the laser source to geological studies by an invitation from Mr. Frederick Brech to visit the research laboratories of the Jarrell-Ash Company in Newtonville, Massachusetts. Sixteen polished sections, ten rock slices and one thin section were analyzed qualitatively with the laser source. The results of this investigation are given in the next section of this report and a detailed description of the apparatus used is given in an appendix.

The equipment consists of the laser head (ruby rod, helical xenon flashlamp, cooling device), a Q-switch or shutter, a metallurgical microscope fitted with carbon electrodes suspended between the objective lens and the surface of the sample resting upon the microscope stage, and

a spherical lens which focuses the light upon the slit of a spectrograph. The power supply, energy storage bank and triggering system are contained in a single control console.

Procedure

The operation of the laser source is simple. The specimen is placed upon the microscope stage and the cross-hairs focused on the sample area. The electrodes are inserted, the capacitors are charged to 4200 volts and the firing button is depressed. A sharp cracking noise and a brief burst of white light at the sample surface are indications that the two-step excitation has occurred. The sample can again be viewed to see the effect of the laser shot. It is then only necessary to reposition or replace the sample, change the electrodes and recharge the capacitors before repeating the shot. A minimum interval of 1 minute between laser firings is necessary in order to operate the laser source at maximum efficiency.

Accuracy of Aim of the Laser Beam

The action of the laser pulse on the sample surface produces a pit which, though irregular in shape, is roughly hemispherical. A pit diameter of only 50 microns has been achieved but for routine operation the diameter is usually 100 ± 20 microns, with a depth of about 50 microns. No wandering of the laser beam occurs and the pit is confined to the desired area, providing that the latter is greater than 120 microns in diameter. Viewing of the target area after vaporization is hindered, however, by the deposition of carbon from the cross-excitation electrodes around the

impact pit. This can be wiped from polished surfaces but rough surfaces provide irregularities to hold the deposit. This smudging can be avoided by the use of copper electrodes, provided that copper is not a constituent being sought; the use of an inert atmosphere such as argon will probably eliminate much of this difficulty.

Nature of Sample Surface

The roughness of the surface is limited by the need to maintain a suitable distance between the surface and the cross-excitation electrodes. Mounting of the specimen on the microscope stage may be done with plasticene and so it is not necessary to have parallel sides. In general, then, the shape and surface nature of the specimen is not critical. For viewing before and after the laser action a relatively smooth surface is convenient, and greatly aids in the selection of target areas. Polished sections are ideal, although further investigation is necessary to determine the possible effect of a highly-polished surface on the excitation process.

Thickness of the Sample

Because the pit produced by the laser action is about 50 microns deep, a minimum thickness of about 100 microns is preferred. This rules out the use of a standard thin section (30 microns thick), for the laser beam burns through the section, carbonizes the cement and vapourizes a portion of the glass slide, producing a very unsatisfactory spectrum.

Sensitivity and Reproducibility

The routine lower limit of sensitivity obtainable with a single shot for certain elements is said by Jarrell-Ash to be 0.001%. It is possible, however, to detect constituents present to the extent of a few parts per million by the technique of superimposing ten successive shots on one exposure, providing that the material is sufficiently homogeneous to permit this.

The reproducibility of the whole process is now said by Jarrell-Ash to be +5%.

Nature of the Spectra

The spectra of the samples were recorded on Kodak 1030 35- millimetre film for the most part, with one series being recorded instead on Kodak 1N film. The spectra obtained on the latter were very light and uninformative, but those on the 1030 film were generally good and showed a high sensitivity for the method. The spectra were complex, consisting of an admixture of arc and spark lines of two or three orders, including heavy carbon lines. Individual exposures varied very considerably in density, especially between those for the ore minerals and those for the silicate minerals; this may be a result of the variation in thermal conductivity of different minerals. A reference iron spectrum was recorded at the beginning and end of each set of sample exposures (two to each film) and the same variation in density is encountered among these also.

EXPERIMENTAL RESULTS

The series of polished sections, rock slices and one thin section were analyzed with the following results.

<u>Polished Sections</u>	<u>Major</u>	<u>Minor</u>	<u>Trace</u>	<u>Faint Trace</u>
Bornite	Fe, Cu	Cr	- - -	Mg, Si, Ca, Ag
Sphalerite	Zn	Fe	Pb, Cd?	Cu, Si, Mg, Ca
Magnetite	Fe	- - -	Mg	Si, Mn, Ca
Sphalerite ^a	Zn	Fe	Si	Pb, Mg, Ca
Chalcopyrite ^a	Fe	Cu	- - -	Mg, Ca
Pyrite	Fe	Ni	- - -	Mg, Ca, Cu, Co, Ag
Chromite	Fe, Cr	Ti	Mg, Al	Mn, Cu, V, Ca
Pyrrhotite	Fe	Ni	- - -	Mg, Cr, Ca, Ag
Galena ^a	Pb	- - -	Fe, Ag	Si, Mg, Bi, Ca
Chalcopyrite ^a	Fe, Cu	Al	Cr, Mg	- - -
Siegenite ^b	Ni, Fe		Co, Mg, Cu, Zn	- - -
	Ni, Fe	Cu, Zn	Co, Mg	- - -
	Ni, Fe	Cu, Zn	Co, Mg	- - -
Niccolite	As	Ni	Mg, Fe	- - -
Meteorite (Kamacite)	Fe	Ni, Si	Cr	Mg, Ca
Meteorite (Enstatite)	Si, Ti	Mg	Fe, Al	- - -

^aTwo minerals in same section

^bThree shots were taken

Polished Sections (Con't.)

	<u>Major</u>	<u>Minor</u>	<u>Trace</u>	<u>Faint Trace</u>
Skutterudite	As, Co	Ca, Fe	Ni, Cu, Cr	- - -
Arsenopyrite	Fe, As	Ni, Cr	Mg	- - -
Chromite	Cr	Fe, Al	Mg	- - -
Chromite	Fe, Cr	Al, Mn	Mg	V, Ca
Chromite	Fe, Cr	Ti, V	- - -	Al

It is seen that identification is correct for all samples.

Additional information was obtained in the case of the high nickel content of pyrite, and in the presence of silver in the galena. The three shots on the siegenite gave almost identical results and confirmed the identity of this mineral. When two minerals in the same section, i.e., sphalerite and chalcopyrite in one and galena and chalcopyrite in another, were both shot, no contamination of the one by the other occurred.

The pits formed were nearly hemispherical in shape and usually occurred without much visible fracturing or shattering of adjacent material. Tarnishing of the surface was visible on all specimens and a deposit of carbon was nearly always present. It could be nearly all removed by rubbing the surface.

Further work must be done to determine the conditions that favour the formation of pits. It would appear that hardness of the material is an important factor; no pits were formed in any of the four chromites

nor in the bornite, but shattering and fusion of the surface occurred.

Extensive micro-fracturing occurred in the bornite and it is of interest to note that the fractures ended abruptly at the contact between the bornite and neighbouring galena.

Only in the case of the bornite was the surficial damage sufficient to warrant repolishing of the specimen.

Rock Slices

	<u>Major</u>	<u>Minor</u>	<u>Trace</u>	<u>Faint Trace</u>
Clinopyroxene ^a	Si	Ca, Mg, Fe	Al	Ti
Orthopyroxene (1) ^a	Si	Mg, Ca, Fe	Mn	Ti, Al, Cr
(2)	Si	Mg, Ca, Fe	- - -	Ti, Al, Cr
Orthopyroxene ^a	Si, Ca	Fe, Mg	- - -	Ti, Al, Cr
Ilmenite ^b	Si, Ca	Al, Fe, Ti	Cu, Mg	- - -
Plagioclase Feldspar (1) ^c	- - -	Mg, Ca	Si, Al	B, Fe
(2)	- - -	Ca	Mg, Si, Al	B, Fe
Plagioclase Feldspar (1) ^c	- - -	Ca	Mg, Si, Al	B, Fe
(2)	- - -	Ca	Mg, Si, Al	B, Fe

^aIn pyroxenite

^bIn gabbro

^cIn anorthosite

Rock Slices (Con't.)

	<u>Major</u>	<u>Minor</u>	<u>Trace</u>	<u>Faint Trace</u>
Potassic Feldspar (1) ^d	Si, Na	K, Al	Mg, Ca	Fe
(2)	Si, Na	K, Al	Ti, Mg, Ca	Mn, Fe
(3)	Si, Na	K, Al	Ti, Mg, Ca	Mn, Fe
Potassic Feldspar (1) ^e	- - -	Si	Ca, Mg	Al
(2)	- - -	Si	Ca, Mg	Al

^dIn metamorphic rock

^eIn granophyre

Shots were also taken on zoned feldspar crystals in granite and in porphyry. An attempt was made to use 1N film in the expectation of getting good potassium lines in the spectra but the resultant spectra were very weak and incomplete. Better results were obtained with the 1030 emulsion.

In the granite and porphyry the pits were easily recognizable although irregular and accompanied by shattering. A good pit was obtained for one of the three shots taken on the potassic feldspar, but the other two shots produced only shattered and fused material. Irregular pits were formed in the others, again with accompanying shattered and fused material, except for the ilmenite, which showed much fracturing and fusion. The results are not as clear-cut as they are in the case of the polished

specimens of ore minerals. The three pyroxenes show acceptable differences in major and minor constituents, and the ilmenite is identifiable. The results obtained for the feldspars, with the exception of the potassic feldspar, are not good; the aim of the laser beam is not in question, however, for it was possible to produce pits in selected zones of the crystals.

Thin Section

A thin section, somewhat thicker than normal (50 microns) and containing magnetite-ilmenite (?) and hercynite was shot in two places. Major Ti and Al, minor Fe and Ca, and traces of Si, and Mg were found for the thicker magnetite-ilmenite (?) but only major Mg and minor Fe, with traces of Ca, Na and Si were found for the hercynite. The material struck by the laser beam was evidently shattered and blown from the section; the only evidence of the second laser shot is a spherical pit in the glass slide.

CONCLUSION

The results given in the previous section indicate that the laser source can be used to identify minerals in polished sections and rock slices. It appears to give more satisfactory results with sulphide and other ore minerals in polished sections than with silicates but the results are preliminary and experimental in nature and too much should not be read into them. Pits were produced in the majority of the samples; the pits were more regular in shape in the ore minerals than in the silicates, and shattering and fusion were usually visible in the latter.

The emphasis at present is on qualitative identification. Semi-quantitative analysis is possible through experience and with the use of appropriate standard samples for comparison, provided that the effect of the excitation process on the material is uniform from one sample to another. Quantitative analysis will be possible only when the many variables involved in the process can be controlled so that the use of proper standards is feasible. Jarrell-Ash are investigating the semiquantitative and quantitative possibilities of the technique.

Satisfactory spectra have been obtained by Jarrell-Ash from alloys, ceramics, glass and powdered material (although the aftermath of the laser action on the powder is messy). No success has been achieved with organic material because carbonization of the sample occurs, nor with liquids because no usable vaporization is obtained. The detection of iron in blood, however, has been carried out by evaporating several drops of blood and using the resulting solid residue.

Although the laser source has been used chiefly to study non-homogeneous material (e.g., inclusions) it can be used in the routine study of homogeneous material; for example, ten successive separately-placed shots are made without cross-excitation on the first nine, so that the vapourized material condenses on the electrodes. By discharging the electrodes on the tenth shot a complete record of the traverse made is obtained. Such a study would require only a few minutes time.

Further study is needed, as mentioned previously, of the relationship between the effectiveness and characteristics of the laser shot and the smoothness of the surface. Are highly polished surfaces more effective? Irregular pits were produced in a highly polished specimen of magnetite; was this because of the high polish or did the magnetic property of the sample cause the spark to discharge to the surface?

There seems to be a relationship between the hardness of the material and the tendency for the laser shot to cause fracturing (e.g., the observation made on neighbouring bornite and galena). This may be a reflection of the variation in the coefficients of expansion of minerals, or of differing thermal conductivities. Does the effect depend upon the crystallographic orientation of non-isometric minerals? Does the nature of the crystal bonds in the mineral affect the magnitude of the laser action? There seems to be little doubt that the effect of laser action is going to vary with the nature of the sample, and herein lies a serious stumbling block to semiquantitative and quantitative analysis with a laser source. The cross-excitation process may also be at fault; elimination of these auxiliary electrodes and vapourization and excitation by an improved laser source alone may increase the reproducibility of the technique when applied to geological materials.

What is the nature of the cloud or jet of vapour produced by the laser action? Is it composed of ionized vapour, a cloud of incandescent particles or, as is likely, a mixture of both? Jarrell-Ash are planning to

use high-speed photography to learn more about the nature of the vapour, with a view to controlling its size and shape.

These are but a few of the problems that remain to be investigated before the laser source can be regarded as a truly effective tool in the study of geological materials. The results that have been obtained in this preliminary study are, however, of considerable interest and the potential application of the laser source in geological, geochemical and mineralogical studies is great indeed.

SELECTED BIBLIOGRAPHY

- Bennett, W.R. Jr.
1962: Gaseous optical masers; Applied Optics, Supplement I, pp. 24-61.
- Collins, R.J. et al
1960: Coherence, narrowing, directionality and relaxation oscillations in the light emission from ruby; Phys. Rev. Letters, vol. 5, pp. 303-305.
- Gordon, J.P., Zeiger, H.Z., and Townes, C.H.
1954: Molecular microwave oscillator and new hyperfine structure in the microwave spectrum of NH₃; Phys. Rev., vol. 95, pp. 282-284.
- Heavens, O.S.
1962: Optical masers; Applied Optics, Supplement I, pp. 1-23.
- Hellwarth, R.W.
1961: Theory of the pulsation of fluorescent light from ruby; Phys. Rev. Letters, vol. 6, pp. 9-12.
- Lengyel, B.A.
1963: Lasers, John Wiley and Sons, Inc., New York (in press).
- Lewis, H.
1961: Optical masers; Int. Science and Technol., Prototype Issue, pp. 60-73.
- Maiman, T.H.
1960: Stimulated optical radiation in ruby; Nature, vol. 187, pp. 493-494.
1960: Optical and microwave-optical experiments in ruby; Phys. Rev. Letters, vol. 4, pp. 564-566.
- Nelson, D.F., and Collins, R.J.
1961: Spatial coherence in the output of an optical maser; J. Appl. Physics, vol. 32, p. 739.
- Oliver, B.M.
1962: Some potentialities of optical masers; Proc. Inst. Radio. Engrs., vol. 50, p. 207.

Schawlow, A.L.

1961: Optical masers; Scientific American, vol. 204, pp. 52-61
(June).

Schawlow, A.L., and Townes, C.H.

1958: Infrared and optical masers; Phys. Rev., vol. 112,
pp. 1940-1949.

Stitch, M.L.

1961: Power output characteristics of a ruby laser; J. Appl.
Physics, vol. 32, p. 1994.

1962: Lasers boost spectrographic utility; Chem. and Engineering
News, September 3, Part I, p. 52.

APPENDIX

DESCRIPTION OF THE EQUIPMENT

Laser research is being done in many countries but to the writer's knowledge only Jarrell-Ash are utilizing the laser as a source in emission spectroscopy and have produced commercially available equipment for the two-step excitation process described in the introduction to this report. The following is a description of their equipment.

The Laser Head

This consists of two parts; the ruby rod and flash-lamp, and the Q-switch (or Q-spoiler) to regulate the laser pulse.

The ruby rod, 4" x 1/4", is fitted at one end with a roof prism in place of a mirror. The rod is surrounded by a glass tube which carries liquid nitrogen vapour as a coolant. The rod and tube are surrounded in turn by a helical xenon flash-lamp which is cooled by a current of air. These are enclosed in a cylindrical chamber mounted upon a stand which can be easily fitted to the bar of an optical bench. Alternatively, the laser head, Q-switch and microscope may be mounted at right angles to the spectrograph and the light focused on the slit by means of a lens and mirror.

The Q-switch (or Q-spoiler, a term used to denote anything which lowers the resonance of an oscillator) is contained in a box fastened in front of the exit end of the laser head. It consists of an interferometer plate of the Fabry-Perot type mounted on a plate which rotates at 3600 rpm. A diode is mounted beneath the plate, and a small light source is mounted

above it. A slot cut in the plate enables the diode to see the light source once in each revolution of the plate. The interferometer plate opens the optical path (i. e., is parallel to the end of the rod) only when a high level of intensity exists and is thus instrumental in producing giant pulses of energy of the order of 1-3 megawatts, energy pulses which may be two or three times the peak power obtained from an unshuttered system. A signal to the diode starts the sequence of events which occur in a matter of milliseconds and which result in the release of a laser pulse.

The Microscope

A metallurgical microscope, fitted with a vertical illuminator and side eyepiece, is used to enable selection of the area to be analyzed, and to focus the laser pulse upon this area. A prism (or mirror) is placed at the upper eyepiece and the laser beam is reflected down the centre axis of the microscope. A special long working distance objective lens (20X) focuses it on the surface of the specimen which is held in position on the travelling stage; the objective lens remains fixed and focusing is done by vertical movement of the stage. The distance between the lens surface and sample surface must be sufficient to prevent contact between the lens and the laser-induced jet of vapour; a distance of 6 millimetres is considered safe but the actual gap used at present is about 18 millimetres. If necessary an ordinary glass cover slip may be placed over the objective lens.

It should be noted that the laser beam will pass through the lens without harm to it, provided that no dust particles are present on it. The sudden explosive vapourization of these particles may cause chipping of the lens. Air bubbles in the cement of the doublet may result in shattering of the lens if they are in the path of the beam.

The Cross-excitation Electrodes

These electrodes, $1/8'' \times 1\frac{1}{2}''$ sharpened graphite rods, are suspended about 6-8 millimetres above the surface of the sample and are separated by a 3 millimetre gap. The support which holds them is fully insulated from the microscope and they are kept far enough away from the sample surface so that the spark will not discharge to the surface. A potential of 1200 volts is continuously applied to them and they discharge only when the laser-induced vapour ionizes the air gap between them. They thus discharge once with each laser pulse.

Because of the condensation of vapour on the electrodes it is necessary to change them for each shot (or the spectrum of the previous shot will be reproduced), but replacement of the electrodes is easy and fast. The use of a revolving multi-electrode head was proposed to minimize the time required for electrode changing but this has not proved feasible. Further work is being done to obviate the need for fresh electrodes by controlling the physical characteristics of the vapour jet.

The Spectrograph

Once the laser pulse has caused vapourization of the sample, the subsequent steps are those of conventional arc-spark spectrography. Any spectrograph may be used but a fast spectrograph is mandatory because of the short period of light emission. Jarrell-Ash uses a 1.5 metre Wadsworth mount grating instrument, with a slit opening of 35 microns and a speed of f30. The slit is kept permanently open but automatic opening and closing of the slit could easily be arranged. A single spherical lens is used to focus the light on the slit of the spectrograph.

In conventional arc-spark spectrography the amount of sample consumed is in the milligram range and a large part of the high energy applied to the electrodes is needed merely to vapourize the sample. Because the sample is vapourized instead by the laser pulse and because the quantity of sample used is in the microgram range, much less energy is needed for this two-step excitation.

To date most of the experimental work has been done with a film-recording spectrograph, chiefly because the complete spectrum of each shot was needed. The laser source has been used with a direct-reading spectrograph also. No operational difficulty is foreseen for the latter arrangement apart from the lesser flexibility of the direct-reader compared with a film or plate recording instrument. Where a large number of detector channels are available however, this difficulty is minimized.

The Control Console

A control console, at present about the size and shape of a three-drawer letter filing cabinet, is needed to provide the power supply, energy storage bank and triggering system. A small separate box houses the actual firing system and it may be operated manually, automatically or by remote control.

General Comments

The preceding description is that of the separate components of the equipment. The commercial equipment will consist of two units: an analysis unit which will incorporate the laser head, Q-switch and microscope and which, weighing about 30 pounds, can be easily fitted to the bar of an optical bench; and a control console unit incorporating the power, cooling and control systems. The analysis unit will be designed for use with any spectrograph, requiring only minor adjustments. The first of these newly-designed laser sources will be displayed in March, 1963 at the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy. The laser head and console are manufactured by Trion Instruments, Inc., Ann Arbor, Michigan, in collaboration with Jarrell-Ash. The cost of the equipment is approximately \$12,000.

The new laser head will feature a new type of Q-switch. The interferometer plate will be kept fixed parallel to the end of the ruby rod, but the roof prism at the other end of the rod will be rotated instead. This Q-switch is more efficient in terms of peak power and as a result the amount of cooling needed for the rod is almost negligible. A temperature

sensor will be included which will activate the cooling system when it is needed. A new alignment system will also be used; a more careful alignment of the system results in a considerable increase in efficiency of operation.

The present microscope is equipped for vertical illumination but it is a simple matter to provide for both transmitted and plane polarized light as well. It is only necessary that the barrel of the microscope be cleared of anything that will interfere with the laser beam before it is fired.

Provision is being made for a sample chamber (possibly some type of diaphragm arrangement) that will make possible the use of an inert atmosphere to reduce the amount of cyanogen banding that occurs when the sparking is done in air.

It is proposed to mount the electrodes in a type of removable drawer fitted with a window to enable the light to escape. Changing of the electrodes is thus easily done.

No individual control of either the laser pulse or the electrode discharge is possible at present, although it is hoped that a more flexible system will be possible in the future. It is considered that the present conditions are more than adequate.