Simple Device for Luminescence Petrography

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Thus far, all reported work on luminescence petrography has been accomplished by use of the microprobe, a very expensive instrument beyond the grasp of most petrographers. A simple device is described here which attaches to an ordinary petrographic microscope and permits luminescent observations as well as ordinary white or polarized light observations. The instrument has the advantages of short pump down time (two min), large area of illumination by the electron beam (1×3 cm), and a minimum of charging and sparking problems. In addition, ordinary uncoated rock or mineral thin sections may be used. The instrument has proven to be superior to the microprobe for luminescent observations of geological specimens. Other (nongeological) applications in fields such as ceramics and solid state physics are also likely.

INTRODUCTION

THE use of electron excited luminescence as a petrologic tool was suggested by Smith.[1,2] He showed that when rock or mineral thin sections are examined microscopically by the light produced by impact of a broad beam of low energy electrons entirely new phenomena and structures are evident which are invisible under ordinary white or polarized light examination. Smith used a microprobe for his observations, an instrument beyond the grasp of most petrographers. He suggested, however, the possibility of building a simple electron emitter device which might be attached to a petrographic microscope.[2] Such a device has been built at this laboratory and has been in constant use for several months. It performs reliably and is actually superior to the microprobe for luminescence studies since much larger areas can be examined and fewer problems of charging and sparking are encountered. In this instrument the electron beam illuminates an elliptically shaped area of the rock thin section about 1×3 cm in size. The illuminated region is centered on the optic axis of the microscope and remains fixed; the specimen may be moved with respect to the beam and microscope axis by independent x and y controls. With a homogeneous phosphor in the specimen position, the light intensity is quite uniform over this entire region, implying that the electron beam intensity is also uniform. Rock and mineral thin sections by no means approximate uniform phosphors however, and thus one observes luminescent structures caused by nonuniform distribution of activator ions or luminescing defect centers.[3] The instrument permits direct and immediate comparison between electron excited luminescent structures and the structure and texture observable by ordinary white or polarized light petrography. These observations give important clues to the origin and history of the rock or mineral specimen examined.

DESCRIPTION OF THE INSTRUMENT

The instrument is hardly more than a discharge tube constructed in such a way as to permit ions and electrons to impinge on the thin section specimen, while keeping the discharge isolated. This prevents light originating in the discharge from obscuring the luminescent light emerging from the specimen. The device is so arranged that ordinary white or polarized light petrographic observation is possible and may be alternated with luminescent observation at will. The electrode configuration arrived at after considerable experimentation is shown schematically in Fig. 1 and in the photograph, Fig. 2. The discharge is initiated in the desired region of the discharge tube by the ring of sharp corona points. The specimen chamber is grounded and alternating high voltage of approximately 10 kV is applied to the curved electrode. On the half-cycle when the curved electrode is positive with respect to the specimen chamber, positive ions may escape through the exit hole in the center of the corona point ring and impinge on the thin section specimen. On the reverse half-cycle, electrons formed in the discharge are accelerated and impinge on the specimen. In addition (on this half-cycle), positive ions proceed to the curved electrode where, on impact, they produce secondary electrons which are also accelerated to impinge on the thin section specimen. The secondaries leave the curved electrode normally to the surface so some degree of focusing is obtained with these electrons. Good focusing would, of course, be obtained only if the mean free path for electrons was long with respect to the curved electrode-specimen distance. This is only possible below the gaseous discharge region where the electron intensity is too low for our purpose so a happy medium must be struck. It is found that a pressure range of about 5–20 μ is best. In this range, the intensity is adequate to excite bright luminescence in rock thin sections while per-

[1] R. C. Stenstrom and J. V. Smith, "Electron-Excited Luminescence as a Petrologic Tool (abs)," Geol. Soc. special paper No. 76, p. 158 (1964).
[2] J. V. Smith and R. C. Stenstrom, "Electron-Excited Luminescence as a Petrologic Tool," Symposium on the Microprobe, Electrochemical Society, 12–15 October 1964, Washington, D. C. (This article was privately distributed.)
[3] R. F. Sippel and E. D. Glover, "Previously Unobservable Structures in Carbonate Rocks Made Visible by Luminescence Petrography," Science (to be published).

haps some modicum of focusing remains. At pressures much higher than this, the discharge extends into the brass specimen chamber with resultant degradation of the image. At lower pressures, the electron density approaches zero as the discharge dims and finally extinguishes. A mechanical leak is used (Fig. 2) to adjust the pressure to the desired operating range. The electrical system is quite simple. The high voltage is supplied by a Precise Company 25 kV potted transformer. The primary is supplied by a Variac having a ballast light bulb of 150 W in series as a current limiter. The light bulb does not protect the transformer against transient surges, however, so a second protecting device consisting of twenty 40 kΩ, 2 W resistors is incorporated in series with the secondary. This latter feature is very useful in providing regulation for the system, and partially accounts for the stability of the luminescence. Needless to say, a good water pipe ground on the specimen chamber is necessary for safety.

For x-ray safety, the discharge tube is covered with a lead shield when the unit is in operation; and the top port through which the specimen is viewed, and the bottom port through which white light is conducted for white light petrography, are leaded glass approximately 3 mm thick. Careful monitoring of the instrument in use shows the x-ray dosage received by the operator to be negligible. This, together with the general theory of the instrument, indicates that the mean electron energy must be quite low.

In ordinary petrography the thin section is rotated between crossed Nicol prisms, something which is quite out of the question here, so the equivalent effect is obtained by rotating crossed Nicols by means of the rod mechanism, indicated in Fig. 2. This was resorted to when it proved too difficult to connect the upper and lower Nicols by means of a mechanical device. A Polaroid sheet acts as the top Nicol and is rotatable in phase with the lower Nicol. The upper Polaroid rotates in a Teflon bearing which is not visible in the photograph. The independent x and y motion

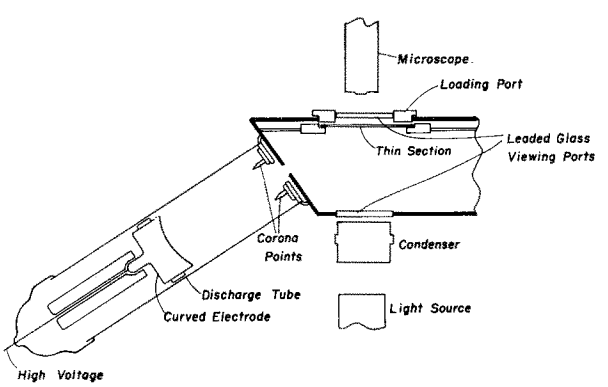

FIG. 1. Schematic drawing of the instrument. In operation, the discharge is confined within the discharge tube, between the curved electrode and the corona point ring.

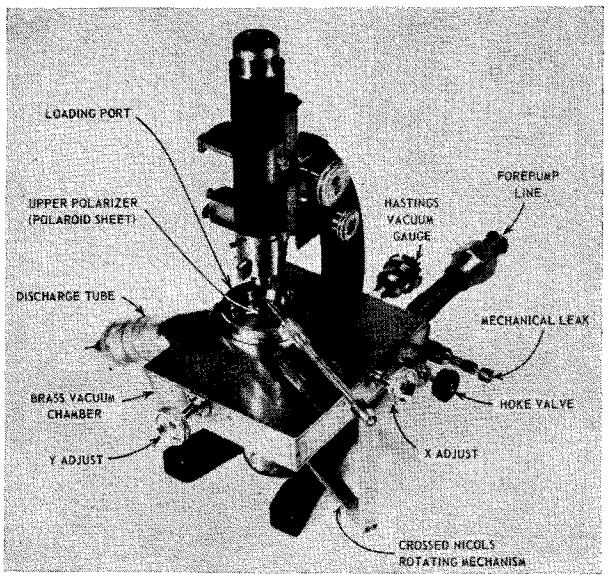

FIG. 2. Photograph of the instrument showing the method of rotating the crossed Nicols about the microscope axis. For luminescence work the top polarizer may be removed.

is achieved by adjusting screws bearing on a slide carrier which is spring loaded in the x and y directions.

INSTRUMENT OPERATION

Thin sections to be bombarded by electrons in the device must be epoxy mounted. Lakeside or Canada balsam mounting is not satisfactory. Considerable heating results from the electron bombardment and low temperature mounting cements boil within a few seconds after the beam is turned on. Since the specimens cannot be treated with immersion oil, it is preferable to use polished thin sections to keep light scattering to a minimum. However, luminescent features are considerably sharper in appearance than the white light features, and it is quite feasible to use ordinary unpolished thin sections at low magnification (×10–×20). At higher magnification, (×50 and higher) special long working distance objectives are required, and light scattering from ordinary unpolished thin sections becomes increasingly disturbing in white light. However, even at ×200, luminescent features can be seen with amazing clarity even in an unpolished thin section. Unlike the microprobe, the instrument does not require thin sections to have a conductive coating. Slide breakage occasionally occurs due to the heating effect of the electron beam. This could be eliminated by using Pyrex petrographic slides. In some cases, however, minerals will craze without breaking the slide. These problems can be minimized by interspersing off times of 5 or 10 sec between observation times of 20 or 30 sec. One should avoid continuous electron bombardment for times of several minutes to minimize slide or mineral breakage, even though many individual slides may be able to take such bombardment without apparent damage.

Photomicrography may be readily accomplished by standard techniques. Exposure times for luminescent photography, using High Speed Ektachrome film, range from one-half second to about one minute. A cadmium sulphide cell ohm-meter combination has been used for estimating exposure times. Some light originating in the air discharge does reach the specimen; and for critical work and the best quality photography, this stray light can be largely removed by use of a gas other than air to support the discharge. This other gas may be admitted through the mechanical leak. Both hydrogen and helium are superior to air in this respect, but hydrogen works best. Using a hydrogen discharge at the optimum pressure, only a very dull red discharge can be observed in the lower region of the discharge tube. This allows only minimal light to reach the specimen. Even this minimal amount could effectively be removed by incorporating an interference filter in the optical path.

Although the instrument has been developed primarily for observation of geological specimens, it should prove useful for luminescent observations of other substances. Applications are likely to be found in such fields as ceramics, cements, synthetic crystal production, and particularly in the area of solid state physics research.

ACKNOWLEDGMENTS

The author is indebted to Professor J. V. Smith who discussed freely the properties which a luminescence device should have, and in particular, very graciously relayed to the author the experience of Dr. J. V. P. Long who found that a simple, poor vacuum, alternating current device was feasible.

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Alternating Current Electromagnet Type Hysteresis Loop Tracer for Minerals and Rocks

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A hysteresis loop tracer employing an alternating current electromagnet capable of providing fields up to 3600 Oe is described. Merits of the present system over other types of loop tracers are pointed out. Some typical hysteresis loops obtained using the apparatus are also presented.

1. INTRODUCTION

HYSTERESIS loop tracers employing air-core coils to produce the required sinusoidal magnetic field have been extensively used.[1–3] The fields obtainable using air-coils are limited to about 1000 Oe on account of (a) the inevitable bulkiness of the winding, (b) the necessity of using high voltage condensers for tuning the coil to the frequency used, and (c) the heating up of the coil during the experiments. To overcome these difficulties, we explored the possibility of using an alternating current electromagnet employing a core having high permeability, low hysteresis loss, and small harmonic distortion. For this purpose, we found a modern grain oriented, laminated transformer core admirably suitable. In association with this electromagnet a special multicoil element has been used to detect the magnetization of the specimen placed in the pole gap. The performance of the apparatus has been found to be very satisfactory and it is felt that the apparatus can be used in many cases with greater advantage than the conventional ones employing air-core coils.

[1] M. V. Scherb, Rev. Sci. Instr. **19**, 411 (1948).
[2] D. H. Howling, Rev. Sci. Instr. **27**, 952 (1956).
[3] J. McG. Bruckshaw and B. S. R. Rao, Proc. Phys. Soc. (London) **63**, 931 (1950).

A. Electromagnet

A "C" core (type HWR/110/32/13) of laminated grain oriented silicon steel manufactured by the English Electric Company was suitably altered to serve as an electromagnet. The cross section of the poles is 5×2.5 cm^2. A piece 1.2 cm in thickness cut from another similar core was interposed on one side of the "C" shaped pieces thus providing a pole gap of 1.2 cm on the other side. For the energizing coil, 2200 turns of 20 SWG super enamelled copper wire were wound on a Perspex former, which fits over one side of the core. The winding almost fills up the window area of the core-loop leaving just enough space for the placement of the multicoil. The energizing coil has a resistance of 11.5 Ω. On account of the relatively large air gap used in the core, the permeability is constant for sufficiently large values of the dc magnetizing force, and hence, the field produced in the gap can be expected to vary linearly with the current in the energizing coil. The dc field in the pole gap has been measured with a Rawson-Lush rotating-coil gaussmeter, type 820S, and found to vary linearly with the energizing current up to a field of 3600 Oe. However, germane to the present discussion, the linearity between field in the pole gap and the energizing current has also