AN APPLICATION OF THE RESONANCE RADIOMETER TO THE REFLECTION SPECTRUM OF QUARTZ

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Abstract

The reflection spectrum of quartz has been previously studied by means of both the rock-salt prism spectrometer and the echelette grating spectrometer. Early investigators have shown that for crystalline quartz, with the reflecting surface perpendicular to the optic axis, there is a double reflection maximum, one lying at 8.4μ and the other at 8.9μ; and that fused quartz exhibits a single maximum at 8.8μ. These regions have been studied under high dispersion by the authors using an echelette grating spectrometer. The high resolving power that was obtained was due to the sensitivity of the radiometric device, the resonance radiometer, which was first described by A. H. Pfund.[1] The complete theory of the instrument was developed by one of us.[2] By the use of this large resolving power and dispersion it has been possible to show that these spectra are considerably more complex than were found previously. The usefulness of magnesium oxide as a filter for excluding the higher orders of lower wave-lengths has been tested with gratifying results.

Introduction

PREVIOUS investigators have studied the reflection spectra of quartz with particular attention to crystals cut perpendicular to their optic axes. Among the various investigators Rubens and Nichols,[3] Coblentz,[4] and Gorton[5] employed a prism spectrometer; Wood and Trowbridge[6] used an echelette grating. The results show some agreement as to the wave-lengths of the reflection maxima, which are located at 8.4μ and 8.9μ respectively, but there is an apparent marked difference in the depth of the minimum which separates the maxima. Closer examination of these dissimilar curves shows that they actually represent very approximately the same structure. The work done with the prism spectrometers shows the ratio of the reflecting power of the quartz surfaces to that of a silver surface, while the work done with the grating shows only the energy curve of the source after three reflections from quartz. This makes it difficult to compare directly the results of the grating with those of the prism. Also, it should be pointed out that as very few workers have restricted themselves to similar crystal forms or similar geometrical systems, it is necessary to be cautious in attempting any comparison of the various results. The conditions for observation in the present case are as

[1] A. H. Pfund, Science **69**, 11 (1929).
[2] J. D. Hardy, Rev. Sci. Inst. **1**, 429 (1930).
[3] Rubens and Nichols, Phys. Rev. [1] **4**, 314–324 (1897).
[4] Coblentz, Carnegie Publications, 1906.
[5] Gorton, Phys. Rev. **7**, 66 (1916).
[6] Wood and Trowbridge, Phil. Mag. **20**, 898 (1910).

follows; the angle of incidence was measured to be very close to 30°; the crystal surface was polished smooth and cut perpendicularly to the optic axis; the results are the ratios between the reflecting power of quartz surfaces and that of clean silver. It is thought that if these conditions are approximated it should be possible to use the positions of the reflecton maxima and minimum for calibrating prism spectrometers in the 8μ region.

In the early part of this year, Professor A. H. Pfund made some observations on the reflection from crystalline quartz (not published) and found results differing markedly from those of Coblenz and others. He found, using a very narrow slit, a much lower minimum at 8.6 and some indication of unresolved fine structure. It was decided, therefore, to examine this spectrum with an apparatus whose sensitivity and resolving power would far surpass anything yet applied to this region. The diagram of the spectrometer is shown in Fig. 1.

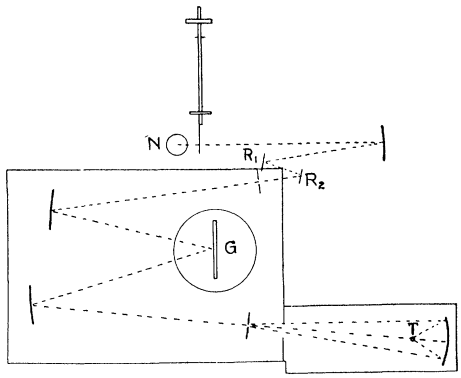

Fig. 1. Spectrometer arrangement for observing the reflection spectra.

Apparatus

The source of light, N_1 was a Nernst glower, run at 0.8 amp. from a storage battery. This light is interrupted periodically by the pendulum of the resonance radiometer; the period of which is adjusted to be equal to twice the optimum period of response of the first thermocouple. The light is then admitted to the spectrometer periodically with the time of admission exactly equal to the time of darkness, and the slit is alternately illuminated and darkened. Light from the Nernst glower is reflected from a concave mirror on to the first quartz plate R_1 which is covered with a layer of MgO. This plate acts as a filter and purifier. The beam is then reflected from a second plate of quartz R_2 to the first slit. This second plate is set in place with a strong spring so that it may be replaced exactly by a silvered surface. The beam then passes through the spectrometer and is focused upon the receiver of the first thermocouple. The grating used was a 5 inch, 1312.5 lines per inch echelette prepared at this University by Professor Wood, and ruled to throw its blaze, or maximum concentration, at 8.5μ. The thermocouple was a Bi-Sb·· Bi-Sn

splashed filament type, with a silver receiver which was covered with bismuth black. It was mounted behind a rock-salt window and evacuated to a non-conducting vacuum. The grating had a dispersion ratio of 570; i.e. at a scale distance of 3.2 meters, 1 mm represented 50A. The calibration was done by means of the higher orders of the 5461 line of the mercury arc, and was accurate to 15A. The slits of the apparatus were 0.07 mm wide, yielding an effective "slit-width" of 15A. This width of slit is the most advantageous in this region both for resolution and intensity giving a resolving power of 80 percent of the theoretical value, or about 4000.

Inasmuch as the resonance radiometer has been recently described in detail, it will be necessary here to outline only its salient points.[2] The first thermocouple is connected to tuned vibration galvanometer 1, which is so constructed that it is highly underdamped with a half-period equal to the time of response of the thermocouple. Galvanometer 2 is identical with 1 except for a device permitting accurate tuning with 1. The two galvanometers and the pendulum are kept in as exact tune as possible. The amplifier light source is a tungsten filament bulb burned at about 3.6 amp. from a storage battery. It lies behind the first grid R_1, and is focused on the concave mirror of galvanometer 1. This mirror then throws an image of R_1 coincident upon a second grid R_2, which is identical with R_1, except that it has a double spacing at the center. Then the beam of light which is transmitted by R_2 is broken into two halves, which are exactly out of phase as regards luminosity. As the mirror of galvanometer 1 rotates, the image of R_1 slides across R_2, darkening one half whilst illuminating the other half. These two beams are then focused by means of a split lens upon a compensating thermocouple T_2, which is in turn connected to the second galvanometer. There will then be two amplifying factors: First, the resonance amplification arising from the periodically interrupted beam falling upon S_1. Secondly, the motion of G_1 causes the image of R_1 to oscillate across R_2, so that the intensity of the light falling upon the junctions of T_2 varies periodically and sets G_2 in turn into a resonance vibration. This second factor may be varied by changing the intensity of S_2. Throughout this experiment the total amplification was kept at about 4000, and deflections which could not be detected on an ordinary high-sensitivity galvanometer ranged as high as 30 centimeters on this instrument.

Method of Procedure

The actual readings were taken as follows: The spectrometer was set for a given wave-length, and the first quartz "purifying" plate was held over a brightly burning ribbon of magnesium. It was coated with a fine, white powder of magnesium oxide. The thickness of the coat was judged sufficient when the whole plate appeared uniform when held against a brilliant back ground. The plate was then placed carefully in its holder, and thereafter left undisturbed. The second quartz plate was put in, and the shutter was removed, allowing the second galvanometer to build up to a maximum. Three or four deflections on each side were noted, with an average deviation, at a scale distance of 1 meter, of about 1 percent. The second quartz plate was

removed, and replaced by a silver surface; the readings were then repeated. The direct ratio of the readings yields the relative reflecting power of quartz and silver. To get the absolute reflecting power, one must correct for the small deviation of the reflection coefficient of silver from that of a perfect reflector. However, this correction is smaller than the experimental error. Inasmuch as direct ratios are taken, it is unnecessary to take any account of water vapor or any other absorbant present in the air path. The magnesium oxide[7] was used with the hope of cutting out the higher orders of lower wave-lengths; an ideal filter would be one which was opaque to 4μ, and perfectly transparent from 7.5 to 9.5μ. As a test for the usefulness of MgO, the first slit was covered by a piece of thin cover-glass, which transmitted fairly well up to 4μ, and which

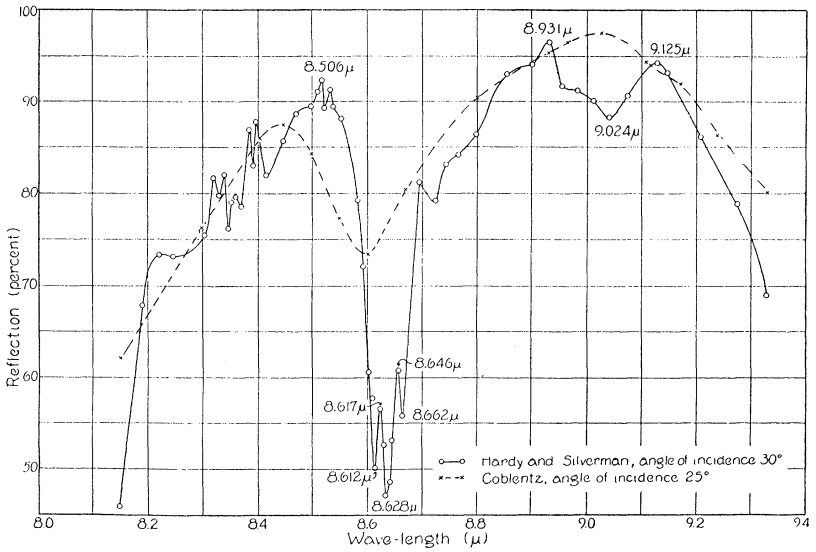

Fig. 2. Reflection spectrum of crystalline quartz. Circles, Hardy and Silverman; crosses, Coblentz.

was opaque to 8μ. With the grating set for 8μ, and with two *silvered* plates in place, one covered with the oxide, no resonance deflection was detected. In passing, it might be noted that even with such a high amplifying factor, the zero unsteadiness was never greater than 2.0 cm in the daytime, and usually around 5 mm at night. Moreover, with an unsteadiness of 2 cm, it was possible to measure deflections of less than four centimeters to an accuracy of a millimeter. In general the deflections were of the order of ten or more centimeters so that the resultant ratios shown on the curves are judged to be accurate to about one percent. The curves shown are the results of several sets of observations which were found to agree among themselves exceedingly well.

[7] A. H. Pfund, Phys. Rev. **36**, 71 (1930).

The position of the grating was checked several times and the wavelengths plotted are accurate to 0.0015μ.

Results

The spectrum of crystal quartz shows a rapidly increasing reflectivity at 8.1μ. Between 8.3 and 8.4 there is considerable fine structure, and a sharp maximum at 8.506μ. The minimum immediately following is triple, showing dips at 8.612, 8.628 and 8.662μ respectively; and peaks at 8.617μ and 8.646μ.

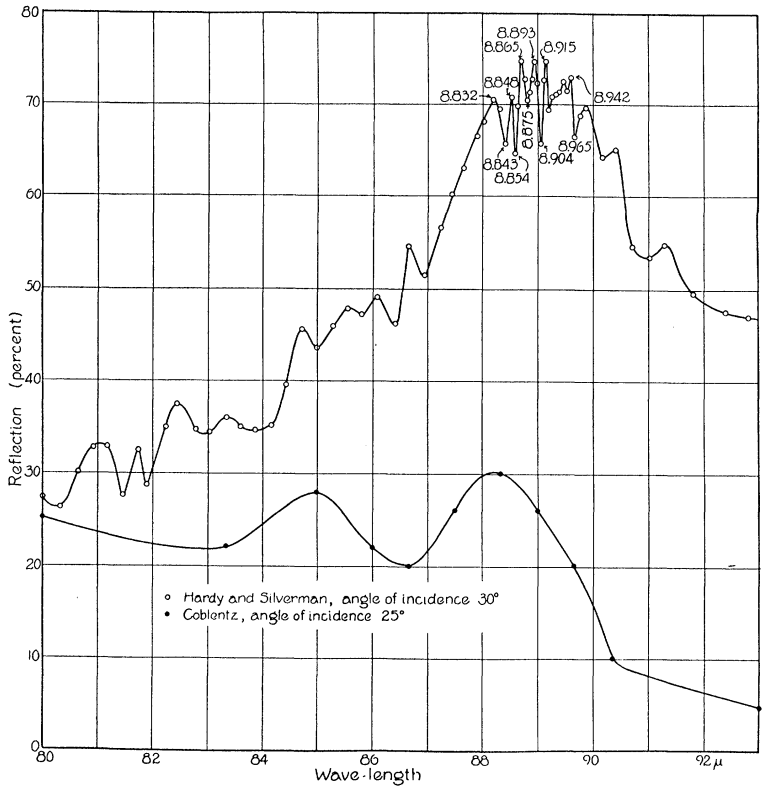

Fig. 3. The reflection spectrum of fused quartz. Circles, Hardy and Silverman, squares, Coblentz.

There is a rise to a maximum at 8.931, a small decline to 9.024 and a shorter rise to 9.125. Following this last maximum the intensity falls off steadily. Dr. A. H. Pfund had previously found indications of the structure at 8.4μ, although he was not able to resolve it, and also of the dip at 8.9μ.

The spectrum of fused quartz is less regular, and nowhere shows any of the regular smoothness attributed to it in the literature.[8,9] It rises from 16 percent

[8] Schaefer and Matossi, "Das Ulrtarote Spektrum," p. 317.

[9] J. Lecompte, "La Spectre Infrarouge," p. 145.

at 7.870μ in a series of staggers to a set of sharp maxima and minima. Maxima appear at 8.832, 8.865, 8.893, 8.913 and 8.942μ. The minimum at 8.904 is particularly sharp. The intensity on the long wave side again falls off in a step-wise fashion.

The resonance radiometer has shown itself to be quite practical in its application to work in infrared spectroscopy. Although it has the disadvantage that in its present form it is not practical to use it as a direct recording instrument, its freedom from unsteadiness and stray disturbances and its reliability in the matter of reproducing readings more than offset its faults.

In conclusion, the authors wish to express their appreciation to Professor A. H. Pfund of this University, whose aid and many suggestions have been invaluable.