Notes on the Preparation of Reststrahlen Plates and the Reflection Power of Powders

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Reststrahlen plates were prepared (1) of natural crystal surfaces, (2) of surfaces of fused and then recrystallized substances, and (3) of pressed crystal powders. The reststrahlen resulting from reflection upon these three types of surfaces were compared. The wave-length of the reflection maxima was in each case the same. The intensity of the reststrahlen of type (2) was the same as that of type (1). The reflection power of the third type, the pressed powders, depended inversely upon the size of the particles of powder. It is shown, that (in cases where no pressure is applied to the surface), the reflection power of a powder decreases as the particle size is made smaller.

Reststrahlen are used in some manner or other in a very large percentage of all infrared investigations. The most modern methods do not make use of them as they were used originally by Rubens and Nichols. Today, the reststrahlen usually play a secondary role, a grating serving as the actual means of producing monochromatic radiation. Use is made of the selective reflection of the crystals in diminishing the intensity of those wave-lengths which are much shorter than the ones being studied, and so reducing the effect of the overlapping orders of the grating. Reststrahlen are thus used as a reflection filter.

It is the chief purpose of this paper to discuss the most efficient method of preparing reststrahlen plates of those substances which may not be had in large single crystals. In such cases, two other types of plates have very frequently been used. Either the substance has been fused, allowed to recrystallize and then ground and polished, or, first powdered and then pressed against a metal mirror surface. By use of a wire grating spectrometer of the type often described in this and other journals, the relative merits of plates of these three types were compared. Particular attention was given to the reflection powers of the various surfaces, or, what amounts to the same thing, to the intensities of the reststrahlen resulting from reflection upon the different surfaces.

Fig. 1*a* shows the results of measurements upon NaCl plates of all three types. It will first be noticed that in all cases the maximum of the reflected energy is independent of the manner in which the reflecting surface is prepared. The recrystallized plate was marred by two bad cracks upon its surface. Making a slight allowance for these cracks, it is seen from curves (1) and (2) that the reflection power of the micro-

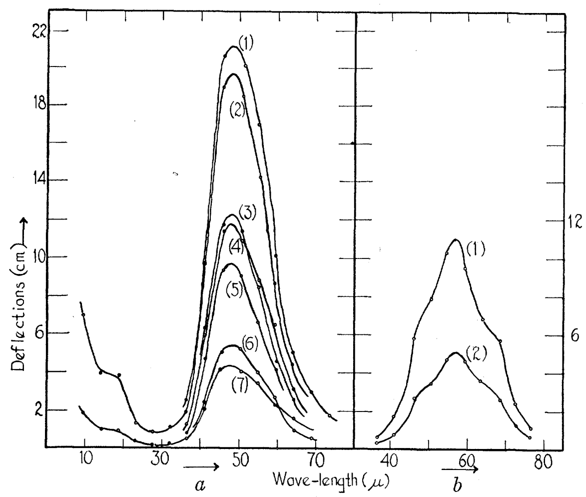

Fig. 1. Reststrahlen from single reflections on NaCl and KCl. Comparison of plates of natural crystals with (1) plates made of materials which had been fused and allowed to recrystallize, and (2) plates made by pressing powdered crystals against a metal mirror.
a. NaCl reststrahlen, (1) natural crystal, (2) fused crystal, (3), (4), (5), (6) and (7) pressed powder.
b. KCl reststrahlen, (1) natural crystal, (2) pressed crystal.

crystalline salt is just the same as that of the single crystal. Curves (3), (4), (5), (6) and (7) show the results for plates of pressed NaCl powder, the size of the powder grains in each plate being different. The average particle in the (7) powder was about 0.1 mm. The others were

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smaller, the finest, those of plate (3), being obtained by scraping the surface of a natural crystal with a razor blade. The reflection power[1] is seen to depend inversely upon the size of the grains of the powder. A still finer powder can probably be obtained by drying a precipitate of NaCl.

No other crystal was examined in all three forms. However, the following results seem to show that the facts found in the case of NaCl hold in general.

Fig. 1*b* shows a comparison between a single KCl crystal and a plate of pressed KCl powder. Here the particle size was approximately the same as in curve (5) for NaCl, and the results are analogous to those shown in Fig. 1*a*.

An old plate of pressed TlCl powder (also one of Rubens plates) was compared with a plate made of new TlCl powder. The former, which with time had taken on a dull gray color, gave values about 20 percent higher than those of the new plate. Since the pressures used in preparing the two plates were exactly the same, namely, 300 atm. per cm^2, it is believed that the differences in reflection power are due to corresponding differences in the particle sizes. Ruben's powder was a dried precipitate and was surely much finer than my powder which was ground in a mortar and pestle.

It follows then, that the reststrahlen from a microcrystalline plate are just as strong as those from a single crystal. Further, the intensity of the reststrahlen from plates of pressed powders varies inversely as the size of the particles, reaching values approaching those obtained from the other two types of plates only if the size of the powder grains is extremely small. These conclusions are well illustrated by the fact that Rubens found for NaCl (natural crystal), KBr (microcrystalline plate) and TlCl (pressed from a dried precipitate of TlCl) reflection powers of 82, 83 and 81 percent respectively.

Upon consideration it is clear that the reflection power of the pressed powder plates, as measured by the intensity of the reststrahlen, must depend upon the size of the particles. Imagine a single crystal reststrahlen plate to be broken into 100 parts, and these parts to be placed together to form a mosaic surface. The reflection power drops slightly due to the losses suffered at the cracks. Let this process be continued. The decrease in the reflection power will be proportional to the increase of the non-reflecting portions of the surface so long as the parts of the original plate are still oriented with respect to one another. On decreasing the size of the parts still further, let us assume that a random orientation begins to appear, the surfaces of the reflecting particles no longer lying in one plane. The reflection power drops still further due to the loss caused by diffuse reflection. Part of this loss is due to the radiation which is reflected at angles slightly different from the angle of specular reflection, and so lost as far as the reststrahlen are concerned. Still another part is caused by the fact that a portion of the incident radiation suffers multiple reflection into the crevices between adjacent particles and so is "trapped" and lost. The smaller the particles are made the greater the chances for losses of these two kinds, and so the greater the decrease in the reflection power of the surface. As soon, however, as the size of the particles becomes small compared with the wave-length of the reststrahlen, in the case of NaCl 0.052 mm, a different phenomenon appears and causes the reflection power to decrease more rapidly and indeed to approach zero. The surface does not reflect in spite of the fact that, as the particles become very much smaller than the wave-length, the relative degree of its "polish" is increased. If the powder is absolutely dry and each small grain acts as a unit, one might imagine that the resulting surface would reflect just as a slightly roughened crystal surface. Usually the grains group themselves and form a very rough fluffy surface. Even if this were not the case, however, such a "polished" surface of very finely divided powder would not reflect well.

In all reflection phenomena it is the surface and a very thin layer of matter approximately one wave-length thick which play the most important roles. The effective extinction coefficient must be such that the amplitude of the incident wave is reduced almost to zero within this thin layer of the substance if a high degree of reflection is to take place. In fact, if the matter within a thin

[1] By reflection power we shall mean the intensity of the reststrahlen or the specularly reflected energy, diffusely reflected radiation not being considered.

sheet of highly reflecting metal be distributed throughout a much thicker layer (by some process such as powdering the metal very finely) the resulting material becomes very transparent and a poor reflector.[2] It will be remembered that various metals such as Bi, Pt, Sb and others, when very finely divided are found to be "black" and do not reflect at all. For this same reason a gas will not reflect, while if the density is increased until the same substance is a liquid, reflection is found. (Pfund has shown that if bismuth-black is gathered in a small pile, and then pressed into a sheet, it becomes highly reflecting. Soot behaves in the same manner.)

The surface of our finely powdered reststrahlen plate should reflect highly then only if the matter within it be compressed until there are enough particles within a layer one wave-length thick to yield an effective extinction coefficient high enough to cause the amplitude of the incident radiation to be greatly reduced.[3] This is exactly what we do when we press the powder into plates as described above. The finer the particles the easier it is to press them into good contact, the less the chance for trapped radiation, the higher the degree of polish of the surface and so, the higher the resulting reflection power. It should be interesting to vary the amount and the time of application of the pressure used in forming the plates. In all the cases measured here 300 atm. per cm^2 was applied for about 3 minutes.

I wish to thank Professor M. Czerny for the use of his wire grating spectrometer.

[2] Phil. Mag. **24**, 440 (1913), Wood has shown that finely divided copper is very transparent to the long waves. "The same amount of metal, in fact a much less quantity, in the form of a uniform film would be absolutely opaque to the heat waves, for we know that the reflecting power of a metal is practically 100 percent for all waves longer than about 10μ."

[3] Phys. Rev. **39**, 64 (1932), Pfund and Silverman showed that quartz, calcite and ammonium chloride when finely powdered do not reflect in their regions of metallic reflection. These powders were not pressed.