Effect of Particle-Size on the Quantitative Determination of Quartz by X-ray Diffraction

A GEIGER–MÜLLER counter X-ray diffractometer has been built in this laboratory[1] for the quantitative analysis of mineral dusts. One of the first tasks of this apparatus was the measurement of quartz content in airborne dusts, lung residues and similar material arising in silicosis research. It was therefore necessary to examine the accuracy with which such determinations can be made, and in particular the effect on their accuracy of the particle-size of the quartz.

Closely graded size-fractions were prepared by repeated sedimentation or centrifuging; their size-distributions were determined by optical or electron microscopy, and the mean size $\bar{d}$ of each fraction expressed in the form $\bar{d} = \Sigma n_d d^3 / \Sigma n_d d^2$, where n_d is the number of particles in a small size-range of mean diameter d. The peak intensities of the $11\bar{2}2$ line of quartz (spacing 1·82 A.) are shown as a function of $\bar{d}$ in Fig. 1; they reach a maximum at $\bar{d} = 2\mu$ approximately, and fall off for $\bar{d}$ both larger and smaller than this value. The lower intensities for larger particle-sizes are to be expected as a result of extinction; those at smaller sizes are attributed to the existence of the 'amorphous layer' previously reported[2]. The broken part of the curve refers to samples from which an attempt has been made to remove the 'amorphous' layer by etching with hydrofluoric acid. (The sizes of the etched samples were determined afresh by microscopy.)

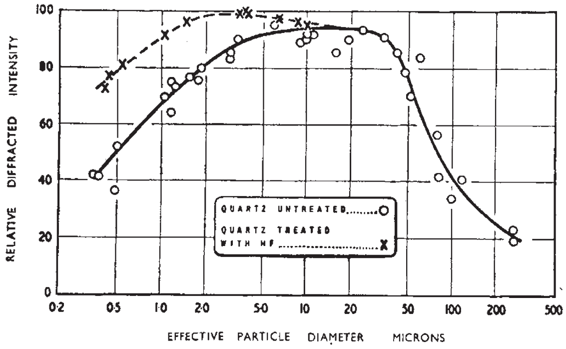

Fig. 1

The effects of this 'layer' have been since studied in detail, and results to be published elsewhere suggest that it is not a clearly defined amorphous shell surrounding perfectly crystalline material, but rather that a gradual increase in crystallinity occurs from the surface of a particle towards the interior. This idea is confirmed by the fact that for particles less than 0·5μ in size the full intensity of the diffraction line is not reached after etching; these particles are presumably too small for the crystal lattice to be undisturbed even at the centre. A few measurements, in which integrated areas of diffraction lines were measured, confirmed these observations; the total reduction in integrated intensity at small particle-sizes is somewhat less than that in peak intensity, since the peak measurement is affected to some extent by particle-size line-broadening; but it is not possible to account for the whole reduction in peak intensity in this way.

Thus in order to make accurate determinations of quartz by X-ray diffraction, it is necessary to know its particle-size distribution and, possibly, whether it has received treatment equivalent to etching. In the size range 1–20μ, the error introduced by ignoring these effects is of the order of 12 per cent; for correlation with medical studies this error is insignificant.

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Unusual Waveguide Characteristics associated with the Apparent Negative Permeability obtainable in Ferrites

B AND H in a magnetized ferrite material are related, not by a scalar permeability, but by a tensor of the form:

$$\left\| \begin{matrix} \mu & -jk & 0 \\ jk & \mu & 0 \\ 0 & 0 & \mu_z \end{matrix} \right\|$$

where z is the direction of the magnetization[1]. If a plane wave is propagated along the z direction and is circularly polarized, the permeability associated with the transverse components in the xy plane assumes the form:

$$B_t = (\mu \pm k) H_t$$

where the sign depends on the direction of the circular polarization. In many ferrites at 10,000 Mc./s., k is greater than μ and the effective permeability for right-handed polarization about the field direction is negative. The propagation constant, which is proportional to the square root of the permeability, is imaginary and the wave is evanescent.

However, an interesting case occurs if such a wave is generated in a waveguide. In order that the electric field at the wall should be zero, a transverse variation of field arises, and for H-modes this is associated with the longitudinal permeability μ_z, which is of the order unity and is positive. If it is assumed that the transverse permeability $(\mu - k)$ is unaffected by the guide walls, which is qualitatively justified by the rigorous theory of Suhl and Walker[2], the propagation constant in the ferrite, γ_g, follows from the equation:

$$\frac{\gamma_g^2}{k-\mu} = \frac{\gamma_t^2}{\mu_z} - \gamma_0^2\,\varepsilon$$

where γ_0 is the free-space propagation constant and γ_t is inversely proportional to the size of guide, and ε is the dielectric constant of the ferrite. For a waveguide of very large size γ_g is imaginary, and the wave is evanescent. As the size of guide is reduced, so the attenuation decreases until a critical point is reached