

30° C., and at 11 min. for ageing at 0° C. The magnitude of the peak at 0° C. is some 4 per cent higher than that of the peak at 30° C. Fig. 2 shows the small-angle scattering obtained from a specimen 0.005 in. thick of the same alloy and given treatments identical with the specimens used for resistance measurements. Ageing of the specimen was halted at the time of the peak in resistance by bringing it to -195° C. (liquid nitrogen), at which temperature the X-ray data were obtained. Balanced filters were used to obtain monochromatic copper $K\alpha$ radiation; step-scanning was employed due to the long exposures required. (The results are plotted as $\log I$ in counts/sec. versus ϵ^2 , where ϵ is the scattering angle, 2θ , in radians.) The slope of the linear region to the right of the maximum gives the radius of gyration, R_g , of the spherical zone⁶. R_0 , the zone radius, is given by $R_0 = (5/3)^{1/2} R_g$. R_0 is an average which emphasizes the largest zones, and since the scattering curves are not linear to the right of the peak (Fig. 2) there must be a range of sizes. The average radius can be expected to be about $\frac{1}{2} R_0$ for any reasonable distribution; this is 5 Å. for both the 0° C. and 30° C. resistance peaks. The maximum probable uncertainty in radius due to counting statistics and stability of X-ray output and counter was about 3 per cent.

The resistance maxima are then associated with zones of an average size of 10 Å. in diameter. Matyas's equation applied to the case investigated predicts about 7 Å. for the zone diameter. This is in reasonable agreement with what has been observed here experimentally; the results confirm the relation between the resistance maximum and zone size.

Re-ageing after reversion yields sizes closely the same as those quoted above, despite the fact that the ageing kinetics are different. These results will be part of a more detailed publication.

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Porous Nickel Oxide

IN the course of an investigation of the oxidation properties of nickel strip used in the manufacture of electron-tube cathodes, a phenomenon was observed which may be of general interest.

Two samples of nickel in the form of strip 0.002 in. thick were oxidized by heating in air for 5 min. at 1,000° C. Dilute nitric acid was used to dissolve the metal and provide small pieces of coherent oxide film about 0.0001 in. thick. Under microscopic examination, the film from one sample was found to be apparently homogeneous and entire (Fig. 1a), while the other contained pores or holes about 1 μ in diameter (Figs. 1b, c). The only distinguishing characteristic of the strip which produced this porous oxide film was that it contained about 0.02 per cent aluminium; the other strip contained about one-tenth of that amount.

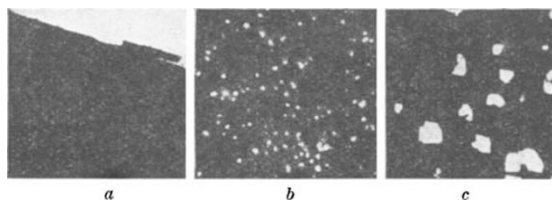


Fig. 1. Electronmicrographs of nickel oxide films formed on: (a) nickel containing 0.002 per cent aluminium ($\times c. 390$); (b) nickel containing 0.02 per cent aluminium ($\times c. 390$); (c) nickel containing 0.02 per cent aluminium ($\times c. 1,880$)

It is possible that small crystals of alumina are formed similar to those found by Allison and Samelson¹ when they oxidized nickel in wet hydrogen. These crystals may have fallen out of the nickel oxide layer when the nickel was dissolved, leaving the sharp-cornered holes in the oxide.

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CHEMISTRY

Infra-Red Spectra of Water-soluble Carbonates

INFRA-RED spectra of some carbonates have been reported by Hunt, Wisherd and Bonham¹, Underwood, Toribara and Neuman² and by Miller and Wilkins³. Hunt *et al.* suggested that the position of the band near 11 μ was a function of the atomic weight of the cation, and also pointed out that Miller and Wilkins's work substantiated this suggestion with the exception of lithium carbonate. We have evidence that this is not so. Further, Underwood *et al.* reported the 11 μ band for sodium and potassium at different positions from that reported by Miller and his workers. Although they indicated their results were in general agreement with Miller and Wilkins, no attempt to point out or explain this discrepancy was made. Finally, the role of water in influencing both band positions and broadness appears to have been neglected in interpreting spectra of water-soluble inorganic compounds and this will be discussed here.

We find that for the soluble carbonates (sodium, potassium, rubidium and caesium), this band appears at 11.4 μ when they are in the anhydrous state and 11.6 μ when they are hydrated. The band is therefore not a function of the atomic weight of the cation (at least for the soluble carbonates) but of the degree of hydration. As the degree of hydration is increased the band decreases in intensity and disappears when complete solution is obtained. No attempt was made to study which hydrate exists when the band shifts from 11.4 to 11.6 μ , but it has been found that only slight traces of water are required, especially for the more soluble carbonates of rubidium and caesium, to produce this shift. Decius⁴ has related this position of the band for calcium carbonate to its crystal structure.

The band at 6.95 μ for the anhydrous carbonates is shifted to 7.2 when complete solution is obtained. The intermediate states of hydration, however, give both bands, and, depending on the concentration of