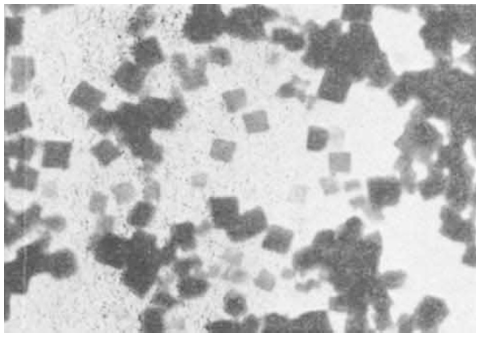

Fig. 1. Precipitates formed in an Fe-3 per cent Si alloy after nitriding and furnace cooling from 640°C. Extraction replica. (Electron micrograph ×20,000)

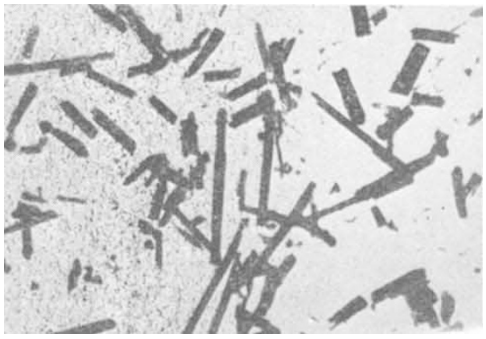

Fig. 2. Precipitates formed in an Fe-3 per cent Si alloy after nitriding and furnace cooling from 640°C., and then further annealing at 820°C. Extraction replica. (Electron micrograph ×2,000)

The results strongly suggest that the cubic-shaped particles are the new precipitate and demonstrate that it transforms within the metal specimen to α-Si_3N_4 when the temperature is increased.

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Identification of the High-Temperature Constituent in Mild Steel Surface-Hardened by Carbo-Nitriding

During an investigation on the heat treatment of mild steel in raw town-gas and ammonia atmospheres[1] an unidentified constituent was observed in the surface layers which appeared as a dark-coloured phase visible in the unetched condition. This phase is unstable at room temperature and can be eliminated by slow cooling or reheating. With the limited information available at the time the constituent was presumed to be an iron-carbon-nitrogen compound; positive identification has not been possible until now.

A method of removing thin oxide films from metal surfaces[2] has been adapted for stripping thicker scales [3,4]. A thin plastic film is applied to the surface and the specimen is immersed in an oxygen-free solution of iodine in alcohol, which penetrates discontinuities in the plastic and oxide films and dissolves metallic iron. When the surface deposits have been undermined sufficiently, the plastic film and the oxide particles adhering to it can be removed for X-ray examination. This technique has been applied to machined surfaces, and both sulphide inclusions and cementite lamellæ have been extracted.

The method was used to extract the iron-carbon-nitrogen constituent from a mild steel rod that had been treated for 50 min. at 800°C. in an atmosphere containing 10 per cent ammonia. Before the iodine extraction the specimen was shot blasted to remove any adherent oxides; it was then coated with a plastic consisting of polyvinyl chloride/acetate resin ('Rhodopas *AXCM*') in acetone[3]. After stripping the plastic film was dissolved in hot acetone and the residue collected by centrifuging. When the residue was completely free from plastic it was dried and a small portion was mixed with canada balsam, coated on a hair and mounted in a 19 cm. X-ray powder camera. The photograph obtained with Co-K radiation (Fig. 1) was measured and could be indexed (Table 1) as an hexagonal structure, having lattice parameters $a = 2{\cdot}636$ A. and $c = 4{\cdot}316$ A.

Table 1

d	I	hkl
2·284	m	100
2·158	s	002
2·017	vs	101
1·567	m	102
1·318	m	110
1·218	m	103
1·126	m	112
1·103	m	201

This structure is the same as the ε-iron nitride (Fe_3N) reported by Jack[5]. The parameters are somewhat lower than any observed by Jack but fit well on an extrapolation of his curves to 4·0–4·5 per cent nitrogen (by weight). Jack has also shown[6] that nitrogen in the ε-phase can be partially replaced by carbon and that this reduces the lattice spacing; this indicates that the observed parameters can be accounted for with rather less extensive extrapolation by the presence of carbon. Iron nitrides are notoriously difficult to isolate by preferential solution of the matrix and the successful extraction of this constituent lends support to the inference that it has been stabilized by carbon, and can be identified as ε-iron carbonitride.

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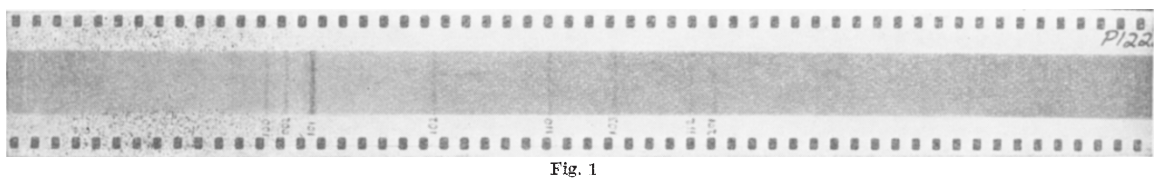

Fig. 1