These whiskers were removed, but after an additional few weeks needle-shaped crystals were again formed. This process has been repeated up to now, about one and a half years after the sample was taken out of the furnace. At present, however, the crystal growth seems to have almost ceased.

Chemical analysis of the sample on which the crystals grew gave the following result (weight per cent): iron, 82.4; silicon, 14.5; carbon, 2.85; calcium, 0; nickel, manganese, cobalt, vanadium, copper, titanium—minor impurities.

A metallographic investigation showed clearly that this substance contained only one ferro-silicon phase, the  $\alpha$ -phase. The carbon appeared as a separate graphite phase.

The crystals forming the whiskers were investigated by X-ray analysis. From the powder diagrams, *d*-spacings were calculated. They were in almost perfect agreement with the X-ray lines given by Popper and Ruddlesden<sup>1</sup> for  $\alpha$ - and  $\beta$ -Si<sub>3</sub>N<sub>4</sub>. Both allotropic forms were present in the crystals investigated.

The results of a quantitative chemical analysis of the crystals correspond to the composition  $Si_{3.04}N_4$ . Earlier investigations<sup>2,3</sup> describe cases where

Earlier investigations<sup>2,3</sup> describe cases where  $Si_sN_4$  was formed during, for example, the nitriding process for case-hardening of steels containing small amounts of silicon. But in all cases the nitride was formed at temperatures not lower than 250° C.<sup>4</sup>

The results reported here, however, clearly demonstrate the formation of  $\alpha$ - and  $\beta$ -Si<sub>3</sub>N<sub>4</sub> on a lump of a ferro-silicon alloy even at room temperature. The kinetics of this process will be discussed elsewhere.

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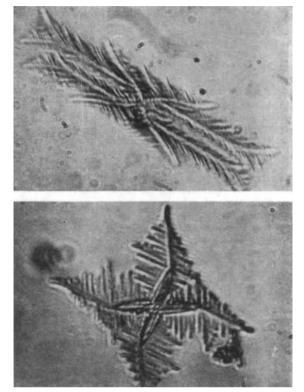
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## CRYSTALLOGRAPHY

## Some Features of the Precipitation of Barium Chromate by an Extra-slow Technique

In the course of experiments concerning the preparation of very pure compounds, the precipitation of barium chromate was chosen as a model. S. Rykowski and I found<sup>1</sup> that a very slow precipitation with dilute solutions produces a precipitate which contains very minute crystals known as 'crystal skeletts'.

Continuing this work I have developed a technique of extremely slow precipitation, and I have observed some interesting features as regards the shapes of the crystals (see Fig. 1). A precipitate was obtained by addition of a dilute barium chloride solution to a large volume of potassium chromate solution and steady and vigorous mixing. After some hours minute crystals could be seen under the microscope ( $\times$  720-2,000). These crystals showed strikingly



Figs. 1 and 2

perfect symmetry, and some had regularly curved shapes. Besides the X-shaped crystals, very regular hexagonal plates and other more complicated forms appeared.

When ammonium chromate instead of potassium chromate solution was used, crystals of similar shapes were formed. Addition of potassium chloride (150 gm./l.) to the solution of potassium chromate produced exclusively crystals of the hexagonal type.

When a mixture of barium nitrate and silver nitrate (10: 1) was added to the solution of potassium chromate X-shaped crystals were formed. These crystals consisted of two parts of different shape and colour. It is assumed that the larger one is formed of barium chromate (pale yellow) the other one being silver chromate (dark brown) (see Fig. 2).

Barium sulphate and barium phosphate precipitates also showed interesting shapes of similar character.

Details of this work, which is being continued, will be published elsewhere. I wish to thank Mr. L. Trojanowski for his assistance.

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<sup>1</sup> Adamski, T., and Rykowski, S., Polish Acad. Sci., Inst. Nuclear Res. Rep. 91/4 (Warsaw, 1959).

## Retention of Carbon Dioxide Bubbles on Calcite Etch-Peaks

THE etch-patterns on cleavage faces of calcite which have been reported<sup>1-3</sup> bear some interesting similarities and dissimilarities to etch-patterns obtained in this Laboratory. Watts etched calcite for 30 sec. in 1 per cent nitric acid and obtained five-