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The Observation on the Phase Transformation of Cobalt and Cobalt-Nickel Alloys in Thin Sections by Means of Electron Diffraction and Electron Microscope

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By using thin sections prepared by electropolishing, the phase transformation in cobalt and cobalt-nickel alloys from h.c.p. to f.c.c. was directly observed by both heating and cooling inside an electron microscope.

Cobalt and cobalt-nickel alloys undergo a phase transformation from hexagonal close packed to face-centred cubic lattice by heating-up. By cooling again to room temperature, the hexagonal phase reappears, but in most cases the restoration is not complete. Stacking faults were found both in h.c.p. and f.c.c. crystals of cobalt by means of X-ray diffraction and electron microscopy.[1] But, an electron-microscopical, dynamical observation on the phase transformation of cobalt, which enables one to discuss sufficiently the transformation mechanism, has so far not been made. In the present study, electrolytically thinned specimens were prepared from recrystallized cobalt and cobalt-nickel alloys, and the transformation was directly observed by both heating and cooling inside an electron microscope.

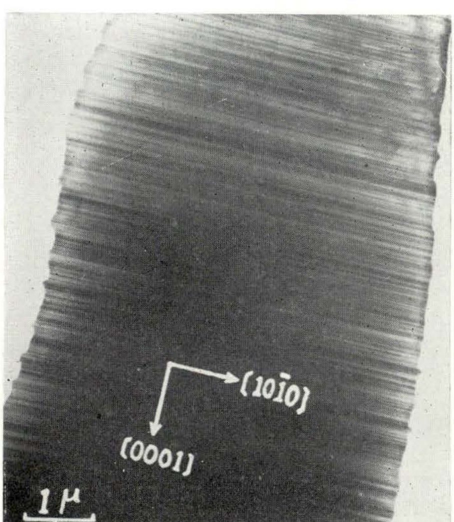


Photo. 1. Electron micrograph of a 90% cobalt-nickel specimen annealed at 1000°C and slowly cooled, taken at room temperature.

Photo. 1 shows an electron micrograph taken from a 90% cobalt specimen annealed at 1000°C, followed by slow cooling. In this micrograph, the basal plane of the hexagonal crystal is nearly perpendicular to the film plane ($(\bar{1}2\bar{1}0)_{h.c.p.}$// film plane). A large number of lines running parallel to the basal plane are observed, their width varying from less than one hundred to several hundred Å. An electron diffraction pattern of such a specimen consisted of spots due to h.c.p., f.c.c. and twin crystals, and strong, sharp streaks passing diffraction spots as well as the direct spot were observed along the direction parallel to the [0001] axis, as shown in Photo. 2. Some of these streaks which are comparatively short, e.g., the one passing the (10$\bar{1}$2) spot, seem to have been caused by

Photo. 2. Electron diffraction pattern corresponding to the specimen in Photo. 1. Diffraction spots due to f.c.c. crystal are indicated by arrows.

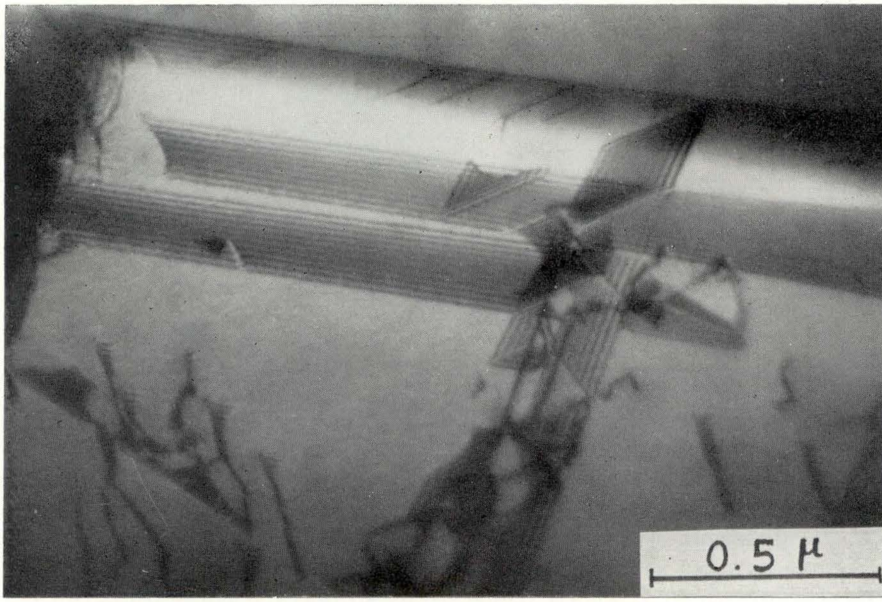


Photo. 3. Electron micrograph of a 70% cobalt-nickel specimen annealed at 900°C and slowly cooled, taken at room temperature.

stacking faults, but the others including that passing the direct spot are longer and are thought to have occurred either from irregularity in the lattice spacing along the hexagonal axis or from very thin layers which are incoherent with each other and parallel to the basal plane.

Purely hexagonal grains with the same orientation as in Photo. 1 were sometimes found, when the specimens were annealed from 300 to 380°C in 200 hours, the grain size being about 10μ. In this case, however, such lines as shown in Photo. 1 were hardly seen on the electron micrograph, and neither f.c.c. spots nor streaks passing diffraction spots were observed on the diffraction pattern. During the heating of such specimens in the electron microscope, a momentary change of the image contrast was observed above the transformation temperature. For example, a line appeared first in the middle of the grain and then it swept one side of the area of this grain for about 1 second. Such a change seems to indicate the transformation from h.c.p. to f.c.c. form. The above lines were intermittently and frequently formed during the cooling. This phenomenon may reveal the incomplete restoration of the hexagonal crystals. The electron micrograph and diffraction pattern similar to Photos. 1 and 2 were obtained after the cooling.

When the basal plane of the hexagonal crystals was inclined from the perpendicular position, fringe-like contrasts were seen as shown in Photo. 3. These contrasts seem to

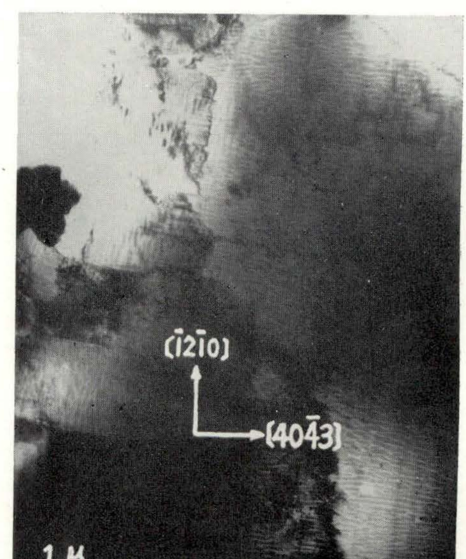


Photo. 4. Electron micrograph of a 90% cobalt-nickel specimen annealed at 300~380°C for 200 hours and slowly cooled, taken at room temperature.

be caused by stacking faults. Photo. 4 was taken from a 90% cobalt specimen. The basal plane was also inclined from the perpendicular position in this grain, but no lines were visible except the pattern of magnetic domain walls. Photo. 5 shows the diffraction pattern corresponding to Photo. 4. During the course of heating, an abrupt formation of many lines covering a whole area of the grain took place above the transformation temperature. Photos. 6 and 7 show the micrograph and diffraction pattern taken from the same grain as before just after the abrupt change. This change indicates the transformation from h.c.p. to f.c.c. form. During the cooling, however, contrast changes due to the inverse transformation were not observed, and the diffraction pattern consisted mainly of the cubic spots.

When the basal plane was parallel to the film plane, some dislocation contrasts were observed, as shown in Photo. 8 which was obtained from a pure cobalt specimen. During the course of heating, a very quick movement of dislocation was frequently observed above the transformation temperature. The dislocations moved from the interior of the grain to the boundary and backwards along the [110] direction of the cubic crystal in this case. It should be noted that,

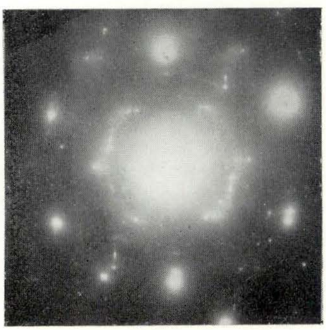


Photo. 5. Electron diffraction pattern corresponding to the grain in Photo. 4. The diffraction spots are due to h.c.p. crystal.

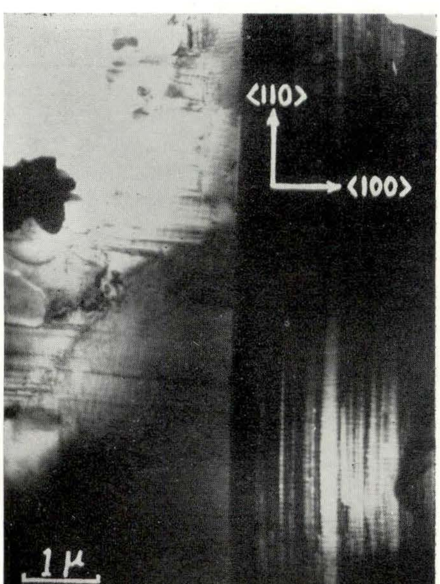


Photo. 6. The same field as in Photo. 4, photographed at about 500°C.

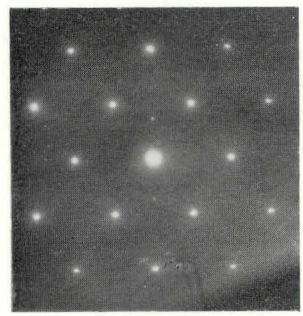


Photo. 7. Electron diffraction pattern corresponding to Photo. 6. The diffraction spots are due to f.c.c. crystal.

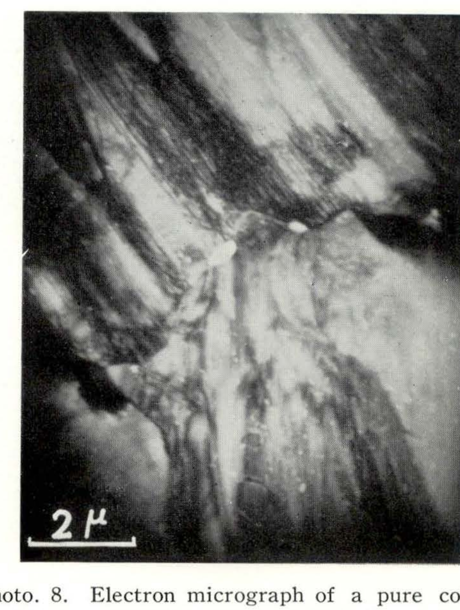
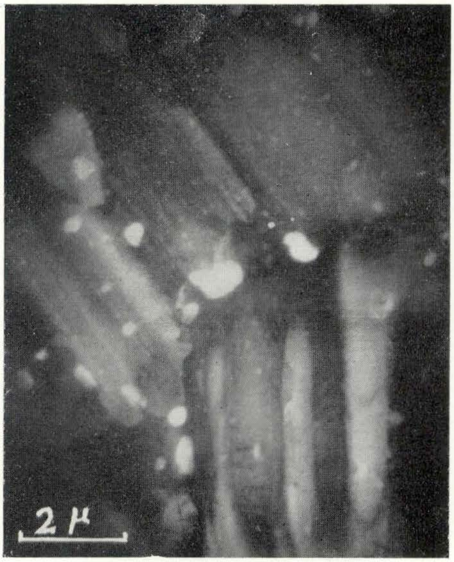
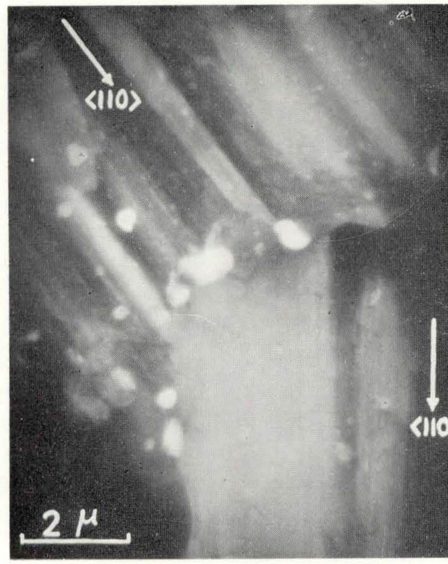


Photo. 8. Electron micrograph of a pure cobalt specimen annealed at 300–380°C for 200 hours and slowly cooled, taken at room temperature. Several grains are seen in this photograph and the basal plane is parallel to the film plane in all grains.



(a)



(b)

Photos. 9 (a) and (b). Electron micrographs of the same field as in Photo. 8, successively taken at about 500°C.

with the movement of the dislocations, white or black broad contrasts having the width equal to the length of dislocation were formed, as shown in Photos. 9 (a) and (b) which were successively taken at about 500°C. These contrasts are considered to be caused by the stacking faults created by the movement of partial dislocation and lying in planes parallel with the film plane.

For the cubic to hexagonal transformation in cobalt, Seeger[2)] proposed a propagation mechanism, consisting of two partials that rotate around a perpendicular screw dislocation. It is uncertain at present, however, whether our results can be explained by this mechanism or not.

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2 A. Seeger: Z. Metallkde. **44** (1953) 247, Z. Metallkde. **47** (1956) 653.