COBALT WHISKERS: THEIR GROWTH, DISLOCATIONS AND PHASE CHANGE

by

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INTRODUCTION

The existence of filamentary and acicular crystals has been realized for quite some time, but it wasn't until 1952 that Galt and Herring (17) demonstrated that some of these crystals possess the strength predicted by theory. These thin filaments were several microns in diameter and a few millimeters in length and were descriptively termed whiskers. Since the discovery of their unusual properties, a variety of whiskers have been grown and extensively investigated.

Whiskers result from a one dimensional growth which may be stress-induced or may be deposited from a supersaturated vapor or solute medium. Most mechanisms proposed for growth have assumed a unique dislocation density and orientation in the whisker.

A dislocation is a line imperfection forming the boundary of a slipped region within the crystal. A Burgers vector signifies the direction and relative distance by which atoms above the slip plane move with respect to those atoms below the slip plane. If the Burgers vector is at right angles to the line imperfection, we have an edge dislocation and if it is parallel to the line imperfection it is called a screw dislocation. Various combinations of edge and screw dislocations give rise to more complicated dislocation structures.

Whiskers grown from vapor deposition are found to grow from supersaturations which are immeasurably small. Frank (16) concluded that whiskers are not perfect crystals but contain screw dislocations which developed during the early stages of their growth and which provide the crystal with permanent growth steps. As material condenses, the step due to the dislocation winds itself into the form of a spiral and one dimensional growth results. Sears (24) demonstrated that for mercury, silver, zinc, cadmium and cadmium sulfide whiskers, the supersaturation of the vapor necessary for whisker growth is less than that required for nucleation of crystal layers on the lateral surfaces of the whisker. A large number of metal whiskers including cobalt have been grown by reduction of their halides. These whiskers have been found to grow at their tip and a vapor transport mechanism seems most probable. However, experimental results (7) indicate that growth does not occur by the direct condensation of metal vapor, and it is most likely that the halide molecules are absorbed on the growing whiskers and reduced at the tip. The reason for the greater rate of reduction at the tip is not clear. Sears, Gatti, and Fullman (26) suggested that these whiskers also contain only axial screw dislocations. In this case the reduction at the tip may be due to the catalytic reduction of the halide at the step associated with the dislocation.

Usually whiskers are straight but spirals, kinks, twists, helices, and many other shapes have been observed. Their cross-sections range from simple to complex geometries. The hollow cross-sections (29) are particularly worth noting. The axis of the whisker is frequently parallel to a major crystallographic direction. Polycrystalline whiskers with a preferred orientation (23) have been reported.

At present it is not known whether the strength of whiskers is due to their small size, their surface perfection, a unique structural perfection, or a combination of these factors. The concept of whiskers being perfect or near perfect crystals (perfection implying here only the absence of extended defects such as dislocations) has created considerable interest because it links the growth and strength of materials.

Eshelby (14) (15) has shown that a whisker containing a single axial screw dislocation should have an appreciable twist about its axis. The magnitude of the twist can be precisely related to the Burgers vector of the screw dislocation by the formula

$$\alpha = K \frac{b}{A} \qquad (1)$$

where b is the Burgers vector of the screw dislocation, A is the cross sectional area of the whisker, and K is a constant dependent on the cross sectional geometry. Dragsdorf and Webb (11), using a high resolution Laue technique, have obtained the lattice twists from the equatorial Laue spots recorded on the film in a cylindrical camera coaxial with the whisker. The twist is given by

$$\alpha = C \tan \rho \tag{2}$$

where ρ is the tilt angle of the equatorial Laue spot and C is a constant dependent on the camera geometry.

It is not known whether most metal whiskers actually have or need a screw dislocation for growth. Palladium (22) and cobalt (grown below transition) (Webb - private communication) are the only metal whiskers that have thus far shown positive evidence for a single axial screw dislocation, while no evidence for lattice twists related to a multiple of axial planar spacing has been observed in zinc, copper, iron, nickel, and manganese (28). The lack of such a lattice twist in these metal whiskers does not eliminate the possibility of operation of a screw dislocation during whisker growth. It is possible that two screw dislocations of equal and opposite sign occur equally spaced from the whisker axis. Such a dislocation pair would not introduce a lattice twist and could glide together and annihilate themselves by combination, thus leaving a dislocation-free crystal. Another possibility is that the dislocation abosrbs trapped vacancies which permit it to climb out of the whisker during or after the growth process.

X-ray measurement of lattice twists is capable of detecting only nonpaired screw dislocations parallel to the whisker axis. Lang (19) and Newkirk (20) have developed a high resolution x-ray diffraction microscopy technique which appears capable of resolving individual dislocations in nearly perfect crystals. The intensity diffracted from regions close to a dislocation is expected to be greater than the intensity diffracted by more perfect regions. However, other defects such as concentrations of impurities, grain boundaries, and surfaces may also cause changes in the diffracted intensity (4).

The phase change in iron whiskers has been investigated by Sears and Brenner (25). They observed that the body-centered cubic to face-centered cubic transition in most of the larger iron whiskers is accompanied by severe kinking and distortion. However, it was possible to cool thin iron whiskers through the transformation temperature without causing distortions. The kinks were due to localized transformations. Brenner (8) observed that cobalt whiskers grown by hydrogen reduction of $CoBr_2$ above the transformation temperature usually contain two phases at room temperature. He examined five crystals and only one had a purely hexagonal structure. The whiskers were straight and not distorted. The presence of the two phases did not affect their strengths.

Cobalt has two modifications in the solid state—face-centered cubic and close-packed hexagonal. It has been found to exist in a two-phase state, whereas according to the phase rule, it should be single-phase. The cobalt transformation is martensitic in nature. It is generally agreed that there is a transition between the two structures between 400°C and 500°C. The transition temperature is more exactly defined for coarse-grained specimen (27). Work with single crystals indicates the transition temperature is about 420°C (18). Henceforth, the close-packed hexagonal and the face-centered cubic phases will be designated as cph and fcc, respectively.

The crystallography of the phase change from fcc to cph involves a very small volume change (0.3%). The crystallographic relations can be summarized by noting that the fcc (111) plane is parallel to the cph (00·1) plane and the fcc [110] direction is parallel to the cph [10·0] direction.

A variety of specimens of cobalt have been examined by various workers—powder (3) (12) (13), sponge (21), filings (21) (27), slivers (27), rods (13) (21), and single crystals (12) (18).

Edwards and Lipson (13) have shown that for powder above transition the cubic form is stable. Upon cooling from above the transformation range the fcc form gradually changed to the cph form, the relative amounts transformed for a given grain size depending only on the temperature and not on the time for which the specimen was maintained at a particular temperature. Powder specimens showed about equal quantities of the two phases at room temperature. X-ray powder photographs of cph cobalt have a mixture of sharp and diffuse lines (12). The powders often contain many growth faults, whereas specimens that have been deformed to complete the martensitic transformation contain mainly deformation faults. Specimens with mixed faulting have also been obtained. Line breadth measurements indicate that the faults are clustered, but results of Fourier analysis of the broadened lines do not lead to the same conclusion (3).

Cobalt rods were found to be pure hexagonal at room temperature before they were heated through the transition (13) (21). After transforming, as the temperature decreased, the amount of hexagonal phase increased much more rapidly for the rods than for the powder. The rods were then found to contain a trace of the fcc phase at room temperature. Broadening of the x-ray diffraction lines from the hexagonal structure was much less for the rods than for the powder; in other words, the probability of stacking fault occurrence was smaller for the rods than for the powder. It has been found that the structure of cobalt below transition temperature depends on its grain-size (13) (21).

Edwards and Lipson (13) indicate that broadening is also shown by x-ray reflections from the planes of single crystals. These reflections show that the underlying irregularities which produce the broadened lines are of a plate-like type perpendicular to the c-axis. They imagined a structure in which occasional faults occur, a sequence ABABAB. . . . changing to BCBCBC. . . . and so on. The resulting regions of perfect crystallization would then be of the required plate-like character. Obviously x-ray reflections such as the (00·1) would not be affected since the (00·1) spacing is maintained throughout the whole crystal.

Kehrer and Leidheiser (18) repeatedly passed cobalt single crystals through the transition point up to 500°C without recrystallization into small grains. In every experiment the same set of (111) planes in the fcc structure corresponded to the (00·1) planes in the hexagonal. X-ray back reflection photographs of the single crystals showed a multiplicity of each of the Laue spots. The multiplicity was attributed to a mosaic type of structure characteristic of small angle grain boundaries with each of the blocks having an orientation only slightly different from its neighbor.

Edwards and Lipson (13) noted that there must be some type of shear mechanism associated with the cobalt transition because the cubic modification of cobalt can be completely transformed to the hexagonal form by moderate deformation at room temperature. Basinski and Christian (5) have shown that a suitable dislocation node may be constructed for the production of a (macroscopically) homogeneous shear. Experimental evidence of macroscopic shear in the transformation has been presented by Anantharaman and Christian (2). They heated a polycrystalline specimen of pure cobalt through the transition temperature and thin martensitic plates appeared; complete transformation was very difficult to obtain.

The work reported in this thesis results from a study of cobalt whiskers grown above the transition temperature. These whiskers were studied since the dislocation structure of many metal whiskers is still in doubt and a complete solution to the cobalt transition problem has not been realized. Work with cobalt whiskers by several investigators indicates that they grow by the vapor deposition mechanism and result from an axial screw dislocation or dislocations. The whiskers after growth contain a lattice twist about their growth axes. This lattice twist allows the lateral surfaces of the whisker to be strain free, while around the immediate area of the screw dislocation there is a highly strained region. The highly strained region and thus the lattice twist may be relieved by torsion, heating, or possible shear resulting from the transformation from the fcc phase to the cph phase. Besides adding to the general information regarding the strength and perfection in crystals such a study could possibly reveal the dislocation structure in metal whiskers and add to the present state of the theory for the cobalt transition.

APPARATUS AND PROCEDURE

Growth

The method outlined by Brenner (6) for hydrogen reduction of the metal halide was used in growing cobalt whiskers. The apparatus is shown in Plate I, Figure 1. It consists of a tube furnace containing a quartz tube. The furnace temperature was controlled within several degrees through a thermocouple connected to a Leeds and Northrup Micromax which fed a variac connected to the furnace.

The thermocouple arrangement, as shown, had the control thermocouple located at the center of the furnace outside the quartz tube. Recording thermocouples were located both inside and outside the tube at the center of the furnace and outside the tube at the end of the material to be reduced.

$CoCl_2 \cdot 6H_2O$ crystals were ground to a fine grain to create a large surface area per unit volume. The crystals were placed inside six porcelain boats (6 cm long, 1 cm wide, and $\frac{1}{2}$ cm deep). The boats were arranged inside the tube three side by side and two deep; this distributed the material over 12 cm of the tube in the center of the furnace.

The H_2O molecules were eliminated from the $CoCl_2 \cdot 6H_2O$ by closing the system, heating to about 300°C and evacuating with a fore pump. A cooled trap was used to collect the water as it was eliminated from the system. This left $CoCl_2$ inside

EXPLANATION OF PLATE I

Fig. 1. Diagram of the whisker growing apparatus.

A. Cross-section of tube furnace.
B. Asbestos packing.
C. Ceramic two-inch tube with coils wound non-magnetically.
D. Quartz tube.
E. Hydrogen inlet.
F. Porcelain boats.
G. Recording thermocouples.
H. Control thermocouple.

Fig. 2. Diagram of tube used for heat treating.

PLATE I

Fig. 1

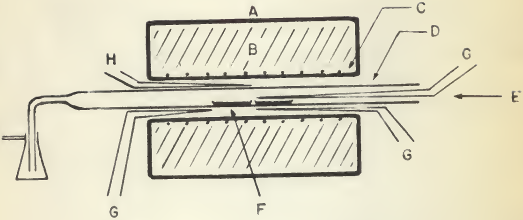


Fig. 2

the boats and the system ready for whisker growth. The furnace was set at a designated temperature and the hydrogen flow was begun. The gaseous products were bubbled through water to separate the hydrogen halide. The frequency of formation of the bubbles served as a measure of the hydrogen flow rate. No attempt was made to obtain an accurate measure of the hydrogen flow.

Runs were made for temperatures from 485°C to 800°C and for times from two to three hours. Upon completion of the run, the system was allowed to cool before removing the boats containing the cobalt and the cobalt whiskers.

Handling of Whiskers

The mounting of whiskers was quite tedious. It was necessary to mount most of the whiskers using a stereo-microscope with a magnification of about 50. Much care was taken not to strain the whisker when loosening its base from the cobalt substrata. The whisker base was fixed to the end of a glass rod by application of Duco cement. The whisker was never touched after it was mounted.

Heat Treatment Apparatus

A separate pyrex tube (Plate I, Figure 2) was used for heat treatment of whiskers at temperatures below the softening point of pyrex (about 500°C). An inert atmosphere was used to prevent the formation of an oxide from coating the surfaces of the whisker.

The system was first evacuated with a fore pump and then filled with argon. This flushing process was continued until the sample was raised to the desired temperature, thus allowing for expansion of the gas. The tube was then completely sealed with the argon at standard pressure.

Optical Microscope Studies

Optical microscope studies were performed using either transmitted or reflected light and magnifications up to 800.

X-ray Studies

A cobalt x-ray tube operating at 30 KV and 10 ma was used in conjunction with a Weissenberg camera in the study of cobalt whiskers. The camera has a cylindrical film holder which is coaxial with the crystal being observed.

All Laue, rotation, and Weissenberg photographs were made with the cylindrical film. The high resolution Laue technique of Dragsdorf and Webb (11) was used to detect the possible twisting of the crystal lattice. The angle of twist per unit length of whisker was related to the angle of tilt of the diffraction spots through equation 2. For the cylindrical camera used, the geometric constant C is given by

$$C = \frac{A + a}{2aA} \qquad (3)$$

where a is the distance from the x-ray source to the sample and A is the distance from the sample to the cylindrical film. The Burgers vector for an axial screw dislocation in a whisker was obtained from equation 1. Laue photographs were also used to study lattice strain and the possible occurrence of multi-crystal whiskers. The whisker axis, line broadening, and line shift were obtained from the rotation photograph. The Weissenberg pattern gave the reciprocal lattice.

High Resolution X-ray Diffraction Microscopy Technique

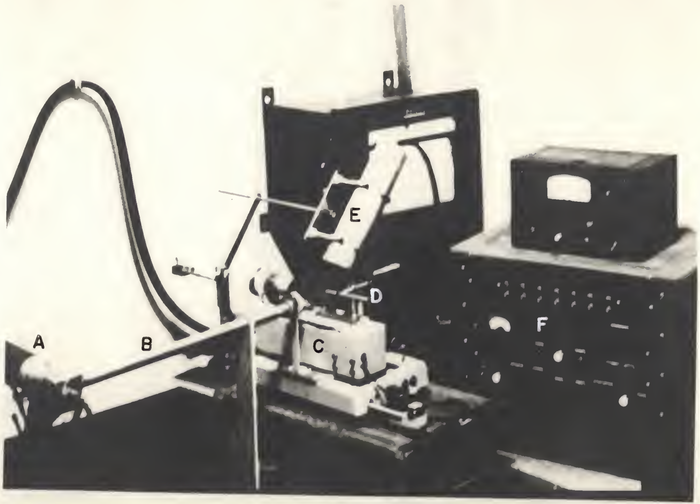
The Berg-Barrett (B-B) technique (4) of detecting lattice irregularities was used (19) (20). Detecting the reduced x-ray extinction with the resulting higher diffracted intensity in the vicinity of lattice imperfections required

EXPLANATION OF PLATE II

Apparatus used for high resolution x-ray diffraction microscopy studies.

A. Cobalt x-ray tube.
B. Slit system.
C. Weissenberg camera.
D. Nuclear track plate holder.
E. Counter.
F. Recording unit.

PLATE II



high resolution through a well collinated x-ray beam. The apparatus is pictured in Plate II.

The parallel beam of x-rays was obtained from a slit system. The slits were mounted at both ends of a one-inch brass tube which was mounted on the head of a cobalt x-ray tube. The slits were separated by 56½ cm and were 1½ mm wide and 6 mm long. A precise alignment of the two slits was obtained by using a special holder, containing the second slit, which fit on the open end of the brass tube. This allowed the second slit to be rotated, raised, or lowered.

The whisker was mounted on the Weissenberg camera and alined in the parallel x-ray beam. A counter, set at the proper Bragg angle for detecting diffraction from a chosen lattice plane, was connected to a recording unit. The whisker was then rotated until the desired plane diffracted the cobalt $K\alpha$ radiation. The whisker was then locked in place.

A nuclear track plate (NTB – 10 micron) was mounted on a specially built plate holder designed to be attached to the Weissenberg camera (see Plate II). The NTB plate was then fixed as close to the whisker as possible so $K\alpha_1$ and $K\alpha_2$ resolution would not be pronounced.

RESULTS

Whisker Growth

Variables affecting the growth of cobalt whiskers were the quantity and particle size of the $CoCl_2 \cdot 6H_2O$ present, the temperature of the furnace and the hydrogen flow rate.

The temperature at which a whisker grew was not accurately known until after the research project had been underway for some time. After improvements had been made on the apparatus a maximum error of 40°C was obtainable for the growth temperature of any whisker.

All whiskers were grown above the transition temperature and were examined at room temperature. The growth temperatures ranged from 485°C to 800°C with the best growth at 600°C.

All of the whiskers grew from previously deposited cobalt. The majority of the whiskers studied were from 1 to 10 microns in diameter and several millimeters in length. The longest whisker grown was 1½ cm and the maximum diameter observed was about 150 microns.

Both single crystal and multi-crystal whiskers were grown. A multi-crystal whisker is here defined as a whisker containing from two to ten crystals, all having a common axis but otherwise having small angle boundaries between the adjacent crystals. These multi-crystal whiskers were found to grow at all temperatures.

A variety of whisker growth bases were observed. Two of the more common are shown in Plate III, Figures 1 and 4. Plate III, Figures 2 and 3 shows a cobalt mass formation on the tip of whisker Co-62; they are the same whisker as Plate III, Figure 1. This overgrowth was observed on many whiskers.

The percentage of straight whiskers was found to be greater for growth just above transition than at higher temperatures. However, many of the assumed straight whiskers revealed slight kinking at high magnifications.

The surfaces of whisker crystal faces varied from smooth to quite irregular. A twisted appearance (Plate III, Figure 5) of several whiskers was observed but x-ray examination detected no lattice twist. Fewer surface irregularities were seen on single crystal than on multi-crystal whiskers. Surface structure on the larger (about 100 microns) multi-crystals was much more pronounced than on the smaller whiskers of this type. The ridge-like botryoidal formation (Plate III, Figure 6 and Plate VII, Figure 1) on the surface of Co-48 (130 microns) was also observed on other large multi-crystal whiskers. Smaller multi-crystals had no detectable ridge formation.

EXPLANATION OF PLATE III

Fig. 1. Photograph of the growth base of whisker Co-62.

Fig. 2. Photograph of the main body and tip of whisker Co-62.

Fig. 3. Photograph of the cone on the tip of whisker Co-62.

Fig. 4. Photograph of the growth base of whisker Co-66.

Fig. 5. Photograph of a cobalt whisker with twisted appearance.

Fig. 6. Photograph of the bumpy ridge formation on whisker Co-48.

PLATE III

Fig. 1

Fig. 2

Fig. 3

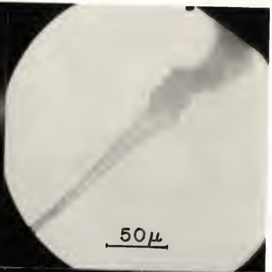


Fig. 4

Fig. 5

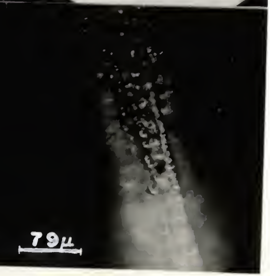


Fig. 6

EXPLANATION OF PLATE IV

Photographs of cobalt whiskers experimenting a change in growth direction, whisker to whisker attachment and helical growth.

Fig. 1. Photograph of the region of a cobalt whisker experiencing a change in growth direction.

Fig. 2. Photograph of whisker on whisker growth or whisker to whisker attachment as seen on a cobalt whisker.

Fig. 3. Photograph of a spiral of the helical whisker Co-74.

Fig. 4. Photograph of a helical whisker, Co-74.

Fig. 5. Photograph of a side of the helical whisker Co-74.

PLATE IV

Fig. 1

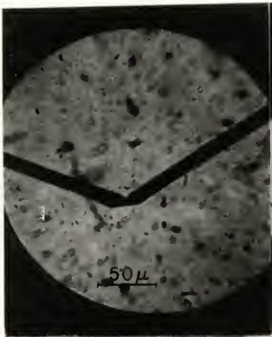


Fig. 2

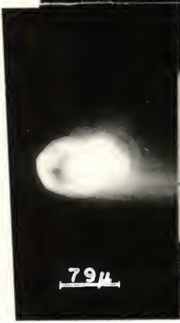


Fig. 3

Fig. 4

Fig. 5

Plate IV, Figure 1, reveals the region of a whisker where a change in the growth direction has occurred.

Whisker attached to whisker or whisker on whisker growth was also observed (Plate IV, Figure 2).

Ribbon whiskers were found but no detailed investigation was made of them.

Helical whiskers were found to grow between 600°C and 700°C. The whiskers grew straight for some distance before the helix was formed. It was often found that after a few spirals were complete the whisker again grew straight, in which case the radius of the helix was uniform. The straight whisker growth, after formation of the helix, was not observed in every instance. When such growth was absent, the radius of the spirals and the pitch of the helix decreased as the helix grew. (Plate IV, Figure 4).

Plate IV, Figures 3, 4, and 5 are of the same helical whisker. Figure 3, obtained by reflecting light from one of the spirals of the helix, demonstrates the hexagonal geometry of the spiral. It was observed (Figure 5) that the corresponding side of each hexagonal spiral lay in a plane. These two phenomena were found to occur for all cobalt helical whiskers examined.

X-ray Analysis

Table 1 lists the whiskers examined with x-rays. Twenty-four whiskers were found with the [10·0] growth axis, two with [00·1], two with [11·1], and two with $[\bar{1}1\cdot 1]$. Twenty-one single crystal and nine multi-crystal whiskers were observed. Every whisker examined was in a two-phase state containing the cph with a trace of the fcc.

Most of the whiskers produced uniform Laue spots, but broken and stair step-appearing diffraction images were also observed. Irregularities of this nature were found for the diffraction from both single crystals and multi-crystals of all whisker diameters.

Table 1. Cobalt whiskers analyzed by x-ray methods.

Whisker Number	Growth Temp °C	Diameter (microns)	Axis	Twist "α" (rad/mm)	Burgers Vector b(A°)	Crystal Type
Co-15		4.2	—	1.9×10^{-3}	0.26	S.C.
Co-20		2.3	[100]	—	—	—
Co-26		2.2	[100]	0	0	S.C.
Co-29		2.4	[100]	0	0	S.C.
Co-31		3.2	[100]	0	0	S.C.
Co-33		2.4	[100]	1×10^{-2}	0.45	S.C.
Co-34		2.2	—	5.9×10^{-3}	0.22	S.C.
Co-36		2.9	[100]	0	0	S.C.
Co-38		5.1	[100]	2.7×10^{-3}	0.56	S.C.
Co-39		34.0	[100]	0	0	M.C.
Co-42		—	[100]	0	0	S.C.
Co-46		7.5	[100]	0	0	S.C.
Co-47		5.3	[100]	0	0	S.C.
Co-48		130.0	[100]	0	0	M.C.
Co-55		3.5	[100]	1.9×10^{-3}	0.75	S.C.
Co-56		5.7	[100]	0	0	S.C.
Co-58		10-14	$[\bar{1}11]$	0	0	S.C.
Co-59		15.0	[100]	0	0	S.C.
Co-61		3.3	[001]	0	0	S.C.
Co-64		4.6	[111]	0	0	S.C.
Co-65	500 ± 20	11.5	[100]	1.3×10^{-1}	131	M.C.
Co-66	725 ± 20	20.6	[100]	0	0	S.C.
Co-67	480 ± 20	5.8	[100]	1.9×10^{-3}	0.50	S.C.

Table 1. (cont.)

Whisker Number	Growth Temp °C	Diameter (microns)	Axis	Twist "α" (rad/mm)	Burgers Vector b(A°)	Crystal Type
Co-68	430 ± 20	2.8	[100]	0	0	S.C.
Co-73	780 ± 20	10.7	[100]	—	—	M.C.
Co-75	785 ± 15	7.5	[100]	—	—	M.C.
Co-76	785 ± 15	2.6	[001]	—	—	—
Co-78	780 ± 20	5.1	[$\bar{1}$11]	0	0	M.C.
Co-79	785 ± 15	4.8	[100]	0	0	M.C.
Co-81	540 ± 20	8.6	[100]	—	—	M.C.
Co-84	540 ± 20	3.2	[111]	0	0	S.C.
Co-85	590 ± 15	7.4	[100]	0	0	M.C.

NOTE: S.C. - single crystal; M.C. - multi-crystal.

All whiskers with detectable lattice twists, except Co-65, were found to have Burgers vectors corresponding to a fraction of the minimum lattice translation vector (see Table 1). Co-65 had a Burgers vector of 131 A°.

The rotation pattern of Co-20 showed a light powder pattern imposed on a single crystal pattern. It appeared that the main body of this whisker was a single crystal and the outer portion contained small randomly distributed cobalt grains.

The larger, greater than 10 microns, diameter single crystal whiskers were found to be highly strained; however, there was no detectable strain for many of the smaller whiskers. It also appeared that the whiskers grown close to the transition temperature indicated less lattice strain.

All multi-crystal whiskers had the [10•0] growth axis except for Co-78 which was [$\bar{1}$1•1]. Co-81 was thought to consist of individual separate crystals, loosely connected with a common axis, because the Laue pattern was that expected for individual strain from each crystal. Co-78, [11•1], was the only multi-crystal found to contain no apparent strain. All other multi-crystal whiskers were highly strained as might be expected for crystals separated by small angle grain boundaries.

Broadening of the diffraction lines in the rotation photographs was observed for whiskers of all sizes and growth temperatures. Heat treatment of Co-48, a multi-crystal, was accompanied by further line broadening. The line broadening is indicative of an increase in the stacking fault population within the lattice.

One straight whisker, Co-65, is worthy of particular notice. The x-ray rotation pattern of the whisker showed it to be mostly hexagonal cobalt with a [10•0] axis. The equatorial layer line showed the presence of some cubic cobalt. The periodicity along the [110] direction in the cubic system is very nearly equal in length to the 'a' axial length in hexagonal cobalt. The Laue pattern showed seven equatorial spots with various segments of each spot displaying either an 80° tilt or no tilt with respect to a line perpendicular to the equatorial line. Some of the spots off the equator showed a small extension characteristic of a lattice strain. The Weissenberg pattern showed a typical pattern for a hexagonal crystal rotated about its 'a' axis except that each Weissenberg spot was made up of six small spots separated by approximately equal incremental distances of 0.36 mm on a 57.3 mm diameter camera film.

After heating this whisker for 96 hours at 400°C, the Laue pattern showed an increased streaking for the off equator spots and a mixture of straight and tilted spots on the equator. The Weissenberg pattern of this heated whisker showed the same Okl spots as for the as grown whisker except that each reflection was made up of four instead of six smaller spots. Again the separation of the spots was approximately 0.36 mm.

Heating the whisker again at 550°C for one hour had a marked change on the Laue pattern. The extended spots had disappeared showing little lattice strain. The equatorial spots consisted of two distinct sets of reflections, one set with zero tilt and the other set tilted at 35°. The Weissenberg pattern of this crystal after the additional heating now showed as before the reciprocal lattice points except that each reflection consisted of only two spots separated by 1.08 mm. The rotation pattern of the whisker after this heating showed as in the case of the as grown whisker, a [10·0] hexagonal whisker axis with a small trace of cubic cobalt.

Heat Treatment

Non-deformed cobalt whiskers with bends and kinks, which were due either to growth irregularities or effects of the transition, were heat treated for five hours at 1000°C. The bends and kinks were found to remain in the whiskers. Deformed whiskers were heated to 400°C for 48 hours and 450°C for one hour with no recovery. Recovery was realized in this one instance for a heat treatment for one hour at 850°C. After annealing at high temperatures (about 800°C) the whiskers were found upon cooling to be polycrystalline.

After heat treatment all whiskers retained both the cph and fcc phases in approximately the same amounts as before heat treatment.

High Resolution X-ray Diffraction Microscopy Results

Berg-Barrett (B-B) photographs were taken of single crystals (Co-55 and Co-56) and multi-crystals (Co-39, Co-48 and Co-65).

In some instances, because of the manner in which the whisker was mounted, it was impossible to place the NTB plate closer than 5 mm to the whisker. Thus, the $K\alpha_1$ and $K\alpha_2$ lines are resolved in such photographs. The Berg-Barrett photograph (BB-30) of the (00•2) plane of Co-56 is shown in Plate V, Figure 1. Plate V, Figure 2 is a picture of a portion of whisker Co-55. The corresponding B-B diffraction imate (BB-35) of the $(01\cdot\bar{1})$ plane of this whisker is shown in Plate V, Figure 3.

The Co-65 diffraction image is revealed in Plate V, Figure 4 (BB-37a). The corresponding B-B photograph after heat treatments of 96 hours at 400°C and one hour at 550°C is shown in Plate V, Figure 5 (BB-39). It is not known whether BB-37a and BB-39 correspond to the same plane. The $K\alpha_1$ and $K\alpha_2$ resolution is clearly seen in BB-37a but is not as pronounced in BB-39.

Because of the large cross-sections of some multi-crystal whiskers, the reduced intensity due to sample absorption was calculated. The thickness of cobalt necessary to reduce the intensity by a factor of $\frac{1}{2}$ is about 12 microns (using 65.9 cm^2/gm as the mass absorption coefficient and 8.9 gm/cm^3 as the density of cobalt). In particular the intensity is reduced by a factor of about 1/7 for Co-39 (34 micron diameter) and 1/2000 for Co-48 (130 microns).

EXPLANATION OF PLATE V

Berg-Barrett photographs of whiskers Co-55, Co-56 and Co-65 and a drawing of whisker Co-39.

Fig. 1. B-B photograph, BB-30, of the (00·2) plane of whisker Co-56.

Fig. 2. Photograph of whisker Co-55.

Fig. 3. B-B photograph, BB-35, of the $(01\cdot\bar{1})$ plane of whisker Co-55.

Fig. 4. B-B photograph, BB-37a, of whisker Co-65 before heat treatment.

Fig. 5. B-B photograph, BB-39, of whisker Co-65 after heat treatment of 96 hours at 400°C and one hour at 550°C.

Fig. 6. Drawing of whisker Co-39.

PLATE V

Fig. 1 Fig. 2 Fig. 3

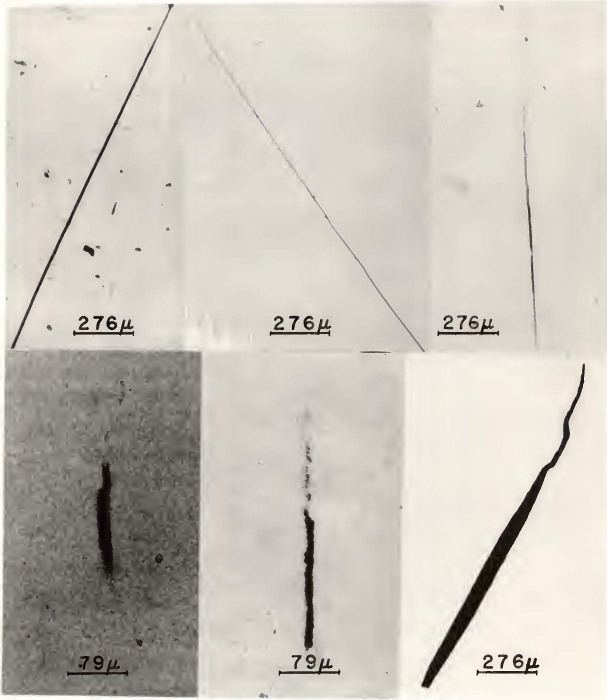


Fig. 4 Fig. 5 Fig. 6

The first multi-crystal examined with the B-B technique (Co-39) was lost before the study was completed. Plate V, Figure 6 is a drawing of the whisker. Plate VI, Figures 1 (BB-19), 2 (BB-21), and 3 (BB-22), are the (00•2), (01•1), and $(01\cdot\bar{1})$ planes, respectively. The whisker was heat treated for 24 hours at 380°C and 24 hours at 530°C. The (00•2), (01•1), and $(01\cdot\bar{1})$ planes, after heat treatment, are revealed in Plate VI, Figures 4 (BB-27), 5 (BB-29), and 6 (BB-28), respectively. $K\alpha_1$ and $K\alpha_2$ resolution is apparent in all diffraction images except for BB-28 and BB-29. The separation between the periodic spots along the whisker axis (especially apparent in BB-19 and BB-27) is about 10 microns.

Optical microscope studies of Co-48, a multi-crystal, show a series of ridge-like botryoidal formations running along the axis of the whisker (Plate III, Figure 6 and Plate VII, Figure 1). The bumps forming the ridges are separated by about 15 microns. The tip of the whisker (Plate VII, Figure 2) reveals that there are eight ridges.

A radiograph (Plate VII, Figure 3) demonstrates that an irregular internal structure is present. B-B photographs were taken before and after the whisker was heat treated even though the results were thought to be questionable because of the large cross-section of the whisker. The first heat treatment was 55 hours at 410°C, the second for 1½ hours at 550°C. The (00•2) plane is revealed in Plate VIII, Figures 1 (BB-34) (before heat treatment), 2 (BB-41) (after first heat treatment), and 3 (BB-43) (after the second heat treatment). The (01•0) plane is similarly compared in Plate VIII, Figures 4 (BB-38a), 5 (BB-40), and 6 (BB-42).

The external appearance of the whisker remained the same after both heat treatments. The $K\alpha_1$ and $K\alpha_2$ were resolved in BB-34, BB-38a, and BB-40. The periodic spots along the diffraction line are spaced about 35 microns. Separation between the two diffraction lines running the length of the whisker on BB-40, BB-41, and BB-42 were found to correspond to the diameter of the whisker.

EXPLANATION OF PLATE VI

Berg-Barrett photographs of whisker Co-39 before and after heat treatment.

Fig. 1. B-B photograph, BB-19, of the (00·2) plane of whisker Co-39 before heat treatment.

Fig. 2. B-B photograph, BB-21, of the (01·1) plane of whisker Co-39 before heat treatment.

Fig. 3. B-B photograph, BB-22, of the $(01\cdot\bar{1})$ plane of whisker Co-39 before heat treatment.

Fig. 4. B-B photograph, BB-27, of the (00·2) plane of whisker Co-39 after heat treatment of 24 hours at 380°C and 24 hours at 530°C.

Fig. 5. B-B photograph, BB-29, of the (01·1) plane of whisker Co-39 after heat treatment of 24 hours at 380°C and 24 hours at 530°C.

Fig. 6. B-B photograph, BB-28, of the $(01\cdot\bar{1})$ plane of whisker Co-30 after heat treatment of 24 hours at 380°C and 24 hours at 530°C.

PLATE VI

Fig. 1 Fig. 2 Fig. 3

Fig. 4 Fig.5 Fig. 6

276μ

EXPLANATION OF PLATE VII

Photographs and a radiograph of whisker Co-48.

Fig. 1. Photograph of whisker Co-48.

Fig. 2. Photograph of the tip of whisker Co-48.

Fig. 3. Radiograph of whisker Co-48.

PLATE VII

Fig. 1

Fig. 2

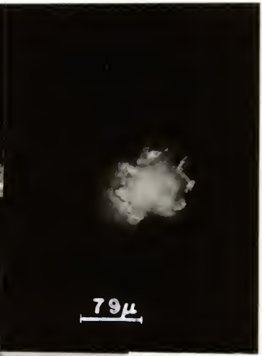


Fig. 3

EXPLANATION OF PLATE VIII

Berg-Barrett photographs of whisker Co-48 before and after heat treatments.

Fig. 1. B-B photograph, BB-34, of the (00·2) plane of Co-48 before heat treatment.

Fig. 2. B-B photograph, BB-41, of the (00·2) plane of Co-48 after heat treatment of 55 hours at 410°C.

Fig. 3. B-B photograph, BB-43, of the (00·2) plane of Co-48 after heat treatments of 55 hours at 410°C and $1\frac{1}{2}$ hours at 550°C.

Fig. 4. B-B photograph, BB-38a, of the (01·0) plane of whisker Co-48 before heat treatment.

Fig. 5. B-B photograph, BB-40, of the (01·0) plane of whisker Co-48 after heat treatment of 55 hours at 410°C.

Fig. 6. B-B photograph, BB-42, of the (01·0) plane of whisker Co-48 after heat treatments of 55 hours at 410°C and $1\frac{1}{2}$ hours at 550°C.

PLATE VIII

Fig. 1 Fig. 2 Fig. 3

Fig. 4 Fig. 5 Fig. 6

358μ

DISCUSSION

Whisker Analysis

All cobalt whiskers were found to grow on previously deposited cobalt. A temperature of about 600°C produced the best whisker growth. These and other observations are in agreement with the original work done by Brenner (6) on the growth of metal whiskers by reduction of the halide.

Brenner and Sears proposed a method for the growth of whiskers by vapor deposition (10). Their method indicates that the formation of an individual whisker growth cone results only when a screw dislocation is by itself or not related to other dislocations. The growth cone would not form for individual screw dislocations spaced too closely or exposed to an excess supersaturation. Plate III, Figures 1 and 4 shows the growth bases of cobalt whiskers that seem to support the mechanism of Brenner and Sears.

It is interesting to note that the whisker containing the unusually large growth cone (Plate III, Figure 1) also has a cone at its tip (Plate III, Figures 2 and 3). The overgrowth on the whisker tip can be attributed to a high supersaturation of cobalt vapor. Such termination is necessary to relieve the strain energy of the lattice associated with the screw dislocations.

The change in growth direction, twisted appearance, and whisker to whisker attachment are just several of the features of whiskers that are also observed for cobalt.

This author knows of no work done with multi-crystal whiskers. About one-third of the whiskers examined were found to be multi-crystalline. The multi-crystals grew at all temperatures and were generally highly strained.

The ridge-like botryoidal formation on the surfaces of the larger multi-crystal whiskers is indeed very interesting. The ridges are probably located at small angle grain boundaries separating the crystals. It is believed that the bumps forming the ridges are results of the shear that occurred during the cobalt transition. Only in one case was it thought that the multi-crystal may not be composed of multiple crystals.

Indications are that the main body of Co-20 was a single crystal but the outer portion contained small randomly distributed cobalt grains. This overgrowth may be realized for high supersaturations of cobalt vapor depositing at random on the crystal surfaces.

The helix developing on a perfectly straight whisker is observed in some cases to terminate the growth of the whisker (Plate IV, Figure 4), and in other cases to be intermediate between two straight whisker sections. The partially straight and partially coiled features of cobalt helical whiskers are in agreement with the growth mechanism for helical whiskers proposed by Amelinckx (1). He discussed a method by which the screw dislocation mechanism could shorten to partially relieve the line tension of the dislocation and thus produce helical growth. This method, however, does not seem to explain completely some of the effects observed for cobalt helical whiskers. The spiral of cobalt helical whiskers was found to have hexagonal symmetry in all cases. For the cases where the helix terminated the growth the radius of the spiral and the pitch of the helix decreased as the helix grew. Other helical whiskers had approximately a constant radius and constant pitch. It is suggested that a mixed dislocation where the dislocation intercepts the edge of the growth surface with a new growth direction resulting seems to be a more feasible explanation for the growth of cobalt helical whiskers. If the cross-sectional area of the whisker remained constant during growth the resulting spirals would be expected to be of constant size. This effect is observed for the spirals intermediate between two straight sections of the whisker. The case of the helix terminating the growth of the whisker with an accompanying decrease of the radius of the spirals and the pitch of the helix is easily explained by noting in this case that the cross-sectional area of the whisker decreases as the helix is growing; therefore, the mixed dislocation line is shorter after each intersection of the surface. The accompanying change in growth direction occurs more frequently as the helix grows, thus resulting in a decreased radius for each succeeding spiral.

For cobalt whiskers, the [10•0] growth axis is by far the most common although the [00•1], [11•1], and [$\bar{1}$1•1] were also observed. The temperature, above transition, at which the whiskers grew did not appear to affect their properties at room temperature.

Webb (private communication) has grown cobalt whiskers below the transition temperature and detected the lattice twists corresponding to an axial screw dislocation. It is, therefore, justifiable to assume that whiskers grown above the transition temperature initially contain an axial screw dislocation. However, at room temperature most of these whiskers are found to contain no detectable lattice twist. For whiskers with some twist the corresponding Burgers vector is a fraction of the minimum lattice translation vector expected for a single axial screw. This particular phenomenon has been observed on many metal whiskers. It may be due to the whisker growing with pairs of screw dislocations and eventually these dislocations glide together during the phase transition and annihilate each other. Another possibility is that a dislocation absorbs trapped vacancies which permit it to climb out of the whisker during or after the growth process. In the case of cobalt it is most probable that the transformation from the fcc to the cph relieves the whisker of its initial screw dislocation or dislocations by the accompanying slip of every second (00•1) plane.

Co-65 appears to be a multi-crystal whisker. The as grown whisker is made up of six crystals with low angle grain boundaries separating each unit. The grain boundary angle between adjacent grains is in each case approximately 0.7°. Otherwise the six individual crystals appear as one with their axis common in the [10.0] direction. There is a certain amount of lattice strain resulting undoubtedly from the vacancies which nucleated the small angle grain boundaries. If the Burgers vector for this crystal is 131 Angstroms, then the distance between dislocation centers along the small angle grain boundary is about one micron (see Plate IX). The line of the dislocations would be parallel to the whisker axis and could be due to a number of edge type dislocations or pairs of screw dislocations of opposite sign.

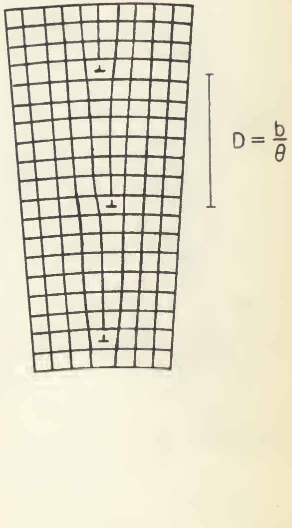
The thermal stress from heating of the whisker below the phase transition temperature, that is 96 hours at 400°C, could cause either the edge dislocations to move and possibly intersect the crystal surface and disappear or the screw dislocations to glide together and annihilate one another. A large amount of lattice strain would most likely result on cooling the crystal. This heating caused two of the small angle grain boundaries to disappear and did introduce more lattice strain.

Heating the same crystal again to 550°C for one hour caused two more grain boundaries to disappear leaving only two small angle grain boundaries or in this case what amounts to two crystals at approximately 2° tilt angle to each other. The Laue pattern indicates that one of the two crystals is no longer twisted while the other is twisted by a screw dislocation with a 16 Angstrom Burgers vector. The lattice strain is very nearly gone for the remaining pair of crystals which make up the whisker. The phase transformation that takes place in heating the whisker to 550°C and then again occurs upon cooling undoubtedly eliminates a number of the dislocations. The few remaining dislocations that make up the tilt boundary of the resultant bi-crystal are probably the reason that the whisker does not become a fine grained randomly oriented filament.

EXPLANATION OF PLATE IX

Diagram of a simple small angle grain boundary separating two crystals with a cubic 100 common axis. The line imperfections or dislocations are denoted by the symbol $\perp$. D, b, and θ represent the distance between dislocations, Burgers vector, and tilt angle, respectively.

PLATE IX



Heat Treatment

An extensive study of the recovery of deformed cobalt whiskers was not attempted although it was noted that no recovery was found for heat treatments to 400-450°C while at 800-850°C recovery was realized. This was patterned after work done by Brenner and Morelock (9) on the high temperature recovery of copper whiskers.

After heating through the transition to 550°C cobalt whiskers did not recrystallize into smaller grains. This is in agreement with observations made by Kehrer and Leidheiser (18) who worked with single crystals of cobalt. It was found for whiskers heated to about 800°C that a polycrystalline specimen resulted.

High Resolution X-ray Diffraction Microscopy Studies

The Berg-Barrett photograph, BB-30 (Plate V, Figure 1), of the (00·2) plane of a nearly perfect single crystal whisker, Co-56, gives a straight uniform diffraction image indicating an absence of internal structure. This whisker had no detectable lattice twist and therefore the diffraction image is a uniform darkened replica of the (00·2) plane. It appears that this whisker does not contain pairs of axial screw dislocations; however the method used may be incapable of detecting closely spaced dislocations.

Co-55, also a single crystal, contains several minor kinks (Plate V, Figure 2). The kinks may correspond to localized transformations as observed by Sears and Brenner (25) on iron whiskers. The B-B photograph, BB-35, of the $(01\cdot\bar{1})$ plane of this whisker (Plate V, Figure 3) reveals an irregular structure. Close examination shows that the kinks are also seen in the diffraction image. The Laue pattern of this whisker indicated a lattice twist and a corresponding Burgers vector of 0.75A°, which is a fraction of the expected minimum lattice translation vector. The B-B photograph demonstrates that a shift in the lattice may have occurred at one of the kinks because of the displacement of the highly diffracting region. The breaks in the diffraction line and also the kinks could have been caused by a shear type transition. The calculated Burgers vector therefore, does not result from a pure lattice twist for the whole whisker but rather from a possible periodic shift of the lattice in some incremental block-like manner.

The multi-crystal Co-65 shows an increase in the length of the B-B diffraction image by a factor of about two after total heat treatment. (Plate V, Figures 4 and 5). This is possibly the result of the disappearance of four of the small angle grain boundaries on heat treatment. The diffraction images seen here are probably equivalent to the segment of the equatorial spots on the Laue patterns showing no tilt. There seems to be no structure within the spots but the displaced extension of the diffraction spot suggests a block like displacement within the whiskers.

The diffraction from the larger multi-crystals presents a very interesting but highly complicated structure. A study of the (00•2) (Plate VI, Figures 1 and 4), (01•1) (Plate VI, Figures 2 and 5) and $(01\cdot\bar{1})$ (Plate VI, Figures 3 and 6) planes of Co-39 indicates a definite change in the internal structure after heat treatment. The periodic structure along part of the axis of the whisker is apparent in all of the photographs. It is especially pronounced in the main body of the whisker (Plate VI, Figure 1). The periodic spots are, on the average, separated by about 10 microns. This corresponds to the spacing (measured from a photomicrograph by Anantharaman and Christian (2)) between thin martensitic plates (caused by shear) observed on a polycrystalline specimen of pure cobalt that was passed through the transition temperature. The distinct lines observed on BB-19 and BB-27 along the whisker axis may be partially due to $K\alpha_1$ and $K\alpha_2$ resolution of the diffraction from the surface, whereas those on BB-28 and BB-29 are definitely not due to this effect because of the very definite cross-over.

The entire structure seen in the B-B photographs cannot be due to surface effects; (intensity is reduced by only a factor of 1/7 in penetrating the main body of the whisker) most of the diffraction must be from internal regions of the whisker. From studying the diffraction patterns it appears that there are two line imperfections (dislocations) running the length of the whisker. The fact that they produce periodic spots along the axis indicates a structure resembling blocks which are slightly oriented with respect to one another. The large kink in the whisker (see Plate V, Figure 6) is easily related to the high resolution diffraction spots before heat threament. It is surprising to note that after heat treatment the kink was eliminated in the B-B photographs but was found to remain in the whisker. The cross-over, revealed after heat treatment, is observed in the region of the kink. It appears that in this region both dislocations lie in the (00•2) plane and they seem to be spaced by about 10 microns. The cross-over seen on (01•1) and $(01\cdot\bar{1})$ is not an actual intersection of these dislocations but only appears to be so because the dislocations are displaced relative to each other and are being sighted along the plane in which they nearly lie. The angle between the (00•2) plane and the (01•1) and $(01\cdot\bar{1})$ planes are 62° and -62°, respectively.

The eight-fold symmetry of the ridges on Co-48 (see Plate VII, Figure 2) suggests that there are eight crystals present. For this whisker, 130 microns in a cross-sectional dimension, the intensity is reduced by a factor of 1/2000 because of sample absorption. A radiograph of the whisker indicates a definite irregular internal structure. The B-B photographs show diffraction from the whisker surface. A periodic arrangement of diffraction spots is again observed along the axis of this whisker. The bumps forming the ridges of the whisker are about 15 microns apart, whereas the distance between the periodic diffraction spots in the B-B photograph is about 35 microns. It appears as though a group of several bumps may account for one of the small diffraction spots. Diffraction from the (00•2) plane reveals a displacement at the tip of the whisker. A displacement along one of the ridges of the whisker was found before and after heat treatments (see Plate VI, Figure 1) and is believed to correspond to the displacement in the B-B photograph. The displacement in the B-B photograph is still seen after the first heat treatment but upon re-heating it is found to be less pronounced. A study of the apparent grouping of the periodic diffraction spots for the (00•2) and the (01•0) planes shows that the same groups of spots remain after heat treatment. There is however, a tendency for the spots to be less pronounced since they appear to diffuse together.

The periodicity in the diffracted intensity along the whisker axis is demonstrated by Co-39 and Co-48. This phenomena is definitely due to surface effects for Co-48 but is found to be at least partially due to the internal structure for Co-39. An attempt at explaining this phenomenon will be made in the discussion of the cobalt transition.

Cobalt Transition

All cobalt whiskers were found to exist in a two-phase state at room temperature, i.e., the transition to the hexagonal structure was incomplete leaving from 10 to 20 percent of the cubic phase. The same structures prevailed for whiskers that were heat treated to 550°C and again examined at room temperature.

Broadening of the diffraction lines for single crystals of cobalt was reported by Edwards and Lipson (13) and was also found for all the cobalt whiskers observed in this work. Larger whiskers demonstrated more broadening and therefore more faults. The amount of faulting did not appear to be affected by the growth temperature of the whiskers. Line breadth measurements will be made, but not included in this work to actually determine the stacking fault probability for at least one whisker.

Considerable lattice strain was observed for the larger single crystal and for all multi-crystal whiskers. Many of the smaller whiskers demonstrated no detectable strain. This is probably due to the ease of transition for a smaller cobalt whisker than for a larger one. A dislocation introducing a shear type transition would probably find difficulty in crossing a small angle grain boundary in a multi-crystal whisker and could introduce considerable strain in the lattice. Whiskers grown close to the transition temperature were found to have less strain than those grown at higher temperatures. This, of course, would be expected for whiskers growing in a two-phase state close to transition as compared to those containing a single phase at higher growth temperatures.

A multiplicity in some of the Laue spots for cobalt single crystals has also been observed by Kehrer and Leidheiser (18). They attributed this to a mosaic structure with each of the blocks only slightly oriented with respect to one another. It is believed by this author that this block structure is also present in some cobalt whiskers and was created by the relaxation of the twisted lattice (caused by an axial screw dislocation or dislocations) as the whisker transformed from the fcc to cph.

This block structure would explain why the whisker becomes polycrystalline when heated to a high temperature where complete transition to fcc is realized. The blocks would not assume their original non-oriented position but would tend to become more randomly oriented. Cobalt whiskers were found to retain their single crystal or multi-crystal structures after heating to 550°C. This implies that the transformation to fcc is not complete at this temperature.

The ridge-like botryoidal formations on the larger multi-crystals can possibly be explained as due to a displacement of cobalt in the small angle grain boundary region between crystals during the phase transition. X-ray examination of this surface phenomenon and also the internal structure of the larger multi-crystals indicates a very sharp plate-like formation as might be expected for a shear type transition mechanism. After heat treatment the B-B photographs revealed a broadening of the sharp periodic diffraction areas indicating a possible relaxation of the strained region surrounding the plates.

All avenues of the investigation of the cobalt transition by its effects on cobalt whiskers indicated a shear mechanism. This has previously been concluded by other workers (2) (5) (13).

CONCLUSION

The striking difference between cobalt whiskers grown above the transition region and other whiskers has been the frequent multi-crystal form in which they are found and their infrequent but definite $[00 \cdot 1]$, $[11 \cdot 1]$, and $[\bar{1}1 \cdot 1]$ growth axes. The most commonly observed growth direction is the $[10 \cdot 0]$.

Since the one-dimensional growth of cobalt whiskers below the transition temperature has been shown to be the result of an axial screw dislocation or dislocations it is assumed that growth above the transition is a result of the same mechanism. Single crystal whisker growth is probably due to a single dislocation or a pair of dislocations, whereas the multi-crystal growth is most likely the result of many axial screw dislocations.

The transition of cobalt whiskers from the fcc to the cph phase is believed to be accompanied by a shear mechanism. The stress created for small whiskers (2 microns) when transforming would probably be sufficient to relieve the whisker of its initial axial screw dislocation or dislocations leaving a relatively strain-free whisker. For the larger single crystal whiskers (10 microns) it seems reasonable that the lattice could be left highly strained even if the screw were lost. After crossing a grain boundary in a multi-crystal, the dislocation producing the shear would be expected to leave a strained region and thus a highly strained whisker.

Indications are that the transition creates either a plate-like or a block-type displacement or both, with a separation of from 10 to 40 microns between the displaced regions. The larger multi-crystals seem to indicate the plate-type displacement, whereas many of the single crystals point to the block structure with the blocks slightly oriented with respect to one another. The latter case explains why many whiskers have a Burgers vector corresponding to a fraction of the minimum lattice translation vector. It is reasonable to assume that there is no actual lattice twist but a periodic lattice displacement approximating a twist.

The study of cobalt whiskers has revealed a multi-crystal growth, varied dislocation arrays, and presents a new tool for studying the phase transformation in cobalt.

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COBALT WHISKERS: THEIR GROWTH, DISLOCATIONS AND PHASE CHANGE

by

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The work reported here is a study of cobalt whiskers grown above the transition temperature. These whiskers were studied since the dislocation structure of many metal whiskers is still in doubt and a complete solution to the cobalt transition problem has not been realized. Work with cobalt whiskers by several investigators indicates that they grow by the vapor deposition mechanism and result from an axial screw dislocation or dislocations. The whiskers after growth should thus contain a lattice twist about their axes. This lattice twist allows the lateral surfaces of the whisker to be strain free, while around the immediate area of the screw dislocation there is a highly strained region. The highly strained region and thus the lattice twist may be relieved by torsion, heating, or possible shear resulting from the transformation from the face-centered cubic phase to the close-packed hexagonal phase.

Cobalt whiskers were grown by hydrogen reduction of $CoCl_2$. Optical microscope studies were performed using either transmitted or reflected light with magnifications up to 800. Laue, rotation, and Weissenberg photographs were taken. A high resolution x-ray diffraction technique was used to detect lattice irregularities.

All cobalt whiskers were found to grow on previously deposited cobalt. A temperature of about 600°C produced the best whisker growth. A variety of growth bases were observed; the most interesting was in the form of a cone. Many of the whiskers had a cobalt mass deposited on their tips. The majority of the whiskers were straight but whisker to whisker attachment, ribbon-like and helical whiskers were also found. Some of the whiskers experienced a change in growth direction, kinking, and other abnormalities. The [10·0] growth axis is by far the most common although the [00·1], [11·1] and $[\bar{1}1\cdot1]$ growth axes were also observed. About one-third of the whiskers examined were multi-crystalline; the rest were single crystals. A multi-crystal whisker is here defined as a whisker containing from two to ten crystals all with a common growth axis. These multi-crystal whiskers were found to grow at all temperatures. Several of the larger multi-crystal whiskers had a ridge-like botryoidal formation on their surfaces. The ridges were probably located at small angle grain boundaries separating the individual crystals. Indications are that single crystal whisker growth is probably due to a single axial screw dislocation or a pair of screw dislocations, whereas the multi-crystal growth is the result of many axial screw dislocations. Many of the whiskers had no apparent lattice twist. The whiskers with detectable lattice twists were found to have Burgers vectors corresponding to a fraction of a minimum lattice translation vector.

All cobalt whiskers examined were found to exist in a two phase state at room temperature. The transition from the cubic to the hexagonal phase is thought to be accomplished by a shear mechanism creating either a plate-like or block-like displacement of atoms on every second close-packed layer or both. The larger multi-crystal whiskers seem to indicate the plate type displacement, whereas many of the single crystal whiskers point to the block-like structure with the blocks slightly oriented with respect to one another. The latter block-like case might explain why many whiskers have a Burgers vector corresponding to a fraction of the minimum lattice translation vector. It seems reasonable to assume that there is no actual lattice twist but a periodic lattice displacement approximating a twist.

The study of cobalt whiskers has revealed a multi-crystal growth, varied dislocation arrays, and presents a new tool for studying the phase transformation in cobalt.