Filamentary Growths On Metal Surfaces—"Whiskers"*

By K. G. COMPTON*, A. MENDIZZA*, and S. M. ARNOLD*

Abstract

Filamentary growths have been found on metal surfaces of some of the parts used in telephone communications equipment, particularly on parts shielded from free circulation of air. The growths are of the same character as those known as "whiskers" and which developed between the leaves of cadmium plated variable air condensers, causing considerable trouble in military equipment during the early part of World War II.

An investigation has been under way in an attempt to determine the mechanism of growth of the whiskers, found not only on cadmium plated parts but also on other metals. This paper summarizes the findings to date as revealed by the study of approximately one thousand test specimens of different metals, solid and plated, exposed under various environmental conditions. The study is being extended in the light of the findings which have developed during the course of the work.

While the whiskers normally are not found on parts such as condenser leaves for some time, often months, after being electroplated, it is possible, by increasing the ambient temperature, to so accelerate their growth that they will develop in a matter of weeks. So as to avoid the introduction of additional variables which so often attend accelerated methods of test, the greater part of the present work is being carried out at temperatures attained by equipment operating under normal exposure conditions.

The effects of humidity and the presence of various organic materials, different film thicknesses and methods of application, surface preparation and supplementary treatments, chemical and physical properties and X-ray studies of structure are discussed in the paper.

Introduction

DURING THE early part of 1948 information was received at the Bell Telephone Laboratories that trouble of an unusual nature had developed in some of the channel filters used in the carrier telephone systems. These filters are essentially networks designed to maintain the frequency bands assigned to the various channels in a multi-channel transmission line. As this maintenance of band is critical, considerable pains are taken in the fabrication of the filters. The quartz crystals are sealed within glass envelopes, the associated capacitors and inductances carefully shielded and the components assembled and wired in a room maintained at a relative humidity of 40 per cent or less. After wiring, the assembly is placed in a steel can and the whole hermetically sealed with solder.

Electrical failure of a filter directly affects the operation of a transmission channel and this type of trouble cannot be tolerated. Emergency removal of the filter and replacement with another unit is required in order to maintain service.

Following removal, the filter is sent to the Quality Assurance group of the Laboratories for examination as to the cause of failure. Frequently when such filters were tested subsequent to shipment to the laboratories, no source of trouble could be located and the units were found to operate satisfactorily. After this had occurred several times, the trouble was finally traced to the presence of microscopic, discreet filaments generally perpendicular to the surface of the metal, which had bridged across critical spacings between components of the filter. These filaments were sufficiently conductive, of the order of 400 ohms, that low impedance paths had been established, effectually short-circuiting elements of the network. Extremely small and fragile, the filaments were easily broken or dislodged, thus explaining why filters removed from service frequently were found to be trouble free when put under test after shipment. In Figure 1 is shown one of the filters while Figure 2 shows more clearly one of the critical spacings across which filaments actually bridged. Figure 3 shows a somewhat enlarged view of the bridging filaments in a filter removed from service.

After the trouble had been traced to the presence of these filaments, it was recalled that suppliers occasionally had had trouble with the development of filamentary growths known as "whiskers" between the cadmium plated leaves of variable air condensers. While this phenomenon was known to exist in the case of cadmium plated metal, the filter parts referred to had been zinc plated. Nevertheless, the growths appeared to be of the same type and it was decided to study their mode of formation and extend the study to determine whether similar growths might develop on metal surfaces other than cadmium or zinc.

While the work has not been completed it was decided to publish the preliminary results of the study in the light of the present world situation. These "whisker" growths on cadmium plated condensers caused trouble in military equipment during the early part of World War II. A considerable volume of material for the Armed Forces, susceptible to this type of growth, is now being maintained in standby condition and must function without question or delay should a crisis develop. Furthermore, as a large quantity of military material is presently

* A paper presented at the Seventh Annual Conference and Exhibition, National Association of Corrosion Engineers, New York, N. Y., March 13-16, 1951.

* Members of technical staff, The Bell Telephone Laboratories, Murray Hill, New Jersey.

short that they were hardly distinguishable from irregularities in the metal surface, while others had developed to a length of over ⅜ of an inch. The diameter appeared to be nearly uniform from base to tip, later found to be approximately 2 microns (80 x 10^{-6} inches). That they were not cylinders was shown when an individual fiber was rotated on its longitudinal axis and illuminated by a narrow light beam. Distinct facets revealed themselves with alternate reflections and extinctions.

Some of the fibers were removed and tested for solubility. Water and various types of organic solvents were found to have no effect. The whiskers did dissolve in an aqueous 5 per cent hydrochloric acid solution and completely disintegrated when exposed in situ to vapors of hydrochloric acid.

Examined under ultra violet light there was no indication of fluorescence, nor was there any evidence of charring when an incandescent platinum wire was brought close to an individual fiber. Instead, the heat converted the latter to a white ash. A probe brought near a fiber caused deflection over a considerable arc. Application of a potential of approximately 10 volts or more caused the individual fiber to disappear.

The first filter units examined and found to have whiskers present had been in service approximately 1 year. In these the steel details had been zinc plated to a thickness of 0.0002 inches. Shortly after, it was found that whiskers had developed in units which had not been in service, but had been stored for a period of 8-9 months. Evidently an electrical potential difference was not necessary for whisker growth as had been suggested when trouble first developed.

As prior to this time whisker growths had been associated chiefly with cadmium plated parts, the question arose as to whether the zinc coating contained cadmium. Also it was known that mercury was sometimes used as an addition agent in zinc plating baths to improve the appearance of the deposit. Analyses indicated that there was no mercury and less than 0.03 per cent cadmium present in the coating.

Preliminary Work

At this time an attempt was made to develop whisker growths in the laboratory. As it was now evident that these growths could develop on both cadmium and zinc plated parts, it was decided to include in the study a few other commonly used metals. Metal specimens were plated and fastened to pieces of phenol fiber. This last was used because organic material in the form of insulation and mounting strips had been present in the affected filters. The assemblies, in glass containers, were maintained at 95 degrees Fahrenheit at a relative humidity of 90 per cent. When examined after three months it was found that numerous whiskers had developed not only on cadmium and zinc plated specimens but also on parts plated with tin.

It was then decided to expand the program still further to include both solid metals and electroplated coatings in a range of thicknesses, with various organic materials as contaminants. Exposures were made under several humidity-temperature combinations, although, to avoid the introduction of additional variables so often associated with accelerated testing, the greater part of the study has been carried out at temperatures maintained by equipment operating under normal conditions. Up to the present time approximately one thousand specimens have been included in this study.

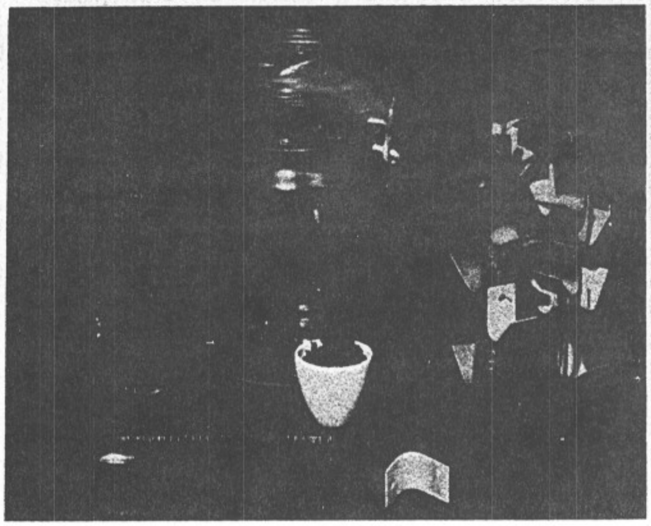

Figure 4—Container and specimens as used in study

Experimental

The type of specimen found most convenient was cut from 1/32 inch metal stock, ½ by 1½ inches on a side, and bent at the center to form a right angle. Following the desired preparation, the angles were supported on glass stands and these in turn placed in 1 pint glass topped preserve jars. The organic contaminant in finely divided form was contained in a

TABLE I
Metallic Materials Studied

Electroplated Metals*	Solid Metals
Nickel	Nickel
Copper	Copper
Silver	Silver
Zinc	Zinc
Cadmium	Cadmium
Tin	Tin
	Solder[1]
	Aluminum Alcoa 3S alloy[2]
	Aluminum Alcoa 24S alloy[3]

* Each plated in 3 thicknesses—.00005 inch.
.0002 inch.
.0005 inch.

[1] 45% Sn—55% Pb Solder.
[2] 1.2% Mn, Remainder Al.
[3] 4.5% Cu, .6% Mn, 1.5% Mg, Remainder Al.

TABLE II
Organic Materials Used as Contaminants

Phenol Fiber	A phenol formaldehyde product widely used for terminal strips and other electrical equipment.
Tenite	An aceto-butyrate materials used for telephone handsets.
Plexiglas	A methyl methacrylate product used for panels and supports for electrical parts.
Varnished Cambric Sleeving	A cotton fabric impregnated with an oleoresinous varnish and baked. Used as an insulating covering for wire.
Hard Rubber	A high sulfur vulcanized rubber.
Lacquer	A resin-modified cellulose nitrate lacquer used as a finish on various types of communications equipment.

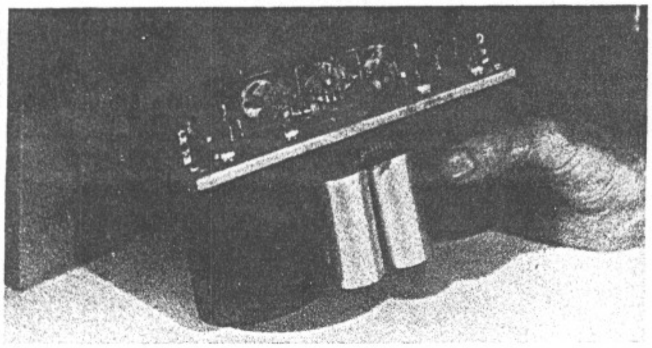

Figure 1—Quartz crystal filter. Steel can in background.

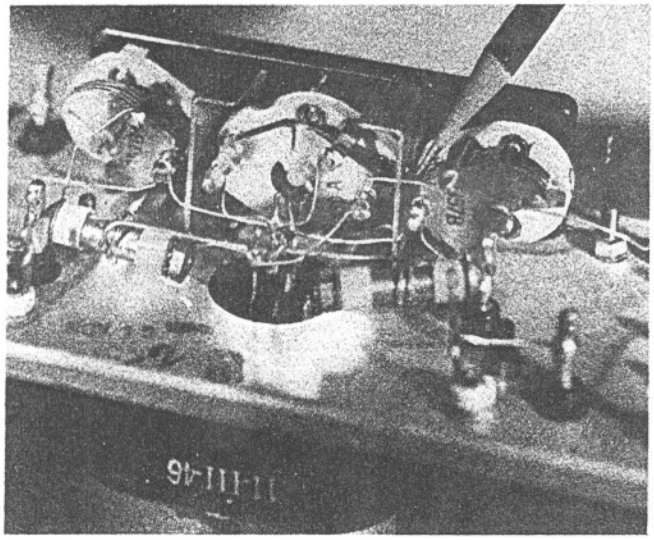

Figure 2—Gap across which filaments actually bridged

being manufactured, much of it embodying electrical circuits, some thought will have to be given to the design and the finishes of the metal components in order to avoid conditions favorable to the growth of these whiskers. Complete failure of a piece of equipment could result because of a low impedance or short circuit caused by the presence of these filamentary growths at a critical point in a circuit.

Published Data

Actually there is very little information in the literature concerning these "whiskers" other than an article by Cobb.[1] This problem does not concern the filmy white or mosaic-like growths sometimes found on cadmium or zinc surfaces, nor a type of "blistering" which develops on such surfaces and is described by Bowerman.[2] This is specifically pointed out since a number of published articles and private communications to which reference was made are concerned with these aspects of the corrosion problem and not with the filamentary growths of the type discussed herein.

It is true that some of the treatises on chemistry, such as that of Mellor,[3] mention filamentary growths on metals, e.g., on silver in the presence of sulfur, but such growths are produced only under exceptional conditions of environment or temperature. Liebhafsky[4] refers to needles which sometime form on vacuum tube leads during the annealing process. Here again this is under very specific conditions and is a result of oxidation at an elevated temperature.

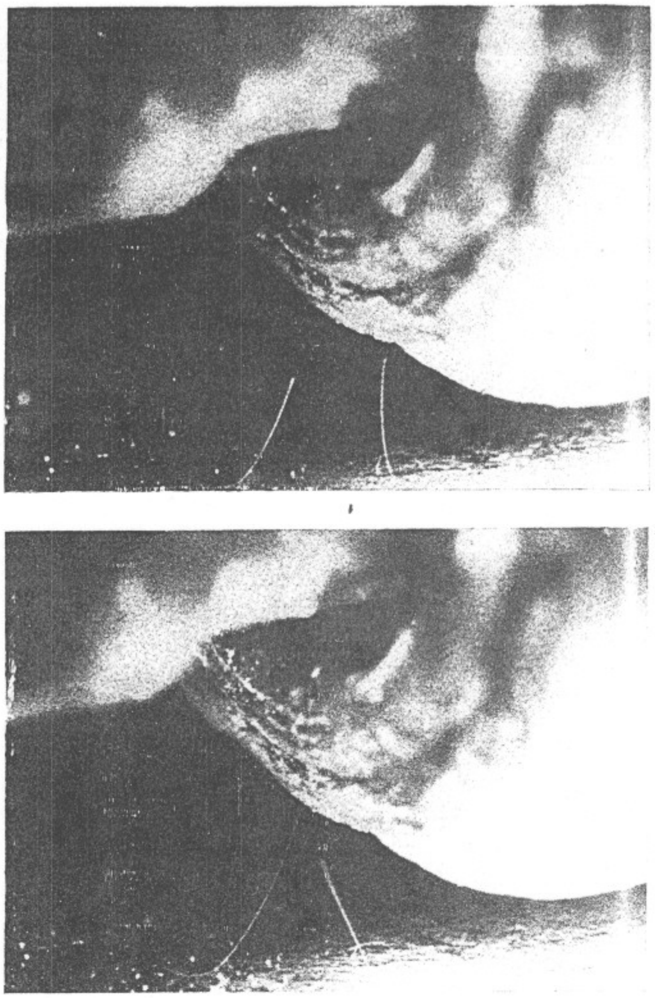

Figure 3—Close-up of whisker bridging gap between condenser post and mounting plate before and after application of voltage

Schenck, Fricke and Brinkman[5] refer to "hair crystals" which they observed, but these were formed when metals such as copper or silver were heated with the corresponding metal sulfides.

Kiplinger[6] describes a method of growing metal crystals from a solution of the metal salt, in particular tin crystals grown from a tin chloride solution. However, it was found that this procedure developed crystals which were much coarser than the whiskers and were quite unlike them in appearance.

Description of the "Whiskers"

The whiskers whose presence adversely affected the operation of the filters and which were responsible for the present study, had developed on cold rolled steel parts which had been zinc plated. While not visible when casually examined, illumination with an intense light beam disclosed the presence of numerous strongly reflecting filaments on the metal surfaces in question. Examination at a magnification of 20 diameters revealed the discreet nature of the filaments. They were quite flexible and moved in the slightest air current. While in general individual fibers had developed perpendicular to the plane of the metal surface, there were also radiating clusters of fibers at various points.

The length varied, some of the whiskers being so glazed porcelain crucible at the bottom of the jar. Figure 4 shows one of the jars, and a glass stand with the specimens in place.

To determine in general the influence of temperature and humidity on susceptibility to whisker growth, and in particular whether temperature changes or condensation of moisture were necessary, four conditions were maintained as follows:

- A. Temperature maintained constant at 110° F.
  1. Initial relative humidity in containers 35 per cent
  2. Initial relative humidity in containers 90 per cent
- B. Temperature cycled between 80° and 110° F., two cycles per day with a temperature change of 1 degree per minute at the end of each cycle.
  1. Initial relative humidity in containers 35 per cent
  2. Initial relative humidity in containers 90 per cent

In Table I are shown the metals, both electroplated and solid, which were first included in the study. Each of the metals was electroplated in three thicknesses—0.00005, 0.0002 and 0.0005 inches, to determine whether whisker growth was a function of coating thickness. Other materials were added subsequently.

In Table II are listed the organic materials used as contaminants. With the exception of the lacquer, these were exposed in the form of fine drillings contained in porcelain crucibles within the glass containers. The crucibles containing lacquer were filled and then inverted, leaving only a coating on the inside walls. Allowed to stand freely exposed for 18 hours, most of the solvent evaporated prior to use. For purposes of comparison a number of test jars were included in which there were no organic materials of any kind.

As the work progressed and more observations were made, a number of questions arose and additional tests were started from time to time. Tin developed the whiskers to an even greater extent than had zinc or cadmium and, as the type of organic contaminant did not appear to be a factor, the greater part of this later work was carried out with specimens plated with a thin coating of tin (0.00005 inch) exposed to a relative humidity of 90 per cent in the presence of phenol fiber.

The reasons for including a number of these specimens may not be self evident and it may be well to point out here some of the questions which arose early in the study and which seemed to require attention before proceeding too far. First, to be certain that initially there were no whiskers present following plating, specimens were examined immediately after being removed from the plating bath. Found free of whiskers, they were put under test and after several months were found to have developed the whisker growths. As a further precaution, plated specimens were immersed in hydrochloric acid following the plating operation, rinsed and put under test. Again whiskers developed.

That an increase in temperature will accelerate the growth of whiskers was shown by maintaining tin plated steel specimens in a high relative humidity at a temperature of 160° F instead of 95° F. Whiskers were found to have developed in a matter of less than 5 weeks. As mentioned earlier, however, the major part of the work was carried out at a lower temperature.

During the first part of the investigation, whiskers were found only on electroplated iron, and the question naturally arose as to whether plating itself was in some way responsible for the growths, and also whether iron as base metal was necessary. The following types of specimens were prepared to answer these questions:

1. Tin applied to bare steel by methods other than electroplating
   - A. Hot dipped
   - B. Sprayed
   - C. Vaporized
2. Tin applied to surfaces other than bare steel by methods other than electroplating
   - A. Evaporated onto mica strip
   - B. Evaporated onto zinc coated specimen
   - C. Evaporated onto black oxidized steel part
3. Tin electroplated on to surfaces other than steel
   - A. Brass plated with tin
   - B. Brass first iron plated and then tin plated
   - C. Steel first copper plated and then tin plated

As it is known that electroplated metals are often under considerable strain, the point was raised that possibly the tin might be in such an unstable condition that the whisker growth accompanied a physical change as the coating gradually changed to a more stable form. Accordingly, some of the tin-plated steel specimens were heated in a vegetable oil at 250° C for 5 minutes to determine whether the usual fused tin coating was susceptible to whisker growth. Other tin coated specimens were maintained at 165° C for 10 minutes and then slowly cooled in an effort to relieve any strains. Still others were cold worked following plating and some waxed and polished.

While it was noted that whiskers had developed on electroplated tin in a container in which initially there had been a relatively low humidity, it was thought that perhaps moisture sufficient to cause the growths to develop had been given up by the various parts in the container. A test jar was therefore prepared in which, in addition to the specimens and the organic contaminant, a dessicant, aluminum oxide, was present.

Another container was prepared in which there was no organic contaminant but in which were placed both a dessicant and an absorbent (aluminum oxide and activated charcoal respectively) in an attempt to remove moisture and also any organic vapors.

Rather early in the study it was found that tin plated specimens had developed whiskers in the absence of an organic contaminant. These specimens, however, had been plated in a bath containing cresol-sulfonic acid and there was the possibility that organic matter from the bath, retained in the tin coating, might be responsible for the growths. Additional specimens were plated from an alkaline stannate bath containing no organic additives. Others were plated from a bath made up from reagent grade inorganic chemicals only, including sodium stannate, sodium hydroxide and hydrogen peroxide.

As the acid pickle prior to plating might have had some effect on the surface structure conducive to whisker growth, some of the specimens were tin plated without a preliminary acid cleaning, being cleaned with carborundum powder by abrasion only.

The greatest whisker growths seemed to have developed on specimens with the thinnest coatings. To determine whether or not the growths were actually a function of coating thickness, a number of larger size specimens were plated with wedge shaped coatings. For any one specimen then, thickness of coating was the only variable.

Whiskers had been found on zinc, cadmium and tin plated surfaces. The metal indium lies between cadmium and tin in the Periodic Table of the Elements and it was thought that this also might exhibit the phenomenon of whisker growth. Accordingly, steel specimens electroplated with three coating thicknesses of indium were included.

As the cause of whisker growth had not been established up to this point, the possibility was considered that the growths might have developed as

TABLE III

Specimens Which Developed Whiskers Within 2 Years[1]

	Low Relative Humidity	High Relative Humidity
No Contaminant Present:		
Plating Thickness, Inches:		
0.00005	Cd	Sn
0.0002	Cd	Sn
0.0005	....	....
Solid Metal	....	....
Organic Contamninant Present:		
Plating Thickness, Inches:		
0.00005	Cd Zn	Sn Cd Ag*
0.0002	Cd Zn	Sn Zn Ag*
0.0005	Cu* Sn Cd Zn Ag*	Sn Zn Ag*
Solid Metql	Cu* Ag*	Sn Zn Ag* Al[2]

* On Silver and Copper whiskers developed only in presence of sulfur (hard rubber).

[1] Other metals being studied may also develop whiskers, but have not been under test for a sufficient length of time.

[2] Alcoa 750 aluminum alloy, 1% Cu, 1% Ni, 6.5% Sn, balance Al.

TABLE IV

Specimens Which Up to the Present Time Have Not Developed Whiskers

METAL		Length of Time Under Test, Years
Plated	Solid	
Nickel		2
	Nickel	2
Copper*		2
	Copper*	2
Silver*		2
	Silver*	2
	Aluminum (Alcoa 3S Alloy)	2
	Aluminum (Alcoa 24S Alloy)	2
Indium		½
Lead		⅓
Terne Plate[1]		⅓

* Except in presence of sulfur (hard rubber).

[1] A low tin, lead coated steel.

TABLE V

Metal or Metal Coated Specimens* Specially Prepared or Treated

	Developed Whiskers
Tin Plated, Waxed	+
Copper Plated, Tin Plated	+
Tin Plated, Heated at 165° C.	+
Brass, Tin Plated	+
Tin Plated, Dessicant in Container	None in 8 months
Tin Plated, Dessicant and Activated Charcoal in Container	None in 8 months
Tin Plated, Acid Dipped Subsequent to Plating	+
Hot Tin Dipped	+
Tin Sprayed	None in 5 months
Tin Evaporated	+
Tin Plated, Fused	+
Brass, Iron Plated, Tin Plated	+
Tin Plated, but Base Metal Abrasion-Cleaned Only	None in 5 months
Tin Plated in Bath Made Up From Reagent Grade Inorganic Chemicals	+
Tin Plated, Mechanically Worked	+
Tin Plated, Maintained at Elevated Temperature	+ Within 4 weeks
Tin Plated in Form of Wedge	+ At thin end within 6 months
Tine Evaporated onto Mica, Zinc, Black Oxidized Iron	+
Tin Plating Solution Residues on Steel	None within 6 months
Zinc Evaporated onto Paper	None within 8 months
Cadmium Evaporated onto Paper	+

* On iron unless otherwise indicated.

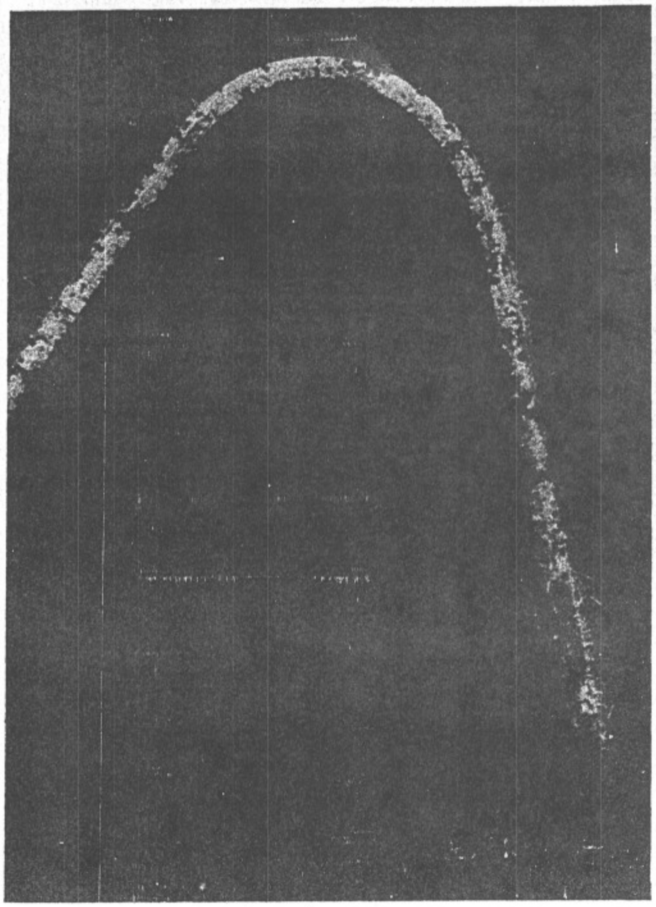

Figure 5—Whiskers on tin plated steel

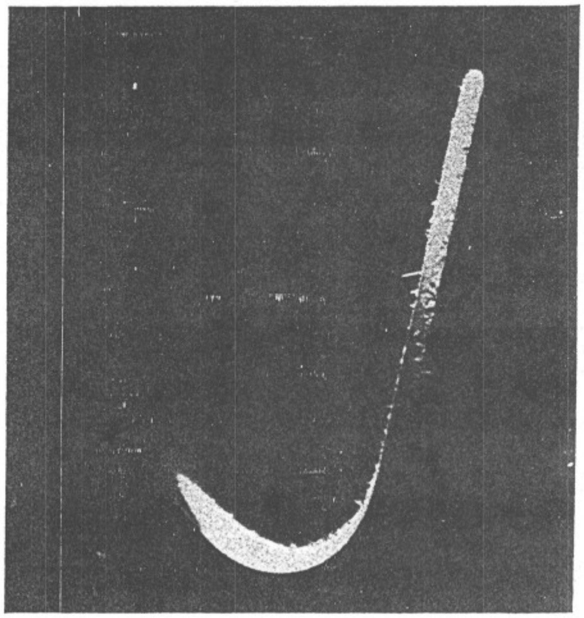

Figure 6—Whiskers on cadmium plated steel

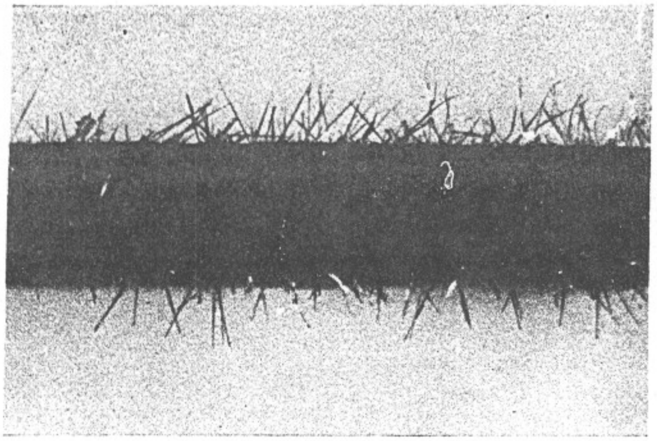

Figure 7—Whiskers on silver plated steel in presence of sulfur

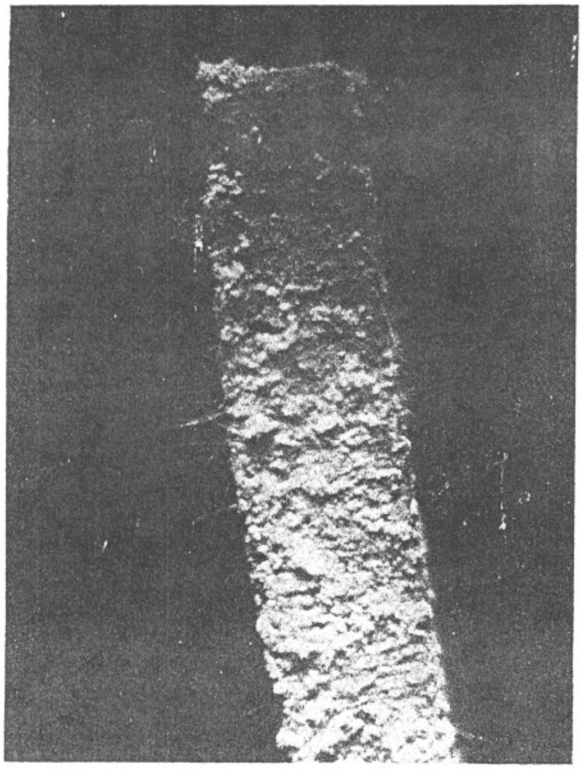

Figure 8—Whiskers on solid zinc

a result of a displacement reaction involving the basis metal and traces of entrapped plating solution. Thus, iron conceivably might react with residual tin salts, being converted to the corresponding iron salt, with tin precipitating in filamentary form. Accordingly droplets of the tin plating solution, both dilute and concentrated, were placed on chemically cleaned steel specimens and allowed to evaporate. These specimens were then put under test.

As one further step toward determining the influence of the basis material, another type of specimen was exposed. Paper was coated by evaporating metal on to it while under vacuum. By this treatment a layer of metal approximately 500 Å thick was obtained. Samples of paper so coated were suspended in the test jars.

Sometime after the study had been started, it was discovered that whiskers had been found on pieces

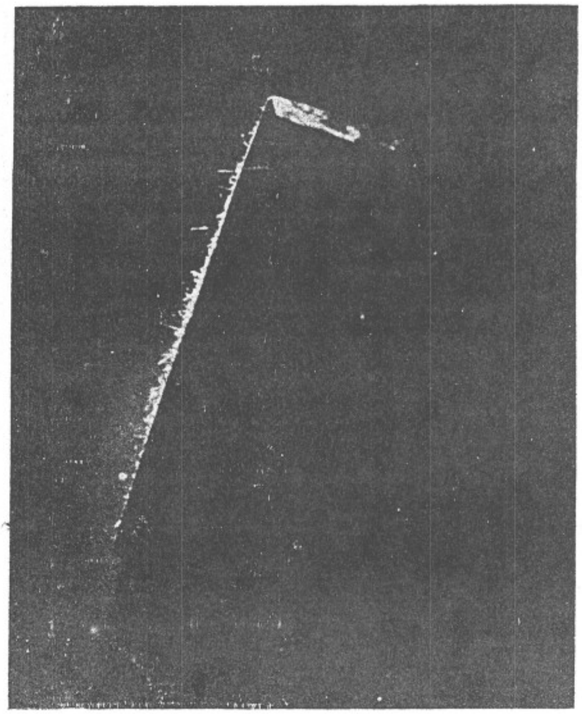

Figure 9—Whiskers on aluminum alloy

of an aluminum casting alloy[7] (Alcoa 750 alloy, 1% copper, 1% nickel, 6.5% tin, remainder aluminum). Accordingly, this material was added to those already under test.

The specimens were examined at intervals for the presence of whiskers and the findings to date are summarized in Tables III, IV and V. In Table III are shown the effects of both humidity and the presence of organic contaminants, cycling of the temperature having had no effect. It will be noted that growths developed on both copper and silver, but only in the presence of sulfur (hard rubber). These growths were unlike the whiskers found on specimens of other metals, being brilliant black, often dendridic or tapered, stiff, about 1/32 inch long, and only on areas which were severely tarnished.

Figures 5 to 9 show some of the specimens which developed whiskers and which are referred to in Table III. In Table IV are listed the metals which as yet are free of whiskers although, as in the case of some of the specimens in Table III, observation after a longer period of test may disclose the presence of growths.

Table V is a summary of the various supplementary treatments and specially prepared specimens as well as of the observations to date. As will be noted, a number of the specimens have been under test only a short time and the picture may change somewhat when later observations are made. Other materials very recently included have not even been listed in Table V as the time of exposure has been too brief to be of any meaning. Figures 10 and 11 show two of the specimens referred to in the table.

As a further attempt to determine the nature of these growths, individual whiskers were mounted across a V notch cut in the small plastic detail shown

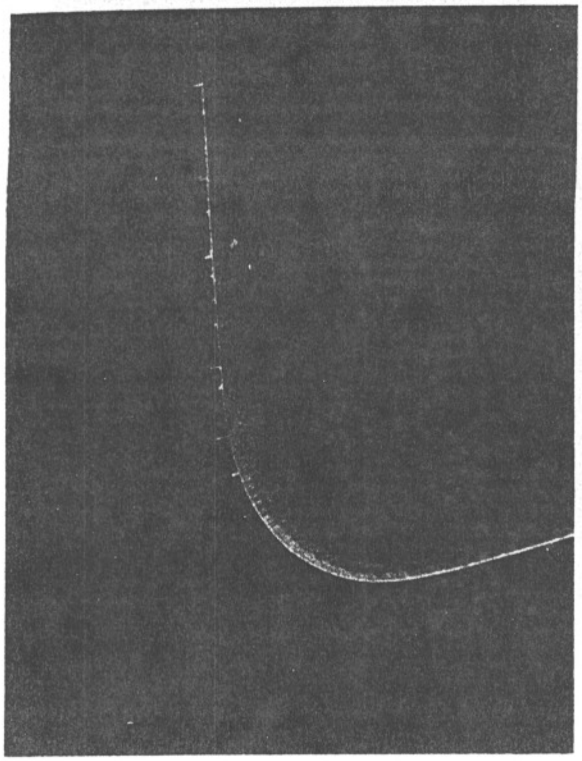

Figure 10—Whiskers on film of evaporated tin

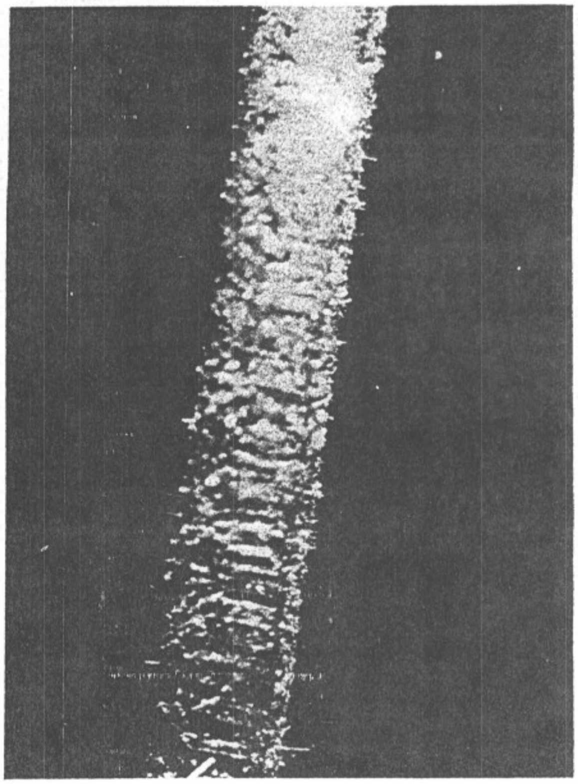

Figure 11—Whiskers on tin plated steel. Tin waxed and polished

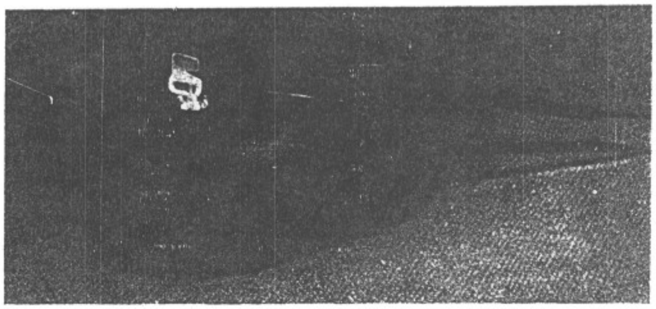

Figure 12—Mount for X-ray studies of single whiskers

in Figure 12, and X-ray diffraction patterns were obtained. It was found that the most satisfactory patterns, weak at best, were obtained using exposure periods of 18 hours or more.

The zinc and cadmium whiskers were found to be single crystals with a close packed hexagonal structure, oriented with an ortho-hexagonal axis parallel to the long axis of the fiber. While the tin whiskers appear to be tetragonal and to be twinned the orientation has not yet been fully established.

Early in the study a tentative explanation for whisker growth was based on the possibility of an impurity in the metal precipitating from solid solution to form a separate phase. Analyses of the metals involved, however, indicate a relatively high purity, maximum impurities being of the order of 0.02 per cent. It is felt that the volume of whiskers formed on many of the specimens is far too great to result from such an action. Furthermore as already pointed out, the X-ray data indicate the whiskers to be crystals of the same metal as that to which they are attached.

Discussion

As this is a preliminary report with considerable work under way and much more to be done, discussion of results must necessarily be brief and sketchy. It may, however, be worthwhile to take stock of the information which has become available and which may be of help in solving the problem.

The major problem of course is to determine the mechanism of whisker growth. Why do these filaments develop on some metal surfaces, under certain conditions, sometimes only after a matter of years? The answer will undoubtedly point to methods of preventing the growths. This latter is of very practical immediate concern, particularly where vital electrical circuits are involved.

There is also the question, at least of academic interest, as to why such growths have not been more widely encountered in the past and why there is available such a limited amount of information. This may be partly answered when the fragility of the whiskers is considered. Slight mechanical shock, air currents or a moderate electrical potential are all that are required to free a surface of the filaments. Further, even in an electrical assembly, unless critical spacings and a sensitive circuit are involved, presence of the whiskers could easily go undetected.

As mentioned previously, many of the specimens and treatments were included to answer specific questions as to the conditions required for the growths to develop, while others were included to shed some light on the actual growth process itself.

While some answers have been obtained additional questions have arisen.

Neither an environment of high relative humidity nor one grossly contaminated with organic material is necessary, as the whiskers have developed under conditions normally considered dry and free of contamination. Whether dessication of the atmosphere and removal of all but minute amounts of organic material will prevent whisker growth remains to be seen.

While at first suspected, electroplating per se is not the cause of the trouble, filamentary growths having developed on specimens coated by methods other than electrodeposition as well as on solid metal. This last also rules out the possibility of a galvanic corrosion process as normally encountered.

As has been mentioned, whisker growth has not been limited to electroplated parts. It has been found, however, that for a given metal whiskers generally have developed first and to a greater extent on the thinner coatings. This was noticed with regard to the hot dipped coatings as well as the plated coatings and the wedge shaped electrodeposits.

Residues of electrolytes used to prepare specimens for plating cannot be responsible, as growths have developed on solid metal and also on metal coated paper and mica where no cleaning was involved.

Transformation of the individual metal to a more stable form does not appear likely as an explanation, particularly in the case of tin where neither heat treatment nor working markedly affected the tendency toward whisker growth.

Conclusions

At this stage of our study while the mechanism of whisker growth yet remains to be established, there are a few points that have emerged which will be of some guidance in the search. Among those which appear to be most significant, at least at present, may be mentioned the following:

Whisker growths are not limited to electrodeposited coatings and may be found on solid metals as well as on surfaces metal-coated by various methods. The growths may develop in an environment in which there is relatively low humidity and in which there are, at most, only traces of organic material.

The whiskers are not compounds but are metallic filaments in the form of single or twinned crystals.

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