from Materials Protection (March 1962)

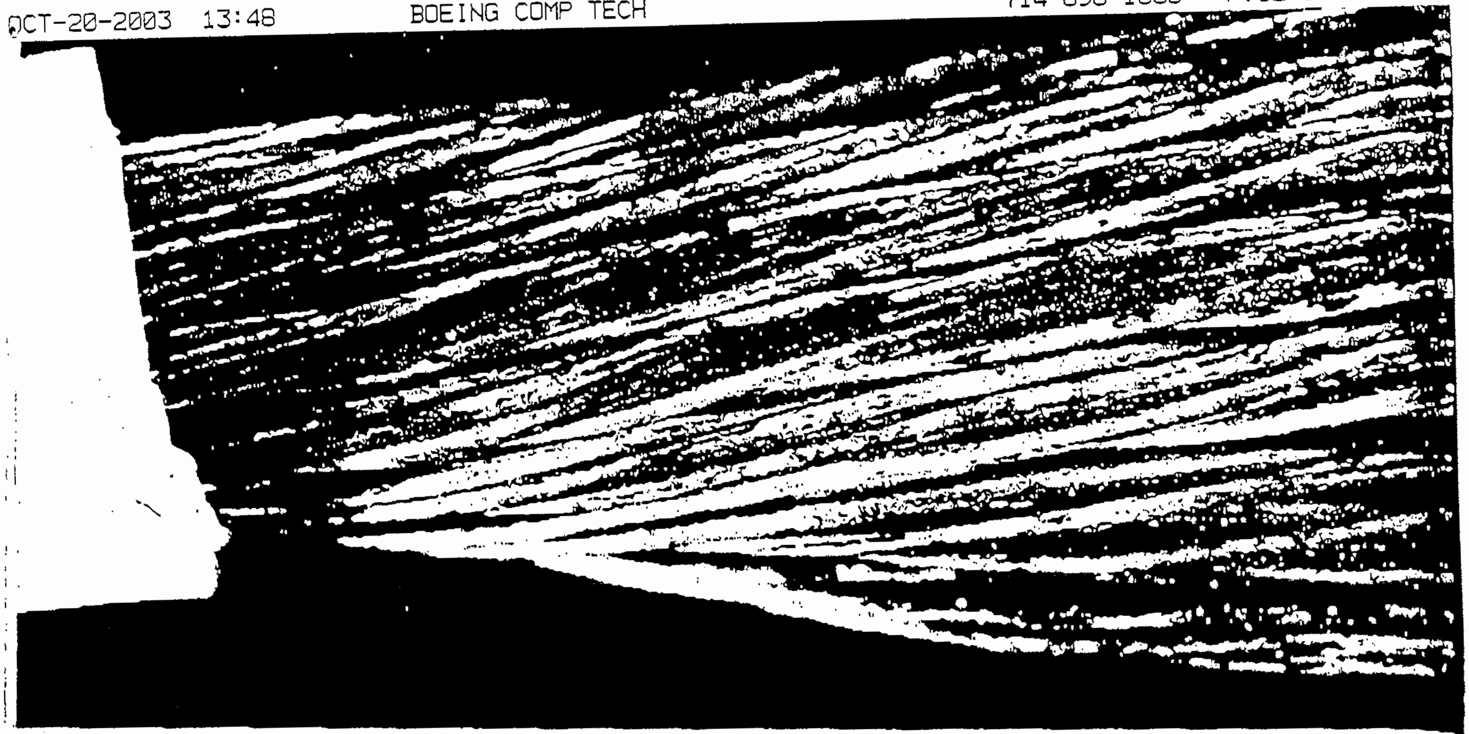

Figure 1—Corrosion products on silver-plated copper wire with Teflon insulation. This corrosion has been called "red plague" by wire manufacturers and users. Also see cover illustration.

RED PLAGUE

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SUMMARY

Describes "red plague" corrosion on silver-plated copper electrical wire insulated with Teflon. Though not a quantitative problem, this corrosion causes serious reliability problems in areospace electronics and similar applications. Gives details of program to determine cause and mechanism of corrosion. Describes method for initiating and sustaining corrosion plus lab tests which clarify conditions necessary for the corrosion process. Includes study of the galvanic cell properties of the copper-silver couple.

ELECTRICAL wire insulated with Teflon is an important material for a variety of modern engineering applications. In particular, this wire is widely specified for aerospace electronics packages because of the superior electrical properties and high temperature resistance of Teflon. However, to use Teflon insulation over conventional stranded copper wire conductors, it is necessary to protect the copper from thermal degradation during the high temperature extrusion and sintering operations involved in the application of Teflon. Wire manufacturers found that a thin coating of silver plate would adequately protect the copper and would not impair other desirable properties of the wire such as solderability and flexibility. Although nickel or tin coatings also are used, silver-plated copper has been the standard conductor material for Teflon insulated wire.

Unfortunately, the silver-plated stranded copper conductors in Teflon insulated wire can be highly susceptible to corrosion. Among wire manufacturers and users, the term "red plague" has been adopted to describe the characteristic red corrosion which appears. Although well known in the industry, this corrosion probably has not been exceptionally great in consideration of the vast quantities of Teflon insulated silver-plated copper wire that have been produced in the past nine or ten years. However, throughout this period, it has remained a persistent, uncontrolled, and unsolved problem. To wire manufacturers, it has represented an elusive quality control problem. To wire users, particularly those in the missile and aerospace fields, it has frequently posed a serious reliability question.

The work reported here was undertaken as part of a study program to determine the cause and mechanism of corrosion in Teflon wire. A method for initiating and sustaining "red plague" corrosion is described, which along with other laboratory tests enabled a clarification of the conditions for the corrosive process to be made. A supplemental study of the galvanic cell properties of the copper-silver couple is included.

Description of Wire Corrosion

The occurrence of corrosion in Teflon coated wire is easily recognized by the singular appearance of the corrosion products on the conductor strands. Typically, the corrosion products are red in color, although occasionally black. The red or black material stands out clearly against the silver-plated conductor strands when the insulation is removed from the corroded wire, as own in Figure 1 and on the cover of is -issue. If the corrosion is only ight, the products occur in scattered, ulated spots on the conductor strands. hen the corrosion is severe, the rands in affected wire sections may coated almost completely with corsion products.

Removal of the corrosion products om even heavily corroded specimens veals the wire strands apparently tact with only a scattering of small ts or breaks in the silver plating. igure 2 shows the same wire section Figure 1 after the corrosion products ere removed by treatment with hyochloric acid.

The extent of corrosion damage to e wire conductor strands can be ssessed in metallographic cross sections. Figure 3 shows a typical section rough a wire specimen which exhibed moderate amounts of external corsion products. Figure 4 is an enlargeent of the heavily corroded strand in e upper right quadrant of Figure 3. is apparent from these figures that, gardless of the external appearance, rrosion damage occurs within the pper cores of individual conductor rands.

As can be seen in Figure 3, a high percentage of the strands are affected and, in many, corrosion of the copper has progressed sufficiently to seriously impair the electrical conductivity and mechanical strength. Figure 4 illustrates the copper corrosion under the silver plate. A break in the silver plate such as was invariably found to be associated with each region of underlying copper corrosion can also be seen in Figure 4, as well as indications of corrosion products which have apparently "crept" outward from the break over the surface of the strand.

Analysis of Corrosion Products and Wire Materials

There are two basic types of Teflon material currently used for wire insulation: polytetrafluoroethylene (TFE) and fluorinated polyethylenepropylene (FEP). Also, there are two different grades of copper commonly used in wire conductors: electrolytic tough pitch (ETP) and oxygen-free high conductivity (OFHC). OFHC copper is a single phase of the pure metal; ETP is an alloy of copper with 0.03 to 0.05 percent oxygen. In ETP copper, the oxygen is concentrated at the grain boundaries as cuprous oxide. Wire samples examined and tested in this work were from several manufacturers and included examples of both types of Teflon in combination with both grades of copper. With respect to the analytical results and corrosion experiments, there were no pertinent differences between the several varieties of Teflon insulated silver-plated copper stranded wire.

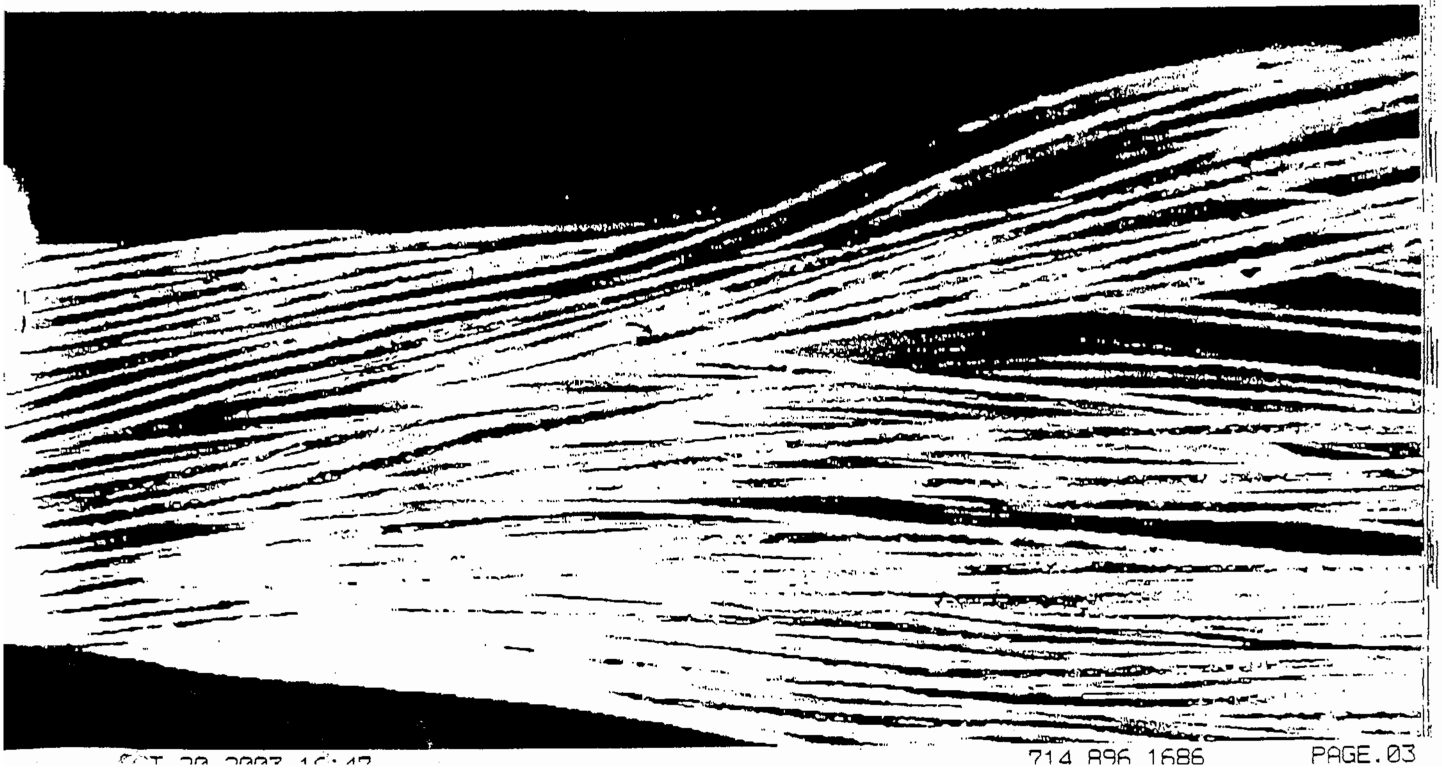
Samples of the corrosion products from a large number of corroded wire sections were subjected to chemical, spectrographic, and X-ray diffraction analyses. The qualitative results were the same in all cases. The red corrosion products were identified as cuprous oxide (Cu_2O) containing traces of cupric oxide (CuO). The black corrosion products were cupric oxide with traces of cuprous oxide. Small amounts of copper sulfate were detected in some samples. The only other substances found were traces of elemental silver and copper. In no case was there any evidence of silver corrosion products.

Various chemical and metallurgical tests were conducted on both corroded and uncorroded wire sections to determine if any anomalies in the conductor or insulation materials existed which

CORROSION

on silver-plated copper wire

Figure 2—Appearance of corroded wire after the external corrosion products were removed by treatment with hydrochloric acid.



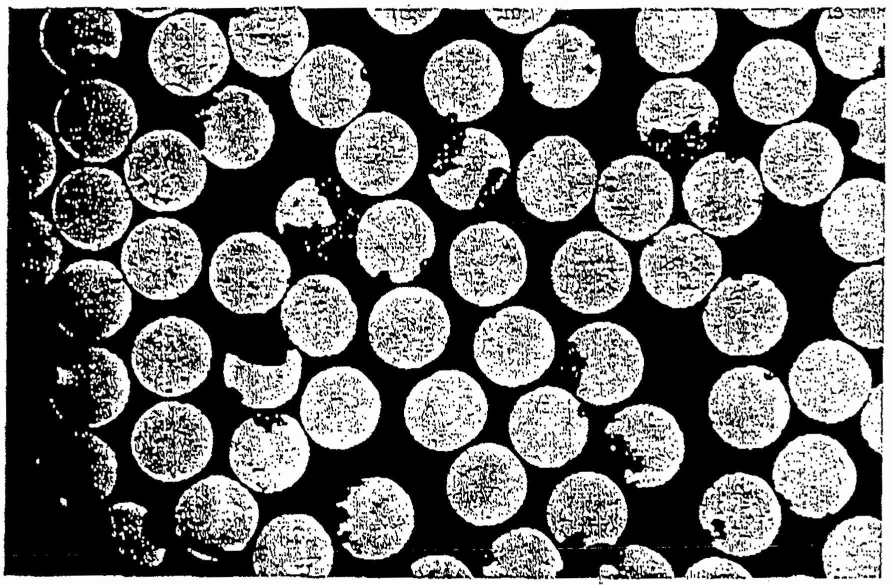


Figure 3—Photomicrograph (36X) of metallographic cross section through a corroded wire conductor.

could be related to the corrosion phenomenon. Infrared and spectrographic analyses of Teflon insulation material indicated no irregularities in the Teflon composition and no contaminants. Metallographic examination of conductors revealed the absence of the grain structure growth such as is usually associated with heat promoted corrosion and which might have been initiated during the high temperature Teflon sintering. There were never any indications of silver migration in the samples.

Breaks in the silver plating were observed in both corroded and uncorroded wire sections. Corroded areas in the copper core were invariably coincident with a break in the overlying silver. Although frequently breaks were found in the plating without accompanying corrosion, there were no cases of corrosion in regions were the silver plate was entirely intact.

Quantitative chemical analyses for silver metal were made on pre-weighed wire sections to determine the average plating thickness on the conductor strands. A minimum of 40 microinches of silver plating is required by most wire specifications. In all wire samples analyzed, sufficient silver was present to provide the minimum plating thickness. More importantly, there was almost the same amount of silver per unit conductor surface area in all cases, and therefore no apparent correlation between average plating thickness and wire corrodability. The amount of water soluble fluoride ion which could be extracted ranged from 165 to 410 micrograms per foot of wire, or the equivalent of only 6 to 10 parts per million. Again, it was significant that the amounts of fluoride ion detected in the various samples did not correlate with the degree of corrosion.

Several preliminary conclusions were reached, based on the analytical results and investigations into the history of corroded wire specimens. First, there seemed to be no inherent chemical or metallurgical condition of Teflon wire which could be shown to predispose to "red plague" type corrosion. Second, the only common, although not exclusive, factor consistently associated with corroded regions of the wire conductors was a mechanical discontinuity or break in the silver plating. Third, several wire lots in which corrosion was later discovered were found to have been previously exposed to excessive moisture during storage and handling. This last factor is complicated by the fact that nearly all wire manufacturers immerse the finished wire in water for electrical test purposes although the wire ends supposedly are protected at this time.

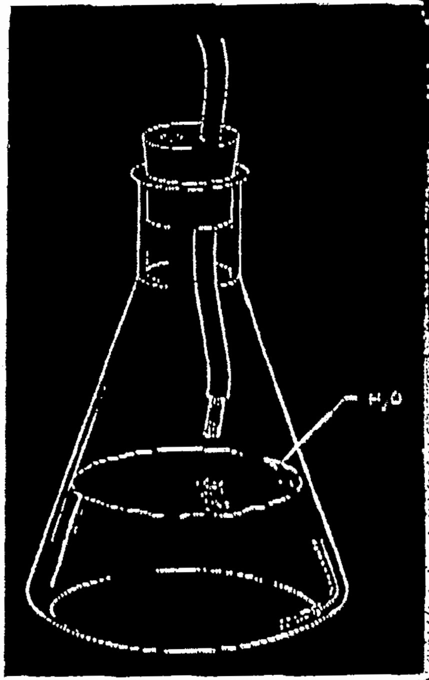
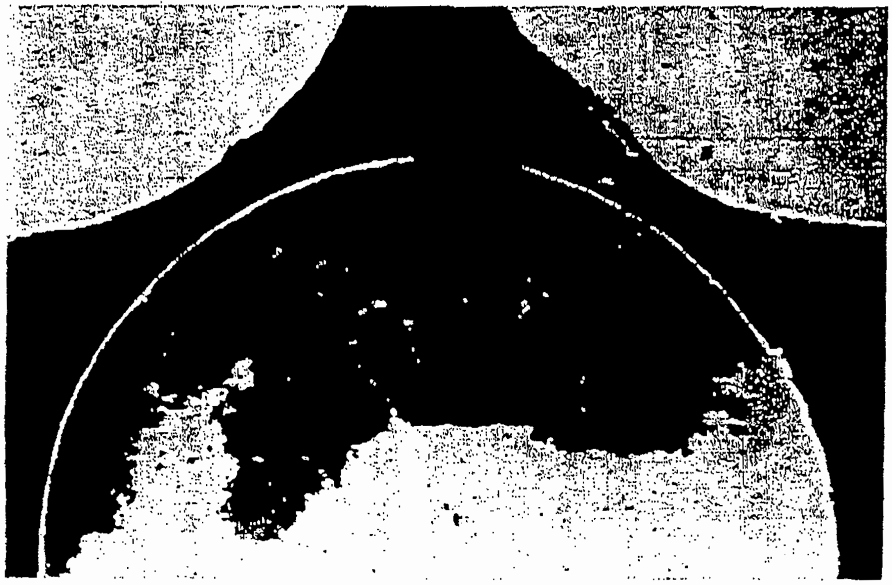


Figure 5—Apparatus for vapor diffusion environment to produce "red plague" corrosion in silver-plated stranded copper electrical wire.

Figure 4—Enlargement (370X) of the heavily corroded conductor strand in the upper right quadrant of Figure 3. Note the corrosion of the copper core under the thin silver plating which has remained intact except for a single break.



Artificially Produced Wire Corrosion

Probably the greatest obstacle to the resolution of the "red plague" corrosion problem has been the inability to reproduce this corrosion in the laboratory under controlled experimental conditions. A means to accomplish this has not previously been reported. All attempts to induce the corrosion by chemical treatment, heating, or conventional humidity or salt spray exposures either give negative results or cause a different type of corrosion.

In light of the exhaustive analytical studies and limited but suggestive his-

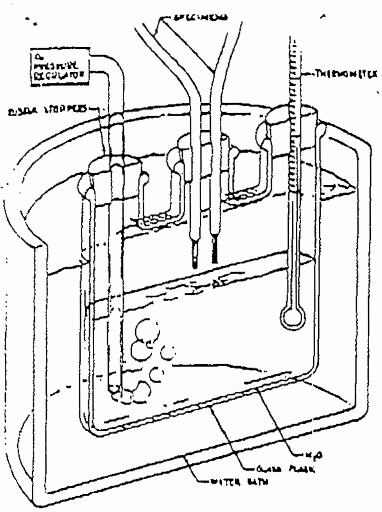


Figure 6—Apparatus for wire corrosion susceptibility test.

ories of corroded wire discussed above, t was deduced that a galvanic process vas the most likely corrosion mechaism. All essential factors for galvanic orrosion appeared to exist in Teflon vire which was susceptible to the "red lague." A dissimilar metal couple was xposed at breaks in the silver plating; noisture was known to have been resent, at least in some cases; there vas an obvious metallic connection beween the dissimilar metals; and air oxygen) was available to the system.

The environmental system which was ltimately successful for artificially proioting "red plague" corrosion in Teflon nsulated silver-plated copper stranded ire is shown in Figure 5. This paricular arrangement was chosen to proide a continuous exposure of the wire onductor to moisture and air by a atural vapor diffusion process.

For this test, wire sections used were pproximately three feet in length with to ¾ inch of the insulation removed om each end. The wire was inserted one hole of a two-hole stopper, and e stopper then fitted into the neck of glass flask partially filled with water, s shown in Figure 5. Length of the ire within the flask was adjusted so at the lower end was slightly above e water surface. The other end of the ire was supported directly above the ask. The second hole in the stopper as left open to the atmosphere. Thus, ere is a slow but constant diffusion water vapor and air upward through e interstices in the wire conductor rands from the flask where the relave humidity is virtually 100 percent the top end of the wire where the lative humidity is the laboratory ament, approximately 50 percent.

Wire specimens which were suscepti e, i.e., had breaks in the silver plat g, developed noticeable corrosion ithin 48 hours when subjected to this st. The corrosion was confirmed by alysis to be identical to the naturally curring "red plague." Furthermore, icroscopic examination before and ter test indicated a definite correlation between the number of breaks in the silver plating and the actual amount of corrosion during the test.

Several variations on the basic laboratory corrosion test were performed to determine the factors essential to the corrosive process. When the lower end of the wire was submerged in the water in the flask, the corrosion rate was much slower. This was probably because the air flow through the wire was blocked off. Similarly, when the entire length of the wire sample was placed in a 100 percent relative humidity environment, the corrosion was greatly suppressed. In this constant environment, the low corrosion rate is undoubtedly attributable to the depletion of oxygen from the stagnant air in the conductor interstices.

The only variation on the basic test method which increased the rate of corrosion was to bubble oxygen through the water in the flask through a tube inserted in the stopper's second hole. This technique not only increased the oxygen supply but also caused a forced flow of the moist gas through the wire.

When silver-plated copper stranded conductors with the Teflon insulation removed were tested, the rate of corrosion was considerably less than for wire from the same lot with the insulation intact. Apparently, the coating of insulation serves to constrain the column of high humidity air about the conductor strands and maintain the gradient necessary for vapor diffusion.

Sections of different types of wire also were subjected to the laboratory test for comparison. Several samples of silver-plated copper stranded wire with polyvinyl chloride insulation were tested. All these wires exhibited considerable damage to the silver plating; all suffered severe corrosion. Apparently, Teflon insulation material is not a prerequisite for "red plague" corrosion. Teflon insulated nickel-plated, tin-plated and silver-over-nickel-plated copper stranded wire developed no indications of copper corrosion when tested. Slight corrosion did occur on some of these samples, but it was the nickel or the tin that was attacked, not the copper.

Adaptation of Corrosion Test for Wire Inspection

Wire manufacturers and users need a rapid method for routine inspection of Teflon wire for susceptibility to "red plague" corrosion. The vapor diffusion corrosion test offers one approach to such a method, particularly if conditions are modified to accelerate the corrosion process so that the test period can be reduced and if the corrosion products are quantitatively analyzed for copper to provide a numerical indication of the corrodibility.

A considerable increase in the corrosion rate can be obtained by raising the temperature and introducing pure oxygen under slight pressure to the flask as described above. As a standard corrosion susceptibility test, a temperature of 140 F (60 C), and an oxygen

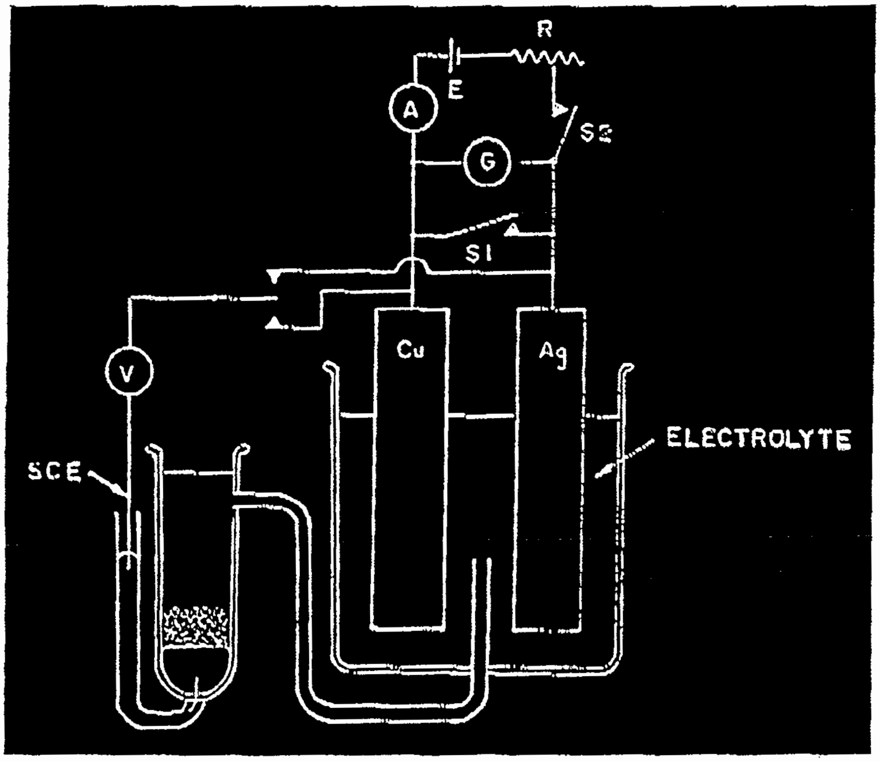


Figure 7—Diagram of copper-silver cell with saturated calomel electrode (SCE) for solution potential measurement and zero-resistance ammeter circuit for galvanic current measurement.

pressure of 1 to 2 psig were used. The temperature was maintained by placing the flask in a water bath, and oxygen was metered to the flask at a rate to balance the flow out through the wire sample being tested. This combination of elevated temperature and forced flow of moist oxygen through the wire accelerated the process sufficiently to readily differentiate between the corrodability of various wire specimens within the 24-hour exposure. The apparatus for the corrosion susceptibility test is diagrammed in Figure 6.

After a wire specimen was exposed to the standard corrosion susceptibility test, the insulation was removed and the corrosion products extracted from the conductor by treatment with a known volume of 6 normal HCl (hydrochloric acid). An aliquot of the HCl solution then was prepared for colorimetric analysis of the copper content as the ammonia complex.

Samples of wire from the same section or roll of wire as specimens used for the corrosion susceptibility test were examined under magnification for silver plating damage and then analyzed for copper compounds to establish a control level. Again, a correlation between the amount of damage to the silver plating and wire corrodibility is indicated.

TABLE 1—Typical Solution and Galvanic Cell Potentials of Copper and Silver in Chloride and Fluoride Electrolytes

Anode-Cathode	Volts	
	1.0M. KCl	1.0M KF
Copper-SCE........	0.18	0.15
Silver-SCE.........	0.05	−0.005
Copper-Silver.......	0.13	0.20

Copper-Silver Galvanic Cell Experiments

To further clarify in chemical terms the wire corrosion phenomenon observed to occur naturally and in the vapor diffusion tests, a series of experiments was conducted with the copper-silver galvanic cell. The galvanic behavior of the copper-silver couple was studied by conventional electrochemical cell techniques. The individual electrode solution potentials relative to a saturated calomel reference electrode, as well as the open circuit cell potentials, were measured in each electrolyte to provide an indication of the cell reaction mechanisms. The galvanic current in the cells was determined as a function of time with the zero resistance ammeter circuit.[1] A diagram of the circuit arrangement used is shown in Figure 7.

Particular attention was given to fluoride electrolytes in the galvanic cell tests because fluoride ion was the only electrolyte forming contaminant detected in the analysis of wire specimens. Also, it has frequently been assumed that, due to the presence of the fluorine-containing Teflon insulation, either fluorine, fluoride ion, or Teflon itself must somehow be involved in the wire corrosion process. Chlorine electrolytes were used as an example of an active copper-silver cell for comparison with the fluoride electrolyte system. However, the experiments with the copper-silver couple in chloride and fluoride electrolytes, as discussed below, were not intended to indicate the chloride contaminants give rise to "red plague" wire corrosion.

Typical values for the solution and cell potentials of copper and silver electrodes are given in Table 1. The response of the copper electrode in fluoride electrolyte was exceptional in that the potential was very unsteady. Transient initial potentials as high as 0.15 volt anodic to the saturated calomel electrode (SCE) were recorded, but even during the course of the measurement the potential dropped to as low as 0.05 volt. Similarly, the "open circuit" cell potential of the copper-silver couple in molar KF solution would first read to 0.2 volt, then immediately drop to less than 0.1 volt. Apparently, the slightest interaction of the copper with fluoride ion sets up a strong polarizing or passivating process. Although the solution potential of copper is more anodic in chloride than in fluoride solution, the reverse effect on the silver electrode causes the initial galvanic cell potential to be greater in fluoride solution. The measured silver solution potential was about 0.04 volt anodic to the SCE in molar KCl but was 0.05 volt cathodic to the SCE in molar KF. Actually, in KF solution, the solution potential of the silver electrode cannot be measured by the test method used. Rather, the reduction of oxygen at the silver electrode is the cathodic reaction which governs the potential difference which was detectable because the true silver solution potential is cathodic to the calomel cell potential.

Figure 8 provides a direct comparison of the galvanic currents as a function of time for the copper-silver couple in molar KCl and KF solutions. Characteristics of the current-time curves for the two electrolytes are strikingly different. In the chloride solution, the galvanic corrosion current changes little with time. After a slight drop from its initial value, the current actually increased, probably due to an increase in electrolyte conductivity and pH as the cell reaction proceeds and more ions are formed. In the fluoride electrolyte, however, the initial current, although substantially higher, dropped rapidly to low levels. The steady state corrosion current of the copper-silver couple was more than 20 times less in the fluoride solution, even though initial currents were about three times greater than in the chloride solution.

To determine the effect of an increased oxygen supply, air was bubbled through the electrolyte between the electrodes. Similar cells were tested with helium instead of air, so that the effects of the agitation alone caused by the rising gas bubbles would be known. In KF electrolyte, neither air nor helium injection had an appreciable effect on the copper-silver galvanic current. There was a momentary small increase in the current when the gas w ,was started, but the current ickly dropped to almost the same ue as before. When helium was in- duced to the cells containing KCl ctrolyte, the current immediately reased more than twofold and then wly decreased. Aeration of KCl elec- lyte cells caused a rapid and sus- ned fourfold increase in the galvanic rrent.

The galvanic current measurements the chloride ion cells provide an in- ation of the effect of coupling with ver on copper corrosion. The steady ate galvanic current densities for the pper-silver cells with molar KCl solu- n were on the order of 10^{-6} amperes r square centimeter. This corresponds a copper corrosion rate of about 68 ld (milligrams per square decimeter r day). The average corrosion rate ported in the literature for copper one immersed in sea water is 12.4 ld. A rigorous comparison of these lues probably is not justified, but as example it indicates that galvanic upling with silver can appreciably rease the corrosion rate of copper posed to salt water.

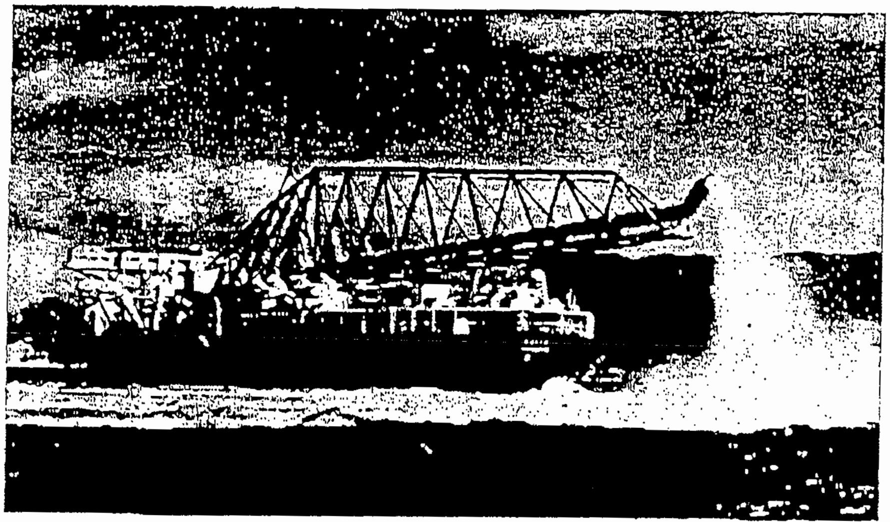
Causative Factors in "Red Plague" Corrosion

Virtually all stranded copper conduc- rs, when examined under high magni- ation, exhibit some degree of damage the silver plating. These imperfec- ns range from minor scuffs on the ver surface to deeper scrapes and ts which penetrate the plating and ach the copper core. In all cases, the mage appeared to have been the re- lt of mechanical abrasion. Unfortu- tely, there is considerable opportunity r mechanical abrasion of the silver ating during wire manufacture and e.

In the manufacture of conductors for flon wire, the individual strands of pper are silver-plated after being awn from rod stock to an interme- ate gauge size. The amount of silver posited is calculated so that when e plated strand is subsequently drawn the final gauge size, the silver coat- g will be the required minimum thick- ss, usually 40 microinches.

The individual silver-coated copper res are then wound and twisted to- ther to form the finished stranded nductor. There are high pressures and avoidable abrading action between e strands during the stranding opera- n. Due to the thinness and natural ftness of the silver plating, most of e damage to the coating and exposure copper probably occurs at this time.

To a lesser extent, similar abrasion tween conductor strands undoubtedly curs when the wire is wound and un- und on reels or spools, and even ring the normal flexing and bending quired for assembly. The effects of e mechanical stresses during fabrica- n and handling possibly are aggra- ted by the inherent granular, multi- ystalline and often porous nature of ectrodeposited silver. It is known that ver plating is susceptible to poor



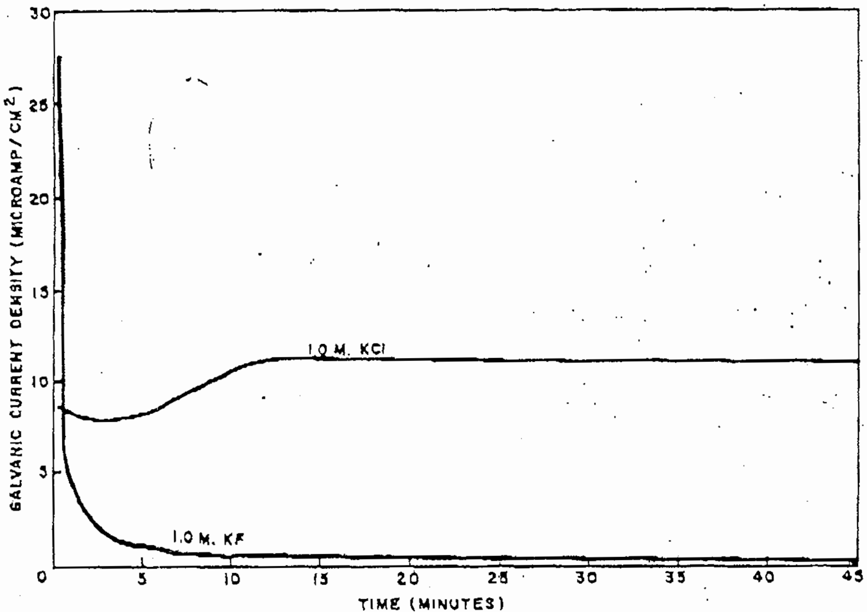


Figure 8—Change of copper-silver galvanic current with time in potassium chloride and potassium fluoride electrolytes.

adherence and tends to flake or peel under abrasive or mildly corrosive conditions.[3]

Once copper is exposed through breaks in the silver plating, environmental factors determine whether or not corrosion will develop. The dissimilar couple between copper and silver can promote corrosion of exposed copper; however, vapor diffusion corrosion tests show that particular environmental conditions must prevail to sustain the corrosion process. Although moisture must be present, the corrosion rate in wire is controlled by the availability of oxygen to the corroding sites. Both the adaptation of the vapor diffusion method to the corrosion susceptibility test and the galvanic cell experiments with aeration show that the corrosion rate is primarily dependent on the cathodic half cell reaction which requires oxygen. Extensive corrosion in Teflon wire is expected to occur only where there is a continual supply of air to a moist area. Thus, most "red plague" corrosion is found near wire ends or pinholes in the insulation.

One other property of the copper-silver galvanic couple as found in Teflon wire bears emphasis as a factor in the corrosion rate. The galvanic cell studies, the vapor diffusion corrosion test results, and theoretical considerations all indicate that this galvanic cell is cathodically controlled. That is, the level of galvanic current, and hence the extent of corrosion, is limited by the rate of the reduction reaction involving oxygen at the silver cathode. With galvanic couples exhibiting cathodic control, the ratio of effective anodic and cathodic areas is a significant factor in determining the amount of corrosion that occurs. As the relative area of the cathodic region is increased, the cathodic current density required to balance the anodic reaction is decreased, and as a result the corrosion rate may be substantially greater. This is exactly the case with silver-plated copper wire conductors, where the anodic areas of exposed copper would typically be small compared to the silver-plated surface area.

The results of the galvanic cell studies and the fact that fluoride compounds were not detected in the wire corrosion products rule out fluoride contaminants as a factor in wire corrosion. It is more likely that fluoride ions, if present in any significant amount, would tend to suppress the wire corrosion process. The solution potential measurements suggest that copper reacts anodically with fluoride ion. The reaction is probably:

$$\mathrm{Cu} + \mathrm{F}^- = \mathrm{CuF} + e^-,$$

which is analogous to the well known

$$\mathrm{Cu} + \mathrm{Cl}^- = \mathrm{CuCl} + e^-.$$

However, the fluoride reaction apparently coats the copper surface with a passivating film of copper fluoride which then inhibits further interaction. The dielectric effects of copper fluoride films have been reported.[3] In chloride solution, such films do not form since the insoluble CuCl can either be converted to the soluble $\mathrm{CuCl_2}$ by secondary oxidation or taken into solution directly as the soluble complex ion CuCl^-_2. Conversely, in fluoride solution, the cupric compound, $\mathrm{CuF_2}$, is also insoluble and the complex cuprous halide ion, CuF^-_4, does not exist as a stable species.

Mechanism of "Red Plague" Wire Corrosion

The experimental results show that wire corrosion will occur simply by exposure to moisture and air. Traces of fluoride contamination do exist in Teflon insulated wire, but the "red plague" corrosion products are exclusively copper oxides. Therefore, the corrosion mechanism probably follows that proposed by Obrecht for the corrosion of copper in pure water with oxygen present.[4]

Copper alone, when exposed to water and oxygen, will corrode by localized cell action on the copper surface. However, in Teflon wire there is an add tional driving force in that the enti exposed copper surface is equipote tially anodic due to the galvanic co pling with silver. The cell reactio then involve the anodic oxidation copper and concomitant reduction oxygen on adjacent silver surfaces. Th complete circuit for the galvanic corr sion current in the wire conductor i cludes the metallic path through t copper and silver, and the electrolyt path through a film of water or mo cuprous oxide corrosion products. C prous oxide is a semiconductor whe dry and when moist provides a reason ably conductive path to complete th galvanic cell circuit.

In the presence of essentially pu water and atmospheric oxygen, the co rosion process in silver plated coppe

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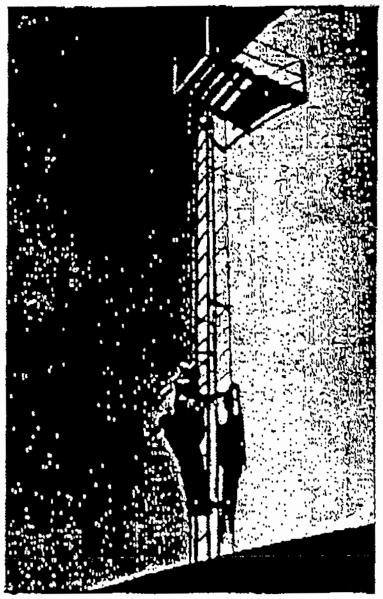

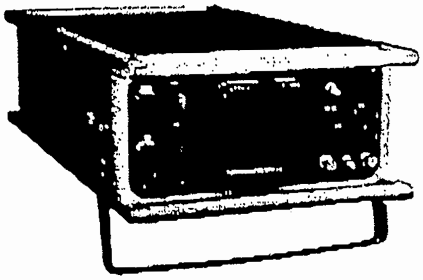

wire would proceed by the fol mechanism:

anodic reaction: $4Cu + 4OH^{-}$ (10
$= 2Cu_2O + 2H_2O + 4e^{-}, E =$
volt

cathodic reaction: O_2 (0.2 atm.)
$(10^{-7} M) + 4e^{-} = 2H_2O, E = 0$
volt

net cell reaction: $5Cu + O_2$ (0.2
$= 2Cu_2O, E = 0.751$ volt

By an analogous mechanism, th potential for the reactions to cupric oxide corrosion products is 0.614 volt. In the rare cases when cupric oxide corrosion products a countered, the wire probably ex enced unusual treatment or the env mental conditions probably were ex tional.

It is significant that by the mechanism water must be presen is not consumed in the corrosion ess. In principle, a given amou moisture once in the wire could su corrosion indefinitely. However, o is consumed by the corrosion pr and must continually be supplied the corrosion to progress.

Conclusions

1. The "red plague" corrosion served in Teflon insulated silver-pla copper stranded wire is due to the mation of copper corrosion produ usually the red cuprous oxide, as result of galvanic corrosion of the co per conductor core at a break in silver plating.

2. Corrosion can occur whenever copper conductor is exposed at a bre in the silver plating and moisture an air (oxygen) are present.

3. The rate of this type wire cor sion is dependent on the availability oxygen to the corrosion sites in th wire.

4. A combination of microscopic ex amination of wire samples for breaks the silver plating and the vapor diffu sion corrosion test can successfully used to inspect wire for susceptibilit to corrosion.

5. Increasing the thickness of th silver plating on the copper conducto and added precautions to minimize th mechanical damage to the conducto during stranding and handling opera tions would probably reduce the inc dence of corrosion considerably.

6. Substitution of nickel plating o nickel under silver plating for the silve plating on copper conductor strand would eliminate "red plague" corrosion These coatings, being anodic to coppe would corrode preferentially and ten to protect the copper core from attack

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