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# INVESTIGATION OF THE TECHNICAL MICRONICS CONTROL (TMC) PROCESS FOR ELECTROPOLISHING VARIOUS METALS

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16. ABSTRACT

The Technical Micronics Control (TMC) process for electropolishing a variety of metals and metal alloys was investigated. The electropolishing solution produced lustrous surfaces on stainless steel, brass, copper, nickel, aluminum, and steel; it produced only semi-bright surfaces on Invar and Rene' 41 but produced a considerable amount of metal smoothing.

The passivation of stainless steel was improved substantially by this method over that of conventional acid passivation methods.

TMC polished surfaces of stainless steel, brass, and carbon steel did not accept an electroplate with good adherence but such surfaces were more resistant to tarnish or corrosion than mechanically polished surfaces.

Based on results of this study, the TMC process is recommended for polishing and smoothing most metals and for passivating stainless steel.



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# INVESTIGATION OF THE TECHNICAL MICRONICS CONTROL (TMC) PROCESS FOR ELECTROPOLISHING VARIOUS METALS


## SUMMARY

An evaluation was made of the Technical Micronics Control (TMC) process for electropolishing a variety of metals and metal alloys. Specific operating details for each of these metals were determined with regard to optimum voltage, time and temperature of the bath. Primarily, the evaluation was based on general appearance of treated specimens. However, other phases of the program included determining the passivating effects of the solution on stainless steel, the tarnish and corrosion resistance of treated specimens of brass and mild steel, and the manner in which a polished surface of brass and stainless steel accepted an electroplate with regard to adherence.

The study showed that this electropolishing method produced smooth and lustrous surfaces on stainless steel, brass, copper, nickel, aluminum, and steel. Smooth but only semi-bright surfaces were produced on Invar and Rene' 41.

The results of subsequent salt spray tests indicated that stainless steel was made considerably more passive by the TMC electropolishing solution than by conventional methods of passivation such as pickling in nitric acid.

Results of controlled high humidity and laboratory exposure tests showed that TMC electropolished surfaces of brass and steel were more resistant to tarnish or corrosion than mechanically polished surfaces, but that "as polished" surfaces of these metals, as well as stainless steel, did not accept electroplates with good adherence.


## INTRODUCTION

Electropolishing is an electrochemical process that, under proper conditions, makes a metal smooth and bright by dissolution of the metal surface. The process is the reverse of electroplating in that the metal being electropolished is made the anode and metal is removed rather than deposited. On the other hand, it is similar to electroplating in that the controlling factor of the solution is the total amount of current that passes through it. This being the case, several other inter-related factors such as current density, temperature (the resistivity of the electrolyte decreases as the temperature increases), and time of treatment have a direct influence on the end results in any electropolishing operation. These variables are adjusted to help produce a particular type of surface finish. Also, as with electroplating, the type and condition of the metal being polished influence the resultant surface finish.

Electropolishing techniques were initially developed for use in the area of industrial decorative finishing. These polishing techniques were reported to provide an improvement in the quality and reduction in the cost of finishing commercial items over that of mechanical finishing. Since the early days of electropolishing, non-decorative uses have evolved and presently many engineering metals are electropolished for various other reasons. Some of the uses and distinct advantages of electropolishing over related mechanical processing methods are as follows (ref. 1, 2): Deburring and polishing in locations inaccessible to mechanical means, (2) removing "smeared" and loosely adhered metal left on surfaces that were mechanically finished, (3) reducing or eliminating surface stresses caused by mechanical processing (4) improving the corrosion resistance of certain metals (5) reducing the friction on bearing surfaces, (6) producing a surgically clean surface by removing contaminants, and reducing the effective molecular trapping surface of vacuum components.

Because electropolishing does enhance the appearance of metals and because of some of the above listed advantages, it has gained extensive industrial use for a number of applications. Many types of baths and procedures have been developed, some of which have been tailored just for special metals and alloys, while other polishing baths are claimed to be effective on a variety of metals and metal alloys. An electropolishing bath, developed by "Technical Micronics Control" was recently reported to polish practically any metal alloy to a fairly high degree of luster, and by changing the operating conditions of the bath, the same bath could be used for producing a matte finish on metal alloys.

In view of this broad claim for the TMC bath and the desirable features generally produced by electropolishing, some of which have direct aerospace application, an investigation of this process was made.

## EXPERIMENTAL PROCEDURE

Two general types of electropolishing solutions are produced by "Technical Micronics Control (TMC)", type I and II. The exact formulation of these baths was held proprietary by TMC, but for evaluation purposes, they reported that the type I solution was basically a phosphoric/sulfuric acid mixture and that type II was basically a chromic/phosphoric acid mixture. The type I solution was recommended for all the common metals where matte finishes were desired and either type I or II was recommended for use on all common metals (except aluminum and brass) where a high luster finish was desired. Type II solution only was recommended for aluminum and brass. Because of the increasing desire for surgically clean and polished surfaces in certain current aerospace applications, the solutions were evaluated, primarily for their ability to produce a high luster finish based on visual appearance of treated specimens. Later in the program, the solutions were also evaluated for their ability to passivate stainless steel and protect brass and steel from tarnish and corrosion in a mildly corrosive atmosphere. Also, to a more limited extent, tests were made to determine if surfaces produced by the electropolishing solutions were receptive to an electroplate.

The metals used in the overall test program included steel (Invar, 4130, 1010), and stainless steel (300 and 400 series), aluminum alloys (2014-T6, 7075-T6, 6061-T6) and to a more limited extent, pure nickel and a nickel alloy (Rene' 41).

Generally, the electropolishing of these metals was carried out according to the recommendation of TMC. However, due to the many variables related to an electropolishing process, the TMC recommendations were very general, and thus, specific operating details had to be determined for the different metals and different types of finishes desired. For example, where a high luster finish was desired on brass, the manufacturer recommended that the polishing operation be carried out at a voltage range of 6 to 15 volts and a temperature range of 130° - 160°F (54-71°C). After varying the voltage within this range, it was found that 12 volts produced the highest degree of luster. The temperature was not critical, and high luster finishes were produced at temperatures in the range of 140°-180°F (60-82°C). Processing details for other metals used in the evaluation program were similarly determined.

Test specimens of the various metals and metal alloys were electropolished under the optimum conditions determined for each of the different metals and the resultant surfaces visually observed for their luster. These specimens were compared with specimens cleaned and electropolished with other baths and also with specimens that were mechanically polished.

After electropolishing under optimum conditions determined for each of the alloys, one group of brass and steel panels was exposed to a plating laboratory environment and another group was exposed to a controlled high humidity environment (relative humidity of 95-98 percent and a temperature of 95-100°F) and compared with mechanically finished panels which were tested simultaneously in the same environments.

Stainless steel panels were contaminated with steel particles by brushing with a wire brush or machining with a steel tool. One group of the panels was electropolished and another group was passivated in a standard nitric acid bath. The two groups of panels were exposed to a 5 percent salt spray environment and observed for the occurrence of rust.

Test panels of brass and stainless steel were electropolished under the conditions determined to be optimum for these metals. An attempt was then made to either electroplate directly on the polished surface, or to plate on the polished surface which had been further treated to remove any film that might have been formed by the electropolishing. Special emphasis was placed on the stainless steel in this particular test because electropolishing has reportedly been used in preparing stainless steels for electroplating (gold plating directly on the stainless steel). Most stainless steels generally require a more severe acid treatment than do other metals, and occasionally need a special "strike" before they can be successfully electroplated. Preparing stainless steels for electroplating by electropolishing would not only be less difficult, but would furnish a brighter and smoother surface upon which to plate, if proven feasible.

## DISCUSSION OF RESULTS

Appearance. - The electropolishing resulted, to some degree, in a brighter and smoother surface on all metals tested. With the exception of a few cases which will be discussed later, the surfaces of all the common metals tested (which included brass, copper, mild steel, stainless steel, aluminum alloys, and nickel) were substantially brightened and smoothed by the electropolishing action. A voltage range of 12 to 14 volts at a temperature of 140-160°F (60-61°C) appeared to be optimum for steel, stainless steel, copper, and brass. The optimum range for nickel was 10 to 12 volts at a temperature of 140-160°F (60-71°C) while a range of 12 to 20 volts at a temperature of

140-160°F (60-71°C) produced the most desirable finish on the aluminum alloys.

The surfaces of the less common alloys tested which included Invar (an iron - nickel alloy) and Rene' 41 (a nickel-base alloy) were smoothed to about the same degree as the common alloys but were much less brightened. A slightly brighter surface was obtained at a considerably wider voltage - temperature range on Rene' 41 than on Invar. In the case of Rene' 41, a fairly bright surface was obtained at voltages ranging from 6 to 15 and at temperatures of 130°F (54°C) to 180°F (82°C). On the other hand, a satisfactory polish could not be obtained on the Invar at voltages appreciably less than 12. The optimum range for this alloy appeared to be from 12 to 15 volts depending somewhat upon the temperature which was more influential than had been observed with the polishing of other metals. The best polishing action occurred on Invar at a bath temperature of between 150° - 170°F( 66-77°C).

In the initial phase of the program, all of the aluminum test panels were prepared from sheet material ranging from about 0.050 to 0.125 inch thick. Generally, all of this sheet material was polished to a fair degree of luster with no particular difference observed between the different alloys. During the latter part of the program there arose a need, in connection with a different program, to electropolish tensile specimens of alloy 7075-T6 (0.180 inch thick) that had been cut from a five inch thick plate material. When these specimens were polished, a significant difference in surface finish was observed between those of the thin sheet material of the same alloy. The surfaces of the plate specimens were etched by the electro-chemical action and were not polished to as great a degree as the sheet specimens. After considerable effort (varying the operating conditions) failed to produce a comparable surface to that of sheet specimens, a metallographic study was conducted to determine the cause of the difference in behavior of the two conditions of the same material. The results of this study showed significant differences in the microstructure of these materials. The sheet materials had much smaller grain size and less intermetallic phases than did the 5 inch plate. (See Figures 1 and 2). The maximum depth of pitting on the 5.0 inch plate material was 0.000625 (Figure 3). These differences in surface appearance resulting from the electropolishing were attributed to the variations in the microstructure which occurred from variations in fabrication technique and heat treating procedures. Similar results were obtained from a further study of another alloy, 2014-T6 aluminum, which further confirmed that grain size significantly affects the degree of electropolish (the finer the grain size, the better degree of polishing).

In conducting polishing experiments with brass, it was found that test specimens reacted differently from the aluminum specimens in the polishing solution. Very similar results were obtained in that some specimens were dull and pitted while others were extremely bright. The results of a metallographic study on specimens of this material indicated that the microstructures did vary somewhat randomly and correlation between this variance and surface appearances could not be established. However, the results of chemical analysis showed that alloy type largely determined the degree of polish or the type of surface finish. For example, yellow brass which contained no lead could be electropolished to a much higher degree of luster than highly leaded brass which contained approximately 1.75 percent lead. Results of further experiments indicated that the presence of any significant amount of lead in brass would, to a degree, affect the electrochemical action and the resultant surface finish.

Passivating Effects. - Tests conducted to determine the passivating effects of the TMC electropolishing solution on stainless steel showed, in general, that this treatment increased the passivity of this metal over that of conventional methods of passivation. Specimens of 321 stainless steel, contaminated and then treated by various methods, were exposed for approximately 560 hours to a salt spray environment. All of the specimens treated conventionally (nitric acid at varying temperatures and concentration) showed a considerable amount of rust after 8 hours and were removed from the test while the electropolished specimens did not show any change until after about 40 hours. At this point, light stain was observed in the center of the specimens, and after 146 hours, light rust was formed. At the end of the test, a moderate amount of rust was present and a few small pits had formed on the specimens. During the course of this study, this test was essentially repeated with the exception of some variations in the conventional acid treatments. In the repeated test, all specimens were exposed for approximately 750 hours, and again, the electropolished specimens showed considerably less rust than did the specimens that were passivated by conventional means.

Tarnish Resistance. - Results of tests showed that the tarnish resistance of brass was enhanced substantially by electropolishing. On the other hand, results of similar tests showed that the rust resistance of steel was only slightly improved by the electropolishing. Specimens of brass (one group mechanically polished and another group electropolished) were exposed to a plating laboratory environment for 25 days. The electropolished panels showed some stain while the mechanically polished panels had changed to a dark color typical of severe tarnish on brass. The only difference between similarly treated specimens of steel exposed to the same environment was that the mechanically polished panels began to rust a few hours sooner than the electropolished panels. At the end of the test, no difference could be observed between the two groups.

Mechanically and electropolished specimens of brass were exposed to a controlled high humidity environment for approximately 190 days and similarly treated panels of steel were exposed to this environment for a few hours. The mechanically polished panels of brass began to tarnish (turn brown) after seven days and gradually became darker as exposure time increased while the electropolished panels retained all of their original luster for 34 days and was still fairly bright after approximately 100 days (Figure 4). At the end of the test period, the electropolished panels were semi-bright with a few dark spots but the mechanically polished panels were severely tarnished (Figure 5). The two groups of steel panels exposed to this environment showed essentially the same degree of rust at the end of a 30-hour test period. The only difference was that the mechanically polished panels began to rust several hours sooner than the electropolished panels.

Electroplating on Electropolished Surfaces. - Test results indicated that surfaces produced by the TMC electropolishing solutions were totally non-receptive to an electroplate in the "as polished" condition and were considerably more difficult to activate for plating than the same unpolished metals. This increased difficulty in activation is understandable from the fact that electropolished surfaces exhibit a fair amount of improvement in resistance to tarnishing and corrosion over non-polished surfaces, as discussed earlier.

Generally, electropolished copper and brass must be activated by cathodic treatment in solutions such as alkaline cleaners or cyanides and followed by dipping in acid solutions such as hydrochloric before plating. Although some degree of adhesion was obtained without the use of cathodic treatment, the acceptance of electroplates with positive and uniform adherence depended upon a more thorough de-passivation treatment.

Electropolished stainless steel behaved similarly to brass and copper in that electroplates were not accepted with a satisfactory level of adhesion. Unlike brass and copper, stainless steel in its ordinary form (unpolished) possesses a very tenacious oxide film which makes it much more difficult to plate. In electropolishing, it is believed that this natural film is removed and the metal is mildly "anodized" again (another oxide film produced by the electrochemical oxidation), making it necessary to reactivate before plating. Whether or not this was the case, the results of this test showed that TMC electropolished stainless steel was left in a passive condition and this passive film had to be removed before electroplating could be carried out successfully.

## CONCLUSIONS AND RECOMMENDATIONS

The TMC method of electropolishing produced bright surfaces on all the common metals and alloys tested which included, copper, brass, steel, stainless steel, nickel, and aluminum alloys. Of this group, copper, brass, and nickel could be electropolished with the greatest degree of luster. It appears that the composition of brass and the microstructure (grain size) of certain aluminum alloys affect the type of surface resulting from electropolishing these metals. Test results show that specimens of brass that contain lead do not polish as well as specimens that do not contain lead. Tests of the aluminum alloys (7075-T6 and 2014-T6) show that the smaller the grain size, the better the degree of polish.

Surfaces of the less common metals tested, which included Invar and Rene' 41, can be made smooth by the TMC electropolishing solution, but cannot be produced with a high degree of luster.

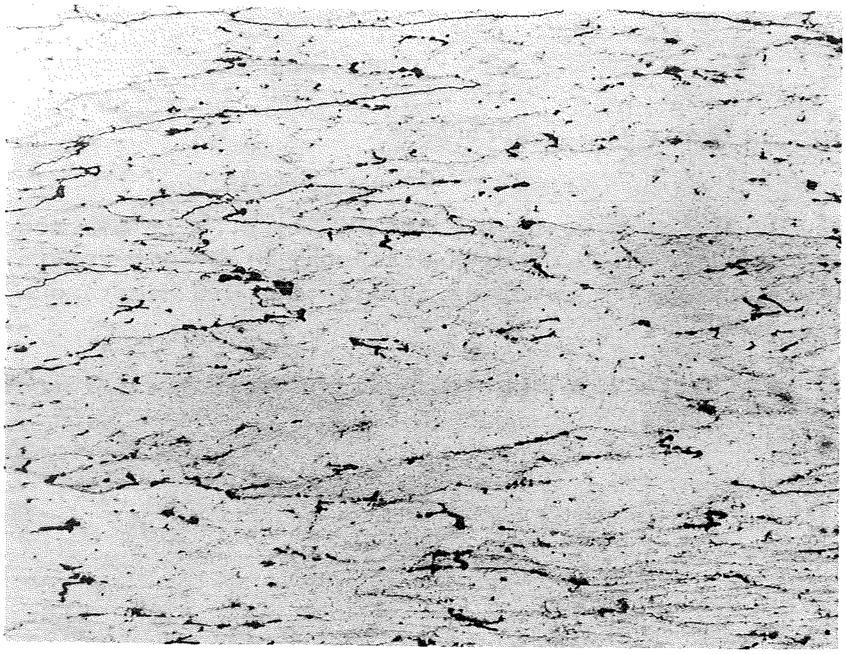
Stainless steel can be made considerably more passive by electropolishing with the TMC solution than by treating with conventional passivating acids. Electropolished surfaces of stainless steel, steel, and brass without subsequent treatment will not accept an electroplate with good adherence, but such surfaces are more resistant to tarnishing or corrosion than mechanically polished surfaces.

The results of this study indicate that the TMC electropolishing process, when carried out according to the recommendations of TMC, is a good general electropolishing process for a fairly large variety of metals and metal alloys and is, therefore, recommended in applications that require electropolishing.

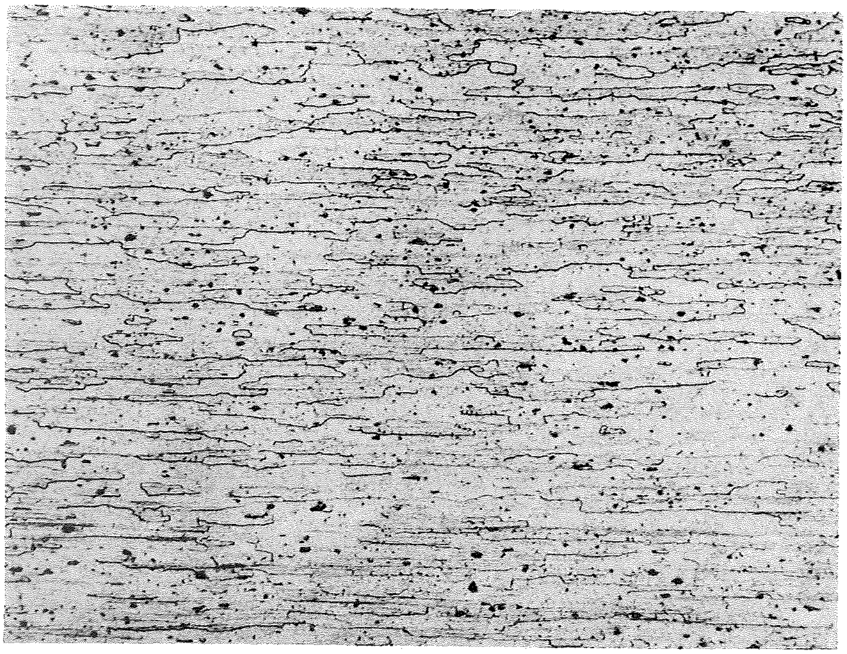
REFERENCES

1. Gurklis, J. A., McGraw, L. D., and Faust, C. L., "Electropolishing and Chemical Polishing of High-Strength, High-Temperature Metals and Alloys," Battelle Memorial Institute, DMIC Memorandum 98, April 12, 1961

2. Cole, J. K., "Electropolish Processing," Technical Micronics Control, Nr: 4001, September 16, 1966

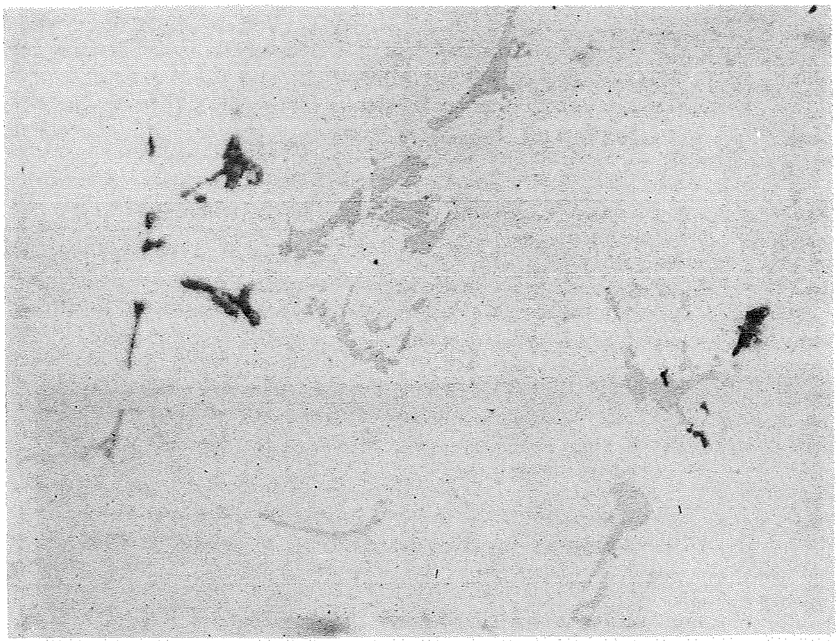


5.0 Inch Thick Plate 100X

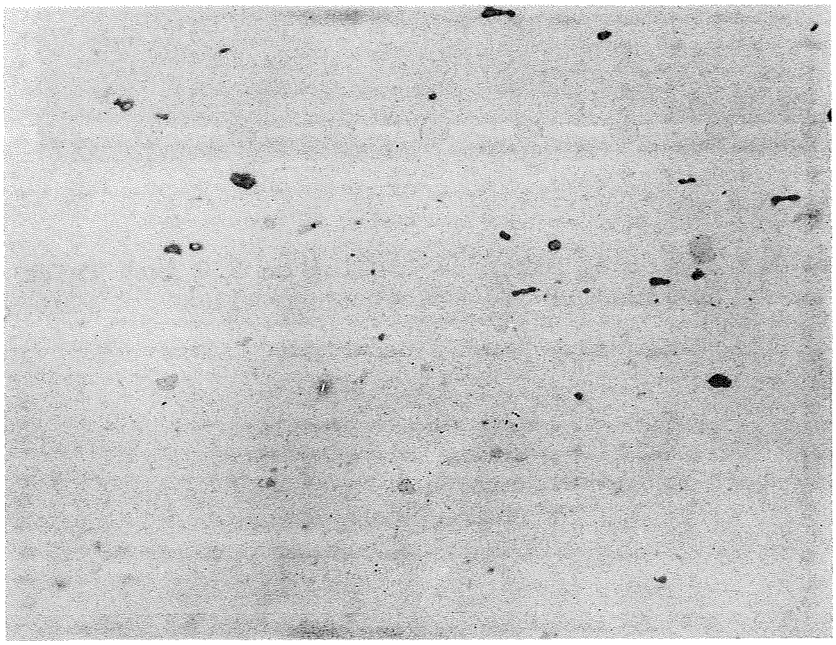


0.125 Inch Thick Plate 100X

Figure 1 - Differences in the Grain Size of 0.125 and 5.0 Inch thick Specimens of 7075-T6 Aluminum Alloy



5.0 Inch Plate 375X



0.125 Inch Sheet 375X

Figure 2 - Difference in the Intermetallic Phases of 0.125 and 5.00 Inch Thick Specimens of 7075-T6 Aluminum Alloy

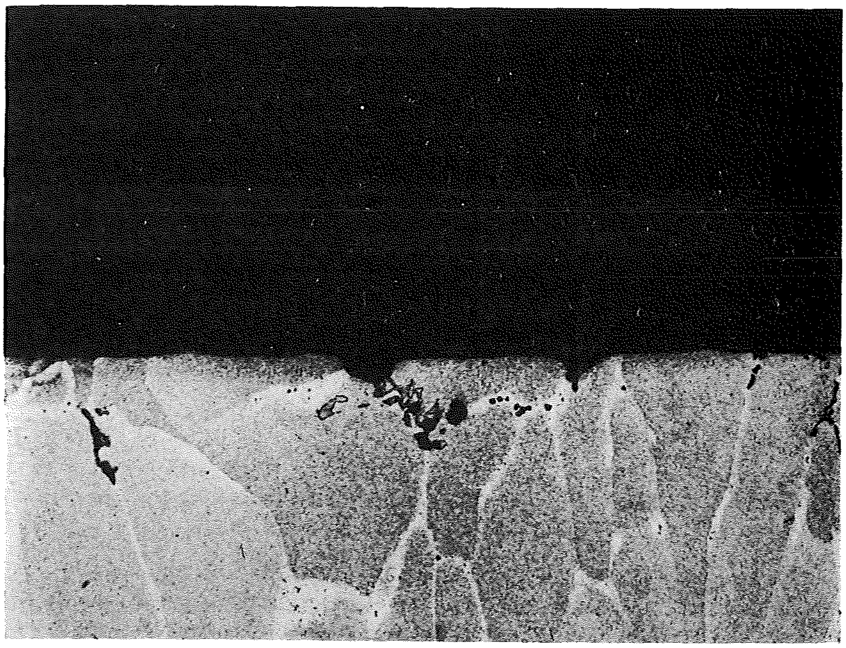
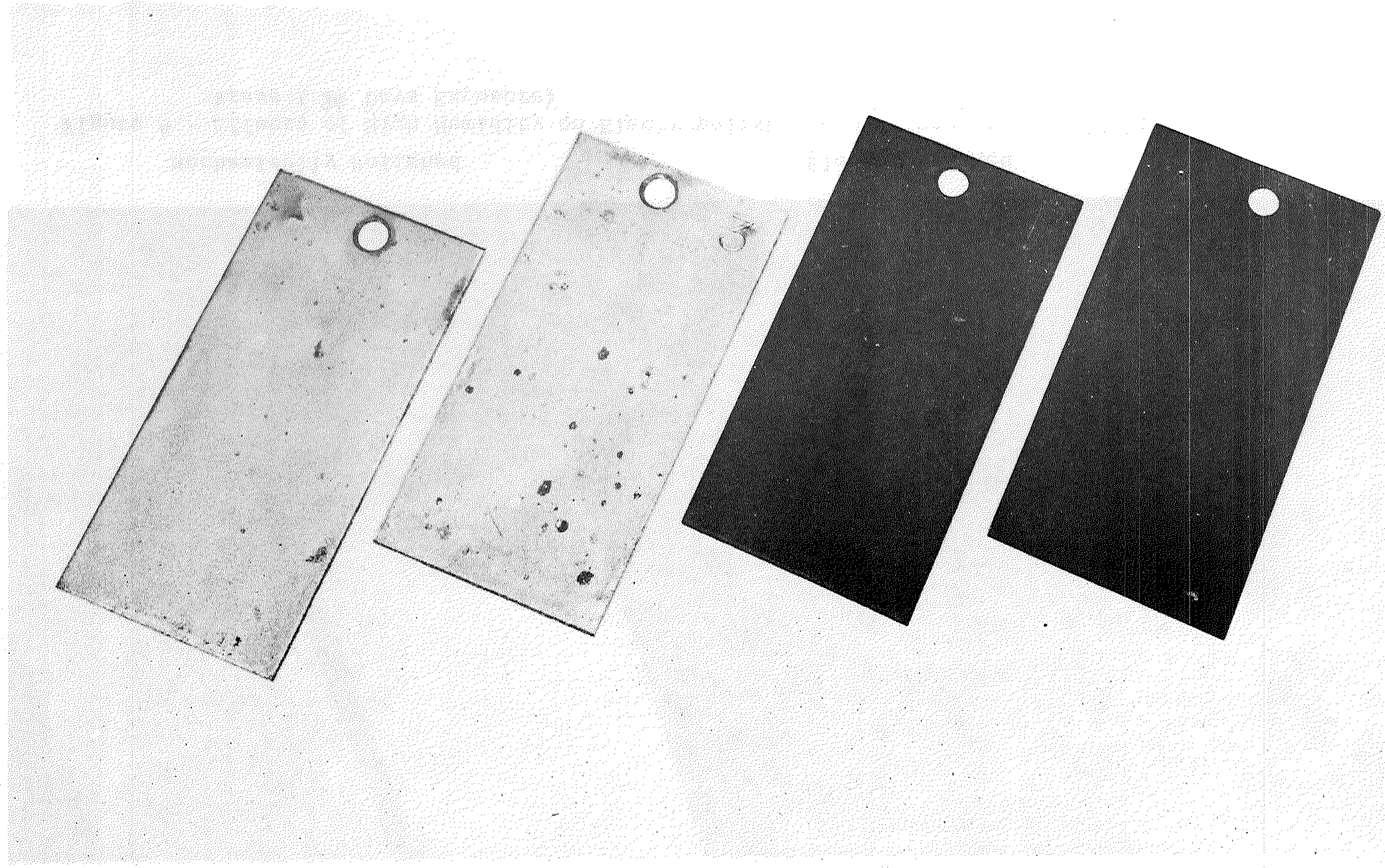
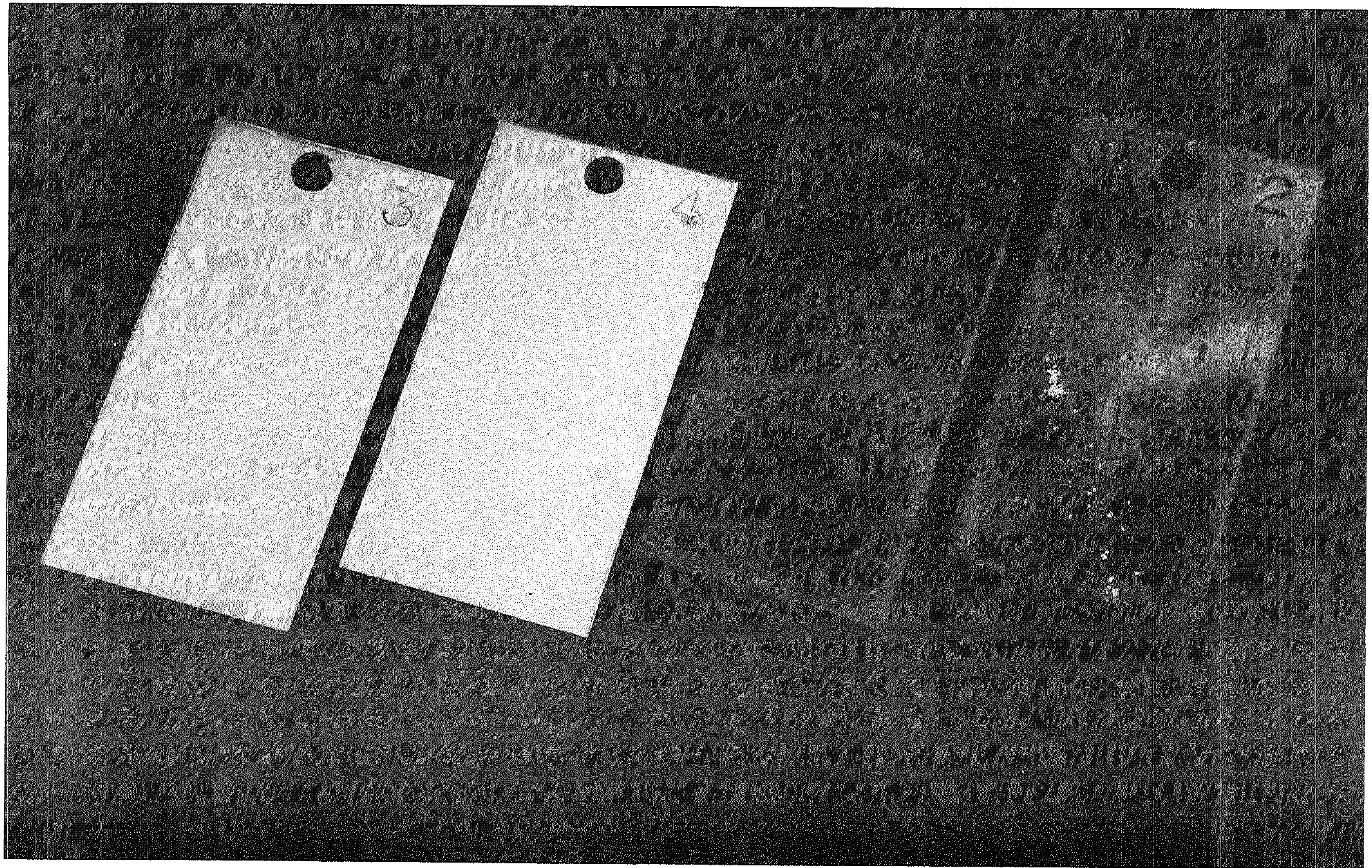


Figure 3 - Effect of Electropolishing on 5.0 Inch Thick Specimen of 7075-T6 Aluminum Alloy 300X



Electropolished                                        Mechanically Polished

Figure 5 - Effects of High Humidity on Electropolished and Mechanically Polished Brass (190 Days Exposure)




Mechanically Polished                                        Electropolished

Figure 4 - Effects of High Humidity on Electropolished and Mechanically Polished Brass ( 34 Days Exposure)

APPROVAL

INVESTIGATION OF THE TECHNICAL MICRONICS CONTROL (TMC) PROCESS FOR ELECTROPOLISHING VARIOUS METALS

By

James R. Lowery

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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