AD-A101 389

STUDY AND DEVELOPMENT OF INFUSIBLE AND INCORRODIBLE IDENTIFICATION TAGS AND MICRODOTS

W. T. Gannon

General Electric Company

Prepared for:

Army Natick Laboratories

September 1974

UNCLASSIFIED
SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER TR 75-27-CEMEL	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) Study and Development of Infusible and Incorrodible Identification Tags and Microdots		5. TYPE OF REPORT & PERIOD COVERED Final 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) W. T. Gannon et al		8. CONTRACT OR GRANT NUMBER(s) DAAG17-72-C-0097
9. PERFORMING ORGANIZATION NAME AND ADDRESS General Electric Company Electronics Laboratory Syracuse, New York 13201		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 1J662713DJ40-39
11. CONTROLLING OFFICE NAME AND ADDRESS Materials Applications Division Clothing, Equipment & Materials Engineering Lab US Army Natick Laboratories Natick, Massachusetts 01760		12. REPORT DATE September 1974 13. NUMBER OF PAGES 44
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Distribution unlimited; approved for public release and sale.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

DEVELOPMENT	LASERS	PULSES	PHOTOMICROGRAPHY
IDENTIFICATION SYSTEMS	LASER TECHNIQUES	SINGLE PULSE	TEMPERATURE
ID TAGS	ETCHING	LASER RECORDING	TENSION
DESIGN	CHEMICAL ETCHING	RECORDING SYSTEMS	MICRODOTS

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The program discussed herein covers the development of a new type of personnel identification tag, conceived by the Army Natick Laboratories, for identifying personnel who have been subjected to cataclysmic events. In particular the tags are to be incorrodible and infusible at 2000°F. The tag is 1" x 2" and contains a facial image, fingerprints, and alphanumeric characters. Both chemical and laser etching techniques have been investigated. For use with the ID tag, a cable type necklace, which will withstand the temperature required but will fail in tension in order to protect the wearer, has been designed. -See reverse-

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

UNCLASSIFIED PRICES SUBJECT TO CHANGE
SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

Continued:

In a parallel part of the program an investigation has been made of microdots, which include the basic features of the tag, but on a structure about 1 mm^2 in area and 0.5 mm thick. Single pulse laser recording, for selective evaporation of a surface deposited on a substrate, is the mechanism investigated.

FOREWORD

Work described in this report was completed in 1972 on a contract with the General Electric Company. Mr. William E. Sollecito acted as business manager of the contract for General Electric. Dr. William J. Barnes, now retired, was NLABS' Project Officer while Dr. Richard N. Macnair was Alternate Project Officer. This report represents the only contractual effort expended to date on identification tags and microdots. It appears that relatively little development effort would be required to produce identification tags whereas more effort would be required to produce microdots.

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

I. INTRODUCTION

A. PROGRAM DESCRIPTION

The program discussed herein covers the development of a new type of personnel identification tag, conceived by the Army Natick Laboratories, for identifying personnel who have been subjected to cataclysmic events. In particular, the tags are to be incorrodible and infusible at 2000°F. The tag is 1" × 2" and contains a facial image, fingerprints, and alphanumeric characters. Both chemical and laser etching techniques have been investigated. For use with the ID tag, a cable type necklace, which will withstand the temperature required but will fail in tension in order to protect the wearer, has been designed.

In a parallel part of the program an investigation has been made of microdots, which include the basic features of the tag, but on a structure about 1 mm^2 in area and 0.5 mm thick. Single pulse laser recording, for selective evaporation of a surface deposited on a substrate, is the mechanism investigated.

The program discussed in this report has been carried out on contract DAAG17-72-C-0097, for the U.S. Army Natick Laboratories. Dr. William J. Barnes, Clothing and Personal Life Support Equipment Laboratory, has been the Project Officer.

B. PRINCIPAL CONTRIBUTORS

General Electric personnel, who have been the principal contributors to the program are listed below, together with their areas of responsibility.

W. T. Gannon	Microdot Development
B. J. Watkins	Identification Tags
J. A. Miskelly	Metallurgy
R. A. Shirk	Polymers
A. H. Hare	Chemical Etching
D. P. Dwyer	Necklace Design
G. E. Ledges	Microdot Materials
A. Martans	ID Tag Format and Mold Design
D. Yoder	ID Tag Laser Etching
M. F. Lowry	Laser Etching Investigation
G. B. Jacobs	Laser Consultation

II. RESULTS AND DISCUSSION

A. IDENTIFICATION TAG

1. Base Material

The requirements for the tag base material are that it should lend itself to chemical or laser etching, should not be frangible, and should be infusible and incorrodible at 2000°F. To satisfy this combination of properties we selected metals; initially, the eleven shown in Table I were investigated.

TABLE I

BASE MATERIALS SURVEYED

Symbol*	Vendor	Alloy Designation
A	Armco	18 SR Stainless Steel
B	Armco	22-13-5
C	-	AISI 446 Stainless Steel
D	-	AISI 309 Stainless Steel
E	International Nickel	Incoloy 840
F	International Nickel	Inconel 601
G	International Nickel	Inconel 600
H	International Nickel	Incoloy 800
I	International Nickel	Nickel 200
J	Kawecki Berylco	Nickel 440
-	-	Anodized Aluminum Coated Steel

* Symbol refers to marking on test samples.

The eleven samples, except for anodized aluminum, were chemically etched with fingerprints on one side and a facial photograph on the other. Each sample was subjected to a temperature of 1994°F (± 1%) for 30 min. in air.

Preliminary baking of the anodized aluminum-coated steel showed fissuring in the anodized coating, probably due to expansion differences between the coating and the base material. The remaining metals showed effects ranging from some oxidation to significant scaling. In many of these the finger prints were legible even with some scaling. However, the three best materials in these tests were the nickel 200, the nickel 440 and the 18-SR. The first two behaved similarly, in that a very thin oxide film developed with virtually no scaling. This film was grey-green in the case of the nickel 200, and light brown on the nickel 440. The 18-SR is a stainless steel alloy developed to resist scaling in forthcoming automotive exhaust reactors. In this application it showed slight scaling, but the deep etched information was still very legible, though discolored. The properties of these three alloys are shown in Table II.

Tests on Ni 200 samples for exposure times of 3, 5, 15 and 30 minutes showed no discernible differences in the oxide film. Thus, virtually all of the oxidation that takes place does so within a few minutes of exposure.

The Ni and NiBe materials behave similarly with regard to oxidation resistance, the latter offering some high temperature springiness in exchange for its higher price. Because of the slight amount of scaling, the 18-SR was considered less desirable than either of the other two metals. For these reasons the nickel 200 is the recommended base material. The tag blank specification is shown in Figure 1.

2. Chemical Etching

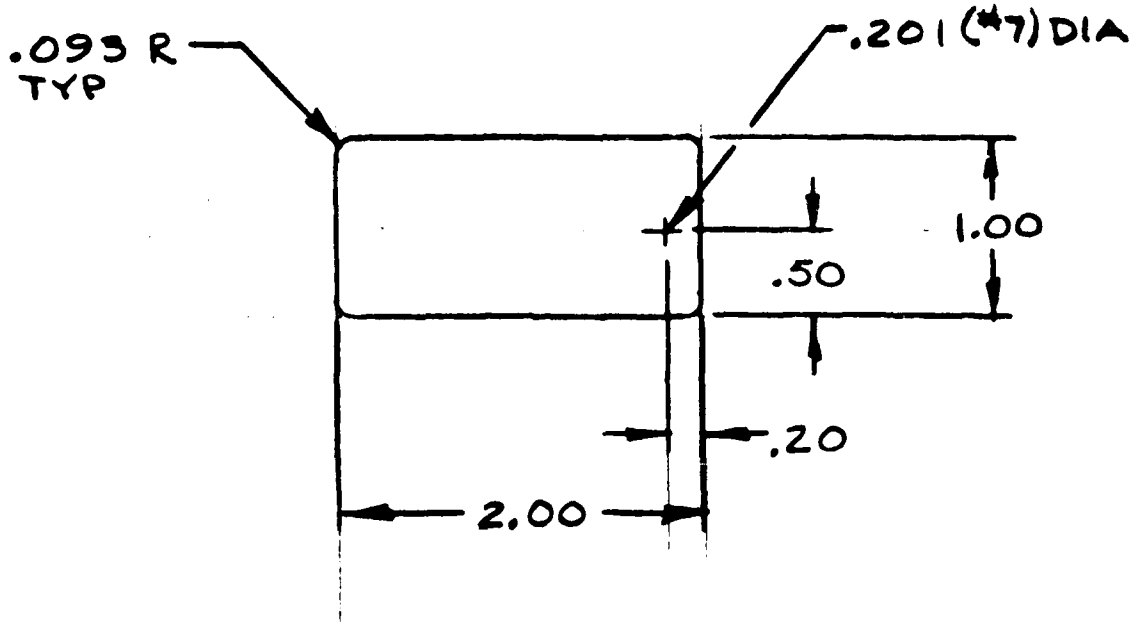
For the initial high temperature tests a ferric chloride etchant was used for all samples. Once the three primary base metals were selected the following etchants were investigated: nitric acid, a nitric-acetic combination, and a sulfuric acid-chromic acid combination. None of these gave the same definition as the original etchant. The ferric chloride etching procedure is described in detail below.

Clean	Degrease in trichloroethylene vapor degreaser. Immerse in Photo-Chemical & Equipment Company CD-3 cleaner for 2 minutes. Rinse in cold water spray for 2 minutes, running water rinse for 5 minutes, then cold water spray rinse for 1 minute. Air blast dry.
Coat	Flow coat with Hunt's IC Photo Resist cut 1:1 with Hunt's IC thinner. Spin at 120 RPM in whirler for 5 minutes (3 minutes at room temperature then 2 minutes under infra-red lamps). Stabilize at room temperature for 1/2 hour. Bake in oven for 1/2 hour at 100°C. Cool to room temperature.
Expose	Thirty seconds in a Millington double sided printer model #VF-E2M is used to harden resist.

TABLE II

PROPERTIES OF PRIMARY BASE METALS

Alloy	Nickel 200	Nickel 440	18-SR
Type	Commercially Pure Nickel	Hardenable Nickel - Beryllium Alloy	Ferritic Stainless Steel
Composition	Ni+Co - 99.0% min. Balance Impurities: Fe, Mn, Si, Cu, C, S	Ni - Balance Be - 1.95% Ti - 0.50%	Fe - Balance Cr - 18% Ni - 0.5% Al - 2% Ti - 0.4%
Mechanical Properties Annealed Tensile Stress Yield Stress Elongation Heat Treated Tensile Stress Yield Stress Elongation	 55,000 psi min. 15,000 psi min. 40% - - -	 105,000 psi typ. 45,000 psi typ. 40% 215,000 psi min. 150,000 psi min. 15%	 80-90,000 psi 60-70,000 psi 25-30% - - -
Melting Point	2615-2635°F		
Temperature Resistance	To 2000°F without detriment. Attacked in sulfur bearing atmosphere at elevated temperature. Resistant to scaling beyond 2000°F.	Resistant to scaling beyond 2000°F.	Resistant to gross scaling beyond 2200°F
Available Forms Include Sheet and Strip	Yes	Yes	Yes



NOTES:

1. MATERIAL- NICKEL (INCO 200), .010 THICK, GRAIN SIZE- ASTM E112, 9-9.5

Figure 1. Blank, Identification Tag

Develop	Spray for 20 seconds using a Spray-on #50-P jet pak unit at 8 inches from plate. Rinse in cold water spray for 1 minute. Air blast dry. Bake in oven for 1 hour at 110°C then cool to room temperature.
Etch	Spray etch for 2 minutes at 130°F in Chemcut Model #800 machine using Hunt's Hi-Speed Circuit Etchant. Rinse in cold running water for 15 minutes then dry.
Strip	Remove photo-resist by immersion in Hunt's Microstrip at 140°F for 1 minute. Pinse in cold water spray for 1 minute, then fifteen minutes in cold running water. Air blast dry.

The 18-SR metal shows a somewhat better etching capability than the Ni 200 and NiBe 440 alloys. In some of the Ni 200 samples, the etching produced a frosted surface with excessive "sparkle". It was determined that these samples had an excessively large grain size. Thus it is necessary to specify the grain size of the base metal for successful etching. This specification is 0.015 mm, or according to ASTM E112, Grain Size 9-9.5.

3. Laser Etching

a. The Laser Etching Process

Images with grey scale are recorded in the base metal with a half-tone technique, the grey scale being a measure of the "lightness" or "darkness" of any point on the image. For both the laser and the chemical etching procedures, the half tone image is recorded by an array of holes of varying size. In Figure 2 light ray A is incident on the highly polished

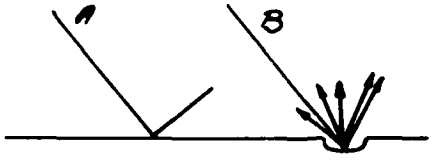


Figure 2. Geometric Representation of Light Reflection

surface of the tag, and is specularly reflected to the observer. On the other hand, ray B, which is incident on an etched spot, is diffusely reflected so that only a small fraction of the incident energy reaches the observer. Thus the fraction of the area that is etched determines the loss of reflected light and, hence, the darkness of the image. Modulation – the variation of this proportion of incident light reflected – is realized by either a) changing the size of

the etched spot, the spots being on a regular spacing, or b) changing the density of spots, the spots being of a uniform size.

The phenomena involved in the absorption of laser energy on a material surface are complex, and do not lend themselves to accurate prediction in the most general case. In Figure 3 the interrelations of the phenomena are shown schematically. Chun and Rose [1] measured some of the energy flow-paths in material removal. For nickel the initial high reflectivity of 70% drops to about 25% after 100 μs of illumination at a power density of 10^7 W/cm^2. Excess kinetic energy losses should be of the order of 0.001% (for normal pulse etching), the re-radiation loss is less than 0.5% and the thermal diffusion loss is about 4%.

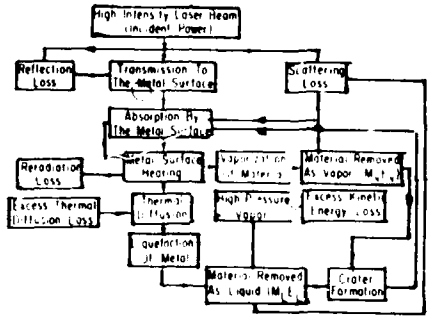


Figure 3. Block Diagram of Material Removal Process [1]

In spite of the analytical difficulties in predicting hole size versus the energy of incident radiation, a rough estimate can be made as follows. From theoretical and experimental data presented in the recent literature [1-11], a power density of 10^7-18^8 W/cm^2 is required. The vaporization time should be of the order of 0.1 to 1 ms for efficient removal. The hole size should be 0.003 to 0.010 in. (0.0085 to 0.025 cm). Thus the energy required per hole is in the range of 0.65-6.5 joules. It is quite apparent that an unexpected efficiency of laser machining is available for a certain range of pulse length, as an increasing fraction of the material removed is blown out of the hole in liquid form. Chun and Rose [1] measured values of about 50% at 100 μs, and nearly 90% at 400 μs for a 30 kW pulse on Ni. This is explained by the effect of evaporative cooling at the surface, with the result that vapor forms beneath the surface. This subject has been treated theoretically by Dabby and Paek [5].

For the present application a laser must have a high pulse rate and high power output for rapid processing. Two types of commercially available lasers have been employed for similar industrial material removal applications; these are the neodymium-doped yttrium aluminum garnet (Nd:YAG) laser and the carbon dioxide laser (CO_2) laser. A summary of their characteristics is presented in Table III. [8, 9, 10, 11]

TABLE III

	Nd:YAG	CO_2
Pulse Rate (sec^{-1})	1000-10000	~ 100
Pulse Length (μsec)	0.1 - 0.2	150
Energy/pulse (J)	0.5×10^{-3}	7.5
Peak Power (kW)	2.5 (at 0.2 μsec) 5.0 (at 0.1 μsec)	50
Avg. Power (W)	0.5 - 5	7500
Wavelength (μm)	1.06	10.6

Obviously, for the tag etching process, a high pulse rate is desirable, and the high rate of the YAG laser does imply a very short etching cycle (the facial image of the tag has about 10,000 picture elements, so that the whole tag has at most 20,000 elements). However, there are data available indicating that there is an optimum pulse length for material removal. This value must be determined experimentally, but it is probably of the order of 100 μsec. For a given energy, very long pulses (on the order of ms) allow much of the energy to be lost by conduction. Under these conditions, the energy penetrates deeply, with the result that there is often a deep fusion but no vaporization of the metal. This, of course, is an ideal situation for welding. On the other hand very short pulses may allow only a small depth of metal to be heated although the peak power is very high. An example, calculated for tungsten, is shown in Figure 4. A further complication is that, at very short pulse lengths, the energy is less efficiently coupled into the metal.

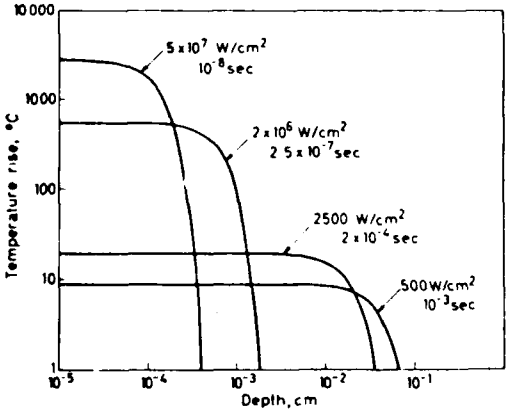


Figure 4. Comparison of Temperature Profiles in Tungsten for Q-switched Laser Pulses and Normal Laser Pulses. The temperatures calculated for flat, spatially uniform laser pulses representative of what is available from present day lasers are plotted as a function of depth. Each pulse delivers the same total energy per unit area.[7]

b. Sample Tags

To create the half-tone laser etched image on the metal tag it is necessary to convert the input data to a form that will modulate the laser output pulses. Existing facilities, however, have no capability for reading this data and performing the conversion. To accomplish this task manually would be prohibitively expensive. Thus, it was concluded that the line drawing simulations of the facial image (Figure 5) and the fingerprint would be recorded. (The characters are drawn calligraphically.) These could be done in an existing GE facility located in Utica, New York. The line segment representations were programmed by hand, while the alphanumeric characters were done automatically. The equipment consists of a Nd:YAG Q-switched laser. The recording was done at a repetition rate of 800 pulses per second with a pulse energy of ~0.1 joules. The target material is carried on a numerically controlled XY table. Although this table is capable of a minimum step size of 0.00025 in. (6.35 μm), a minimum step of 0.004 inch was used for the tags because of the hand programming cost.

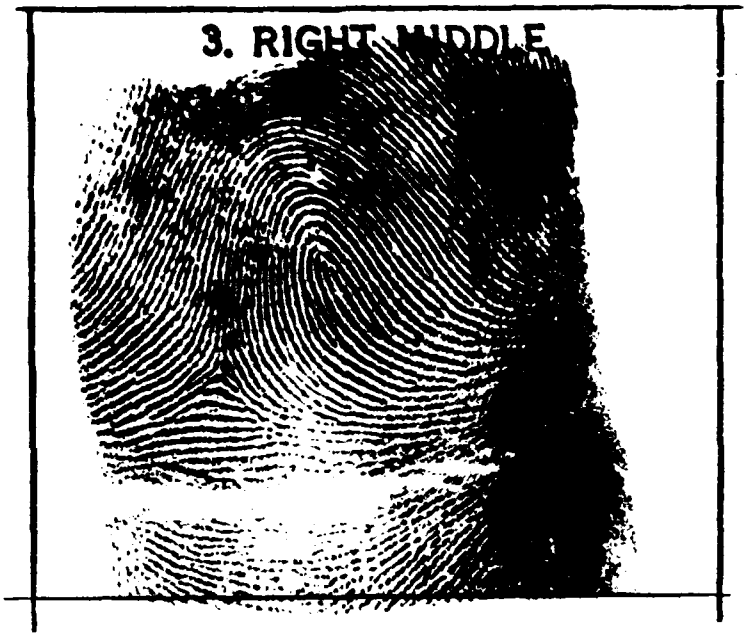
Figure 5. Artist's Line Drawing for Laser Tag

4. Comparison of Chemical and Laser Etching

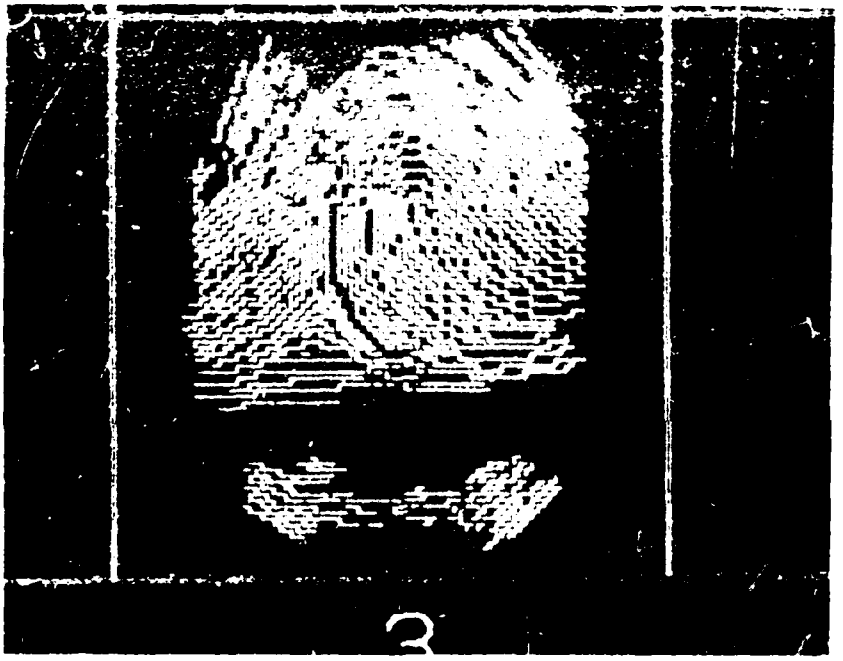
A number of ID tags have been made using both the chemical and laser etching techniques. Those made by the first method were etched in both a "positive" and a "negative" configuration; that is, the "positive" tag shows a positive image for most angles of viewing and illumination. With either, it is possible to reverse the image by viewing the tag from a particular angle. Two tags are shown in Figure 6, one with a negative back, the other with a positive front. When etched solely as line segments, as discussed above, the laser etched tag appears as a positive for all viewing angles.

Figure 6. Chemically Etched ID Tags

Figure 7 a, b, c and d are photographs of several fingerprints. Figure 7 a is taken from the fingerprint card, 7 b is the simulated fingerprint on the laser etched tag, 7 c is the negative chemically etched tag, and 7 d is the positive chemically etched tag (the images appear reversed in 6, 7b, 7 c and 7 d. because of the angle of illumination on the specularly reflecting surfaces).

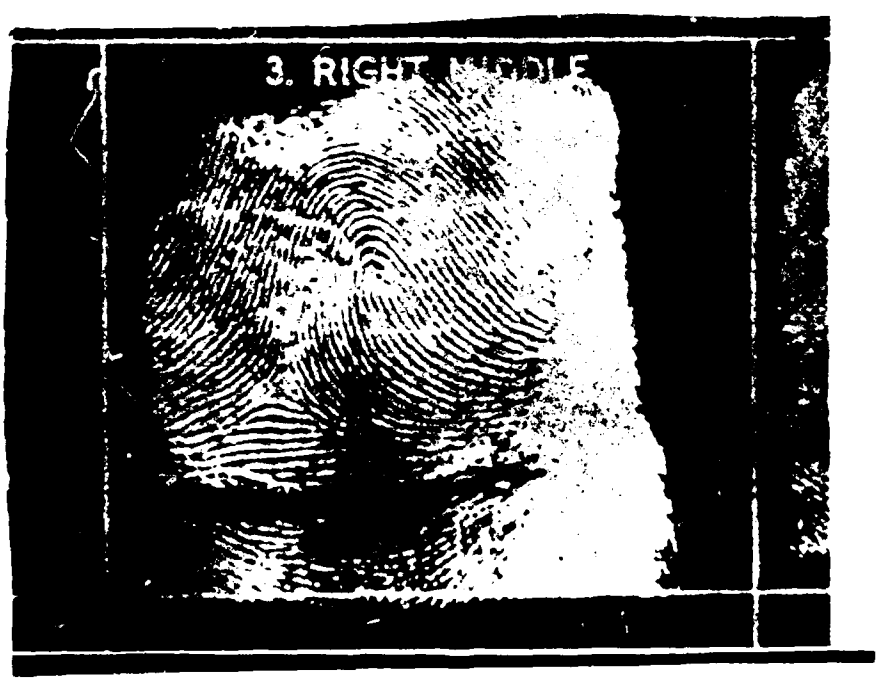


a. Fingerprint Card



b. Simulated Fingerprint

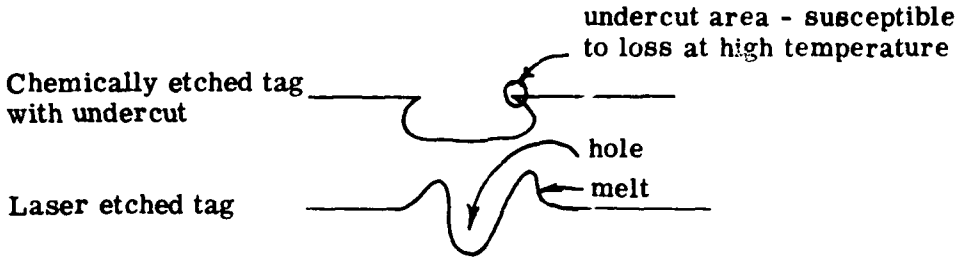
c. Negative Chemically Etched Tag



d. Positive Chemically Etched Tag

Figure 7. Fingerprint Comparison - concluded

In the high temperature tests 1994°F (±1%) for 5 minutes the nickel tags show buildup of an oxide film and loss of contrast. This contrast in the unoxidized tags is due to the difference in the specularly and diffusely reflecting surfaces; once oxidized, the entire surface reflects diffusely. However, under microscope examination, with the illumination incident at a low angle to the surface, the patterns are quite evident. In the case of the chemically etched tags, a compromise must be reached. If the etch is carried too deeply, an undercut of the walls occurs, as shown, leaving a sharp edge that can be



eroded at the high temperature with loss of information. If the etch is too shallow, the information disappears in the nonuniformities of the oxidized surface.

In the laser etched tag the line or spot cross section (above) shows the blow-out of the liquid from the etching process. When oxidized at the elevated temperature the greater surface/volume ratio of this melt allows it to oxidize to a greater extent and, since this oxidation is a volume-increasing process, the lines are somewhat accentuated by the oxidation.

Table IV presents several characteristics of each etching technique. As discussed above the optimum depth of etch in the chemical approach is a compromise. For the laser this is quite variable, since a laser can even be used for deep piercing of holes. The limitation is that of energy available - repetitive pulsing would probably be necessary for any depths greater than about 0.003". Fabrication time estimates for the chemical tags are based on the procedures described, and show reduced values for many of the step times. Those for the laser technique assume two lasers and are conservative estimates based on commercially available lasers.

The present samples of the chemically etched tags show the information in detail and with prominence, more so for the positive than the negative tags. Because of practical limitations of the available laser etching facility, the line image and simulated fingerprint are not as prominent on the laser tag. A future laser tag, however, should have the same appearance as the present chemical tag when etched with half-tone images. It appears that the laser tag has an advantage in temperature resistance, because of the greater etch depths possible.* The categories of ease of automation, logistics support and other requirements pertain to the fabrication equipment rather than to the tags themselves. In these areas the laser technique has strong advantages.

---

* In Appendix C, a photomicrograph of an oxidized, laser-etched line is shown.

TABLE IV.

COMPARISON OF ETCHING TECHNIQUES

Parameter	Chemical Etching	Laser Etching
Resolution	——— Either capable of the 5000 - 10,000 picture elements ———	
Optimum Depth of Etch	0.002"-0.004"	~0.005" - depends on energy available
Contrast (modulation)	0.003"-0.010" OK	OK - limits choice of lasers
Fabrication Time (Etching only)	20-72 min	4-8 min
Fabrication Rate (Tags/hr)	30-60	8-15
Ease of automation	Must control temp, pH, etc. of solutions, various timings, sprays, etc. — more costly to automate	Lends itself quite readily. Equipment not unduly complex.
Logistics support	Preprocessed tags Film Developer Etchant Stripper	Tag blanks CO_2, N_2, and Ar (if CO_2 laser) Periodic flash lamp (if Nd:YAG)
	——— (Adhesive and plastic) ———	
Other requirements	Proper disposal of spent chemicals. Cooling water Handling of chemicals	Cooling water Handling of gases (not toxic, or explosive)
Development time	Basic technology well documented Effort required to - minimize times - automate	Basic technology in rapid stage of growth. Effort required to - Select laser: CO_2 or YAG - set parameters - automate

5. Plastic Coating

The plastic coating on the tags offers corrosion protection and reduces noise. It must be clear, compatible with the skin, resistant to chemical and environmental attack, and lend itself to lamination or molding. The following materials were considered:

1) Acrylonitrile - Butadiene - Styrene
2) Cellulose Acetate Butyrate
3) Cellulose Propionate
4) Polycarbonate
5) Polypropylene
6) Polysulfone
7) Polyterephthalate
8) Polyvinyl Chloride

The various materials that were considered for this program are listed and discussed in the following paragraphs.

1) ABS - Marbon Chemical's CIT Polymer

   This material has excellent toughness, good clarity, UV resistance, and processes well. Unfortunately, when the material is stressed, it turns opaque white. For this reason, it was placed in a secondary category of interest.

2) Cellulose Acetate Butyrate - Eastman Chemical

   The consideration of this material was terminated early because of its odor, an integral property of the material.

3) Cellulose Propionate - Eastman Chemical # 319A - 48620-H2

   This material has the same properties as CAB, but it does not have the associated odor.

4) Polycarbonate - General Electric Lexan #9400

   This material is particularly sensitive to stress concentration and for this reason was eliminated from the study early. The material processed well and had superior toughness but long times under molded-in stresses did not augur well for Lexan.

5) Polypropylene - Eastman Chemical #423DF-X-11111-123B

   This material processed very well. Unfortunately, no adhesive was found that would adequately bond the metal to the film. Because of this fact, further work with polypropylene is not being considered until an adhesive is found.

6) Polysulfone - Union Carbide P-3500

This material was not readily available in film stock and because the material possesses a yellow-amber color, further work on this product was halted.

7) Tenite Polyterephthalate - Eastman Chemical #7DROF
- X-12023-73C

This product appeared to be one of the more promising early candidates. Unfortunately, initial trials showed an adhesion to all metals such that teflon coated molds would have been required or, at least, the use of mold releases. Since some bubbling occurred during lamination and other materials processed easier, this product was also discarded.

8) Polyvinyl Chloride - Local Supplier

This material can be formulated so that all property requirements are met. The basic reason CP was chosen over PVC was the presence of plasticizer in the PVC. If PVC is made tough enough to resist breakage, the plasticizer will migrate from the film and give a cracked yellow appearance after aging. This material, or one of its many formulations, could be a satisfactory answer to our laminating material problem. On the other hand, if moisture can be eliminated or controlled, the CP is equal to and better than PVC.

The properties of these materials are tabulated in Appendix A. It was determined that the best combination of properties was exhibited by cellulose propionate, although a different formulation of PVC might fill the qualifications. A characteristic of this material is its tendency to absorb moisture. This is a problem only in the molding step and can be eliminated by drying the sheet as described below.

As a measure of the possible effects of plastic decomposition products on the base metal, tags coated with ABS, PVC, and polypropylene were subjected to a temperature of 2000°F. In a few seconds the plastic was completely destroyed leaving only carbon powder. This was easily brushed off and the resultant oxidized metal surfaces were no different from those without a coating. It is significant that no residue is left adhered to the base metal.

a. Adhesive Evaluation

All of the liquid materials listed in Table V were applied to the clean metallic surface, dried, and reactivated at 325-350°F during the laminating process. All of the samples were then allowed to cool to room temperature before examination.

All of the adhesive primers bonded well to the metal substrate. None of the materials tested showed satisfactory adhesion to polypropylene. Only a few gave adequate adhesion to polyvinyl chloride and cellulose propionate. The best adhesive tested was Staley Chemical's #U-8795. This product performed better after being diluted with distilled water. Eastman Chemical

TABLE V

ADHESIVES EVALUATED

Material	Supplier	Adhesion				Application		Rating	
		Metal	PVC	PP	CP	Coating	Drying	Application	Adhesion
SR-529	General Electric	E	P	P	P	G	S	F	P
76G5	Morton	E	G	P	G	G	S	F	F
PZ4333.09	Dow Chemical	E	P	P	P	E	E	E	P
504	Morton	E	E	P	G	G	P*	G	G
U8795	Staley Chemical	E	E	P	E	G	S	G	VG
P-961	Staley Chemical	G	P	P	G	G	S	G	F
P-528-F	Staley Chemical	E	G	P	F	G	S	G	F
XPD-375	Picco	G	F	P	F	G	S	G	F
XPD-374	Picco	G	F	P	F	G	S	G	F
Z-1	Picco/Shell	G	F	P	F	G	S	G	F
Z-2	Picco/Shell	G	F	P	F	G	S	G	F
Z-3	Picco/Shell	G	F	P	F	G	S	G	F
#1112	General Mills	G	P	P	P	G	G	G	P
Versalon 1175	General Mills	E	P	P	P	G	G	G	P
50% U8795	Staley Chemical	E	E	P	E	E	E	VG	E

* During processing, yellowing occurred.

Z-1 = 5% Piccotex 75 & 5% Krator 1102
in a solvent containing 3 parts N-Hexane
1 part Toluene
1 part Acetone

Z-2 = 5% Piccotex LC & 5% Krator 1102
in the solvent above.

Z-3 = 5% Piccotex S-125 + 5% Krator 1102
in the solvent above.

Company has recently recommended Staley's #AF-725 as an alternative. Greater dilution may be an aid in the application in an automated process.

The degree of adhesion obtained using U-8795 (50% dilution) and cellulose propionate is such that the laminate must be placed in a solvent in order to recover the metal tag from the lamination.

b. Tag Lamination Process

Following is a description of the process that has been developed for laminating the base metal to the cellulose propionate coating.

Cleaning of Metal Tag

a. The metal can be washed with detergent and water, thus providing a wettable surface for the adhesive.

b. An alternative approach, which appears to be more practical for use in a continuous production situation, is to use 1,1,1, trichloroethane as the degreasing solvent. This solvent also provides a wettable surface.

Note: In either case, a degree of cleanliness of the metal will be required in order to reduce the cleaning effort.

Adhesive Application

The adhesive may be applied by brush, roller-coating, dipping, or spraying. The only requirement is that a thin, reasonably uniform coating be applied. The (50%) U-8795 was dipped with good results. This coating may be air-dried or force-dried in an oven or by infra-red lamps.

Preparation of Plastic

Although the experimental samples have been cut from sheet using a shear/paper cutter, samples processed for an engineering model would be cut to size using a steel rule die. These parts must then be placed in a desiccator to remove moisture.

This problem is more prevalent during the higher humidity periods in summer. If moisture is not reduced, bubbles will be formed during the lamination step. Four hours in a good desiccator is enough to remove any excess moisture. Alternatively the uncut sheet would be stored in a dry atmosphere in the engineering model.

Lamination

The early samples were prepared using a plate (0.060" thick) with a cavity of the appropriate size and a flat plate on each side. This produced parts that were quite satisfactory for adhesive studies but, often, bubbles were formed in corners. The requirement that pressure be developed on the plastic necessitated that a mold be designed; the mold is described in SK-56157-D161-12. The final parts have all been made using this mold and show no bubbles whatever.

The process is as follows:

a. Place 0.030" of plastic on either side of the metal tag and set this sandwich into the mold.

b. Apply contact pressure ($<$ 100 lb. force) to the mold and heat mold to a temperature of 330 $\pm$ 10°F.

c. When mold and plastic reach this temperature apply 1000 $\pm$ 200 lbs. force to the mold and cool immediately.

d. Remove the parts from mold and trim.

The machine used is a Preco Hydraulic Press #PA-7 with thermostatically controlled platens. In addition, there are cooling coils in the platens for reducing the cycle time. On this machine, the heating cycle is 15-20 minutes and the cooling cycle is 5 minutes. It is anticipated that a 3-4 station rotary laminating press could cut the process time to 5-6 minutes.

6. Necklace

A method for attaching the ID tag around the neck has been developed; the method is compatible with the requirements of high temperature resistance and personnel safety. The necklace method, proposed as a simple substitute for the existing beaded chain, consists of a flexible stainless steel wire rope attached at one point by a mating stainless steel clasp. Wire rope is used because it is readily available in a variety of high temperature materials, can be plastic coated, is strong, and is easily and securely attached to end fittings. The clasp proposed here provides a simple method of attachment both to the cable and to itself, and can be designed to release at a predetermined force.

a. Material

The wire rope can be any of several materials that meet the temperature requirements; the one proposed, and provided as samples, is nylon coated stainless stell. The nylon coating is probably not the best plastic for this application (polyurethane is recommended) but nylon and vinyl are the only coating materials that are commonly available from wire rope manufacturers. However, these manufacturers point out that other materials, such as polyurethane, can be economically coated on the wire rope assuming a large

enough quantity. The rope is available in a range of diameters and stranding, both of which influence the strength and flexibility. The samples provided are 3/64 inch diameter cable, of 7 × 7 strands, or 7 different ropes wound together of 7 wires each forming one cable. The nylon coating yields a final diameter of, approximately, 1/16". The rated pull strength of such cable is 270 pounds although the actual strength, on the average, would be considerably greater.

This rope size was selected because it provides a strength much greater than required at the attachment point, yet it is small enough so that it can be easily attached to end fittings of reasonable size and weight. It is also a standard size. However, this particular stranding of wire rope may be a little too stiff for wearer comfort. To increase the flexibility of the necklace a finer strand can be used. For example, a 7 × 19 stranding is a standard product and is substantially more limber. However, it is not normally stocked by cable vendors with plastic coating, although it can be easily obtained on special order. Since the nylon coating provides an inert noiseless surface that does not irritate the skin, the samples were made with the available but stiffer material. A disadvantage of the 7 × 19 stranding, is its greater surface area and, subsequently, greater scaling at the elevated temperatures.

b. Attachment Method

In addition to the temperature and corrosion resistance of the stainless steel rope, another advantage of this material is the ease with which it can be joined to a clasp or attachment device. Many standard fittings are available for wire rope but it was felt that the simplest method of securing the wire to an attachment device would be to crimp the cable into a tubular ferrule, as is commonly done with stranded copper wire in the electrical and electronics industry. This procedure permits use of a small diameter fitting that is simple to manufacture and assemble with standard tools. The strength of such a crimp joint is far in excess of that required and in all tests performed to date no failure has been found with any crimp attachment.

With this method, the ferrules are first crimped to each end of the wire; then they are mated after the tags are slid over one ferrule onto the wire. The way in which the ferrules are mated is, then, the principle design problem. The following criteria have been assumed as requirements for the mating device, hereafter called the clasp.

1) The method of assembly should be simple, requiring only simple tools, if any. The clasp material must be corrosion resistant (at ordinary temperatures) and meet the high temperature requirements.

2) A predetermined force on the chain at any point must separate the clasp. This force should be less than that required to strangle the wearer.

3) The clasp should be coated for skin protection and to eliminate noise.

4. The clasp should be capable of being reassembled if pulled apart.

The proposed clasp details, shown in Figure 8 *, appear to meet these requirements. Both parts are stainless steel. A male ferrule with a slightly projecting pin is forced into the female until the detent position is reached. The amount of interference between the pin and the female part determines the insertion force, which is very similar to the withdrawal force. Once engaged, a short length of heat shrinkable plastic tubing is slid over the joint. A heat gun or any similar device can then be used to form the plastic around the clasp.

The axial force required to insert and withdraw the clasp attachment can be predetermined by varying the interference between the pin and the female part. It can be calculated quite closely by assuming that the portion of the female part beyond the detent towards the crimped end is a cantilever beam with a certain deflection defined by the interference. A limit is soon reached in the amount of interference allowable, however, since overstressing produces a permanent set in the female legs. To allow as large a range of force as possible with a small diameter part (1/8" maximum) the material selected is type 405 stainless, which can be heat-treated to provide a yield strength of over 200,000 psi.

It was at first assumed that a release force only somewhat less than the weight of the wearer would be the appropriate safety provision. However, it was found that a much smaller separation or insertion force, of the order of 5 pounds, must be used to prevent strangling under certain conditions. One condition occurs when the clasp is being pressed against the throat and the rope pulled from behind. Now the force required to separate the clasp is substantially greater than that used to pull apart the same clasp under a straight axial pull. Since the plastic jacket also adds to the required separation force, the breakaway force of the metal clasp alone must be kept fairly low. This is an advantage since the insertion force is very similar to the separation force and, therefore, no special tool is needed at either the initial assembly or any subsequent reassembly. The parts are simply pushed together by hand.

A considerable number of tests have been made to determine the forces required to separate the clasp under a variety of conditions. The first tests were simple axial tests made on the metal clasp only, using a standard laboratory tensile test machine to find the force. Next the heat shrinkable plastic sleeving was added, and finally tests were made on a mockup simulating a human neck.

This latter condition, where the force was applied on the side of the neck opposite the clasp location gave the highest forces for a given set of dimensions for the parts. This condition probably represents the most dangerous of those likely to be experienced by the wearer. The clasp has the tendency to sink into the soft part of the throat. Although the exact force required to cause strangulation under these conditions is not known, it probably does not exceed 30 pounds, depending on the individual. Therefore this value was assumed to be the maximum that should be allowed. This, then, restricted

---

* GE specification P4A-GP2, listed on the drawing in Figure 8 is found in Appendix B.

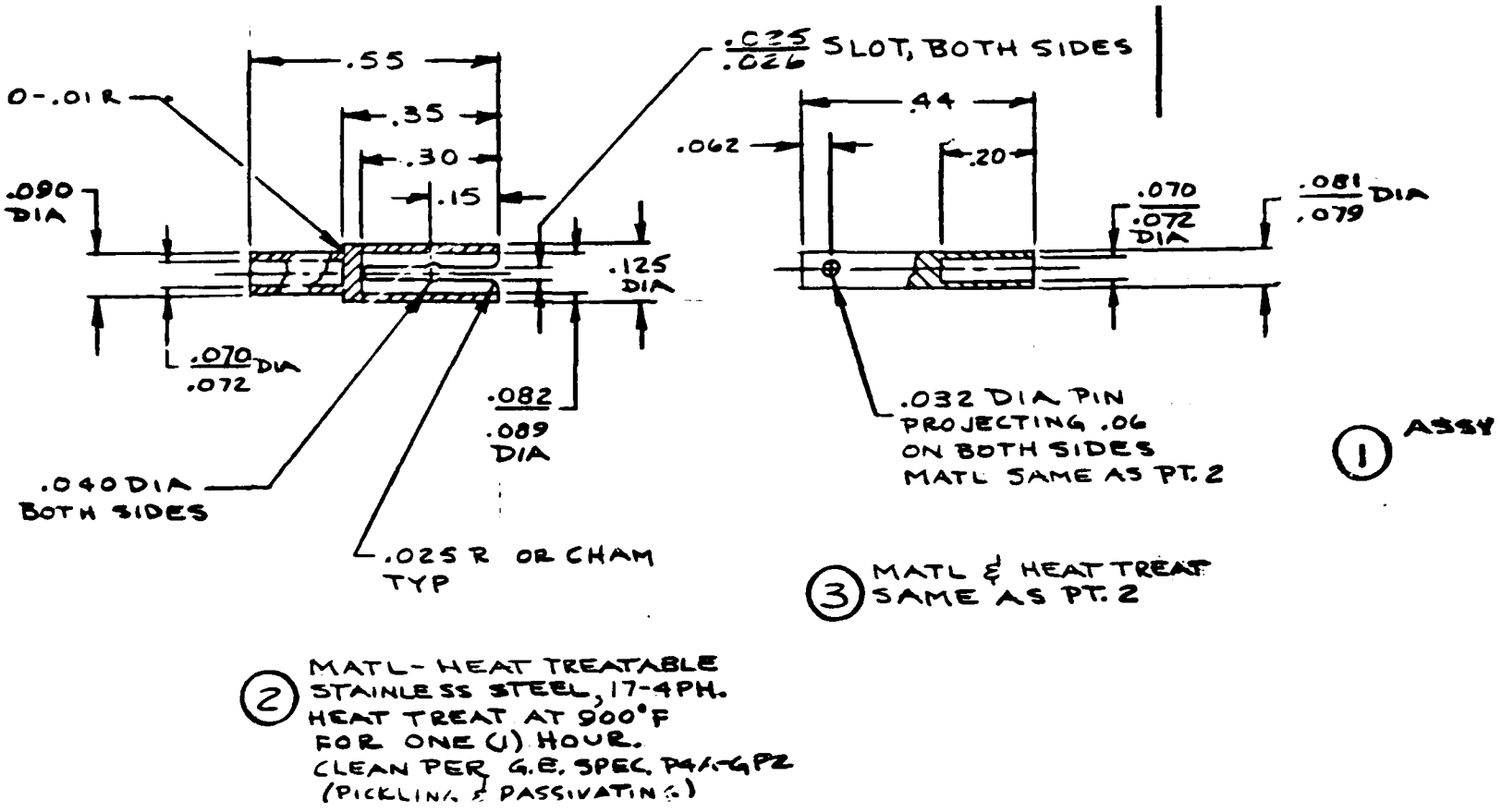


Figure 8. Clasp

the straight axial pull value of the metal clasp to about 5 pounds, since the plastic heat shrinkable sleeving added a considerable amount, depending on its thickness, length and material properties.

Figure 9 illustrates the fixture and test machine used to perform the pull tests. A simulated "neck" was constructed from a hard plastic tube, 13" in circumference, with a soft foam filled insulation wrapped around it, such that the final circumference was 15". The rate of pull was varied on the machine from 1"/min to 20"/min. Table VI indicates the range of forces required for separation for 2 samples, one of which represents the final samples.

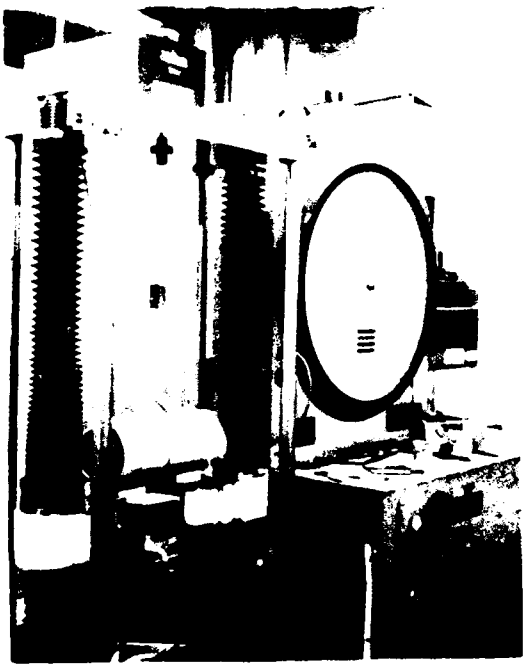


Figure 9. Test Fixture with Simulated Neck

Table VI indicates that the shrinkable sleeving contributes greatly to the separation forces, as shown by conditions 3 and 4. Also, an increased rate of applied force reduces the separation force (condition 6). However, the force for the worst condition (#4) is still above 30 lbs. for both samples. Rather than reduce the metal clasp force below about 5 lbs. (condition 1) we feel that the more logical choice is to reduce the shrink tubing plastic strength. However, at the time the samples were made up, a thinner tubing material was not available. Therefore, all sample parts submitted have the characteristics of sample B, which differs physically from sample A by only a 0.001" decrease in the width of the slot.

TABLE VI
CLASP SEPARATION FORCES UNDER VARIOUS CONDITIONS

	Condition	Pull Rate Inch/Minute	Separation Forces, lbs. Sample #A (Note 2)	Sample #B (Note 3)	Sample #C (Note 5)
1.	Load applied along axis of clasp. No plastic sleeving	1	4.8 lbs.	5.3 lbs.	4.8 lbs.
2.	Straight axial pull. With plastic sleeving (note 1)	1	9.5 lbs.	10.3 lbs.	11.8 lbs.
3.	Around dummy neck. No sleeving. Clasp opposite force.	1	15.3 lbs.	19.6 lbs.	12.5 lbs.
4.	Around dummy neck. With sleeving 1.0 inch long. Clasp opposite force	1	39.5 lbs.	57.0 lbs.	39.1 lbs.
5.	Around dummy neck. Clasp at side of neck, with sleeving.	1	25.8 lbs.	—	19.0 lbs.
6.	Around dummy neck. Clasp opposite force. With sleeving 1.0 inch long. Identical to condition 4, except pull rate is faster.	20	30.5 lbs.	—	

Note 1 - The sleeving is polyolefin heat shrinkable sleeving, 1/8" nom. diameter, 7 mils thick.
Note 2 - Sample A is the same as in Figure 1, except the groove width is 0.026/0.027 wide.
Note 3 - Sample B is the same as in Figure 1, and the same as sample parts provided.
Note 4 - All values are the average of the last 2 tests. After the first few pull tests, the values remained approximately constant (±10%)
Note 5 - Same as sample B except that the poloyolefin is Raychem RT 876.

Another difference between samples A and B did occur, however, in the order of testing. Conditions 2 through 4 were tested on B prior to condition 1. On sample A, several tests of condition 1 were required to lower the average value down to the final 4.8 lbs. It was consistently noted that the initial force falls off slightly, apparently as a result of the immediate wear on the machined surfaces. This effect probably accounts for most of the increase between sample A and B; and if the order were reversed the results could also be expected to change slightly. Therefore, the method of manufacture, e.g. molding, stamping, or machining, may have some effect on the initial forces, depending on the wear characteristics.

The tests and samples do indicate that a consistent and safe limit on the separation force can be provided with the proposed design. With the dimensions of Figure 8, a force of less than 30 lbs. for the "worst" case can be achieved with a thinner or weaker shrink sleeving. It can also be achieved as other tests have shown, with a shorter length of the same sleeving, but the length on the samples provided was cut to cover the entire clasp rather than leave any exposed metal.

A length of approximately 28 inches was selected for the final samples. The maximum head circumference (99 percentile) of a sample of trainees has been measured [12] and found to be 60.0 cm (23.6 in.). The minimum (1 percentile) was 52.7 cm (20.7 in.). This length should then fit every intended wearer, yet will not be so loose as to come back over the head except when deliberately removed.

7. Identification Tag Format

The final format of the information on the identification tag should be determined on the basis of user requirements; the proposed layout shown in Figure 10 has been used on the tag samples. The major points in this arrangement are:

- The front or image side of the tag is arranged so that the information can be read in the position that the tag normally hangs.
- All the alphanumeric information on this side is of high priority, i.e., blood type, name, serial number, pharmacological limitations,* and religious preference.
- The image is prominently displayed on the front side.
- The reverse side format conforms to the arrangement of fingerprints on standard fingerprint cards.
- The blood type and serial number are repeated on the reverse side, but at opposite ends of the tag from the respective locations on the front side.
- Physical attributes are listed on the back side.
- There is an area on the back side for unspecified alphanumeric characters, perhaps coded information.

8. Coding of Security Information

For the ID tags, several methods of coding secure information into the tag were considered and two are being recommended for evaluation; these are 1) alphanumeric characters and 2) modification to the half-tone spatial structure. Of these the first is inherent in the tag — criteria of the alphanumeric characters on the reverse side are set aside for coded information, as discussed in Section III. A. 7.

The second approach is an optical technique and is a more subtle one. The "signature" built into the tag is not apparent to the eye, or to the usual methods of inspection of the identification tag. This technique is described below.

The usual half-tone screen is regular and periodic and when its diffraction pattern is examined it is predictable and uniform. The phase and amplitude of the diffracted orders are controlled by the half-tone pattern.

*Ultimately, medical personnel should designate appropriate abbreviations.

If an irregularity is intentionally placed in the half-tone screen a predictable change will occur in the diffraction pattern from the half-tone picture on the identification tag. By matched filtering techniques in the diffraction plane it is easy to distinguish a picture made by a half-tone screen that has intentionally been made unique.

It is, therefore, possible to use the half-tone pattern, which must be present in any case, to give a grey scale to the picture as a coding device. For chemically etched tags a rotating screen with distinct half-tone structures could be incorporated into the fabrication equipment, giving each tag a predetermined code. In the laser etched case, the structure could be computed for each based, perhaps, on the individual's input data. With this variety the fact that coding is included is further concealed. Part of the coded information could be an anti-counterfeiting check of the tag itself.

Other techniques were considered but are not recommended. The inclusion of a magnetic pattern in the plastic fails because the base metal is magnetic. Placing a chemical code in the plastic is possible, but is more difficult to implement. Care would have to be taken that the trace chemicals did not diffuse or react with time. This code would be lost at the high temperature, whereas the one recommended would be preserved.

B. MICRODOT DEVELOPMENT

This work was based on a process for etching thin metal films with a laser, which had been previously developed by the Electronics Laboratory of the General Electric Company. In this process metal films up to several hundred angstroms thick were deposited on a substrate and then selectively removed or etched, in accordance with a prescribed pattern, by a single pulse of radiation from a ruby laser. The pattern developed by this technique was characterized by high resolution, up to 1000 line-pairs per millimeter in the best films, and was a reasonably exact copy of a master original pattern of much larger size. Master original patterns or masks were made both photographically and by stencils; the laser-etched copies were positives if the original was a positive and negatives if it was a negative. Optics from standard microscope systems were used.

The microdot development project utilized the basic experimental apparatus already assembled and shown in Figure 10. The objective was to demonstrate the feasibility of producing small accurate patterns that were still durable and interpretable after exposure to temperature of 2000°F for periods of 5 minutes or more. To accomplish this objective various tasks were undertaken; these included film materials, substrate selection and various preparation and laser-etching techniques that were capable of packing a sufficient amount of data in a one-half to one millimeter microdot to adequately identify a person who was its bearer. Specific elements to be recorded on the microdot structure were identification photographs (which for ease of recording and subsequent identification could be processed into line drawings), letters and numbers, and fingerprints.

1. Substrate Selection

Until the beginning of this project the most refractory substrate on which laser etchings had been supported was glass. The initial work was done on borosilicate glass while, at the same time, more refractory substrates were being sought and procured. The borosilicate glass distorted badly in temperature tests and the resultant recorded laser etchings were also badly distorted. Aluminum oxide ceramics, both glazed and unglazed, were also tried, but the resolution on metal films deposited on these substrates was less than one-third that of the same films on glass and other homogeneous substrates. Sapphire and fused quartz were tried and both proved to be quite successful in terms of resolution and refractory properties. Both of these materials can easily be polished flat, although quartz is easier to work. While sapphire is more refractory both materials can withstand temperatures in excess of 2000°F for hours. Once tested these two substrates were used for all subsequent development work. They were prepared by evaporating metal films on substrate pieces one-half to one millimeter thick.

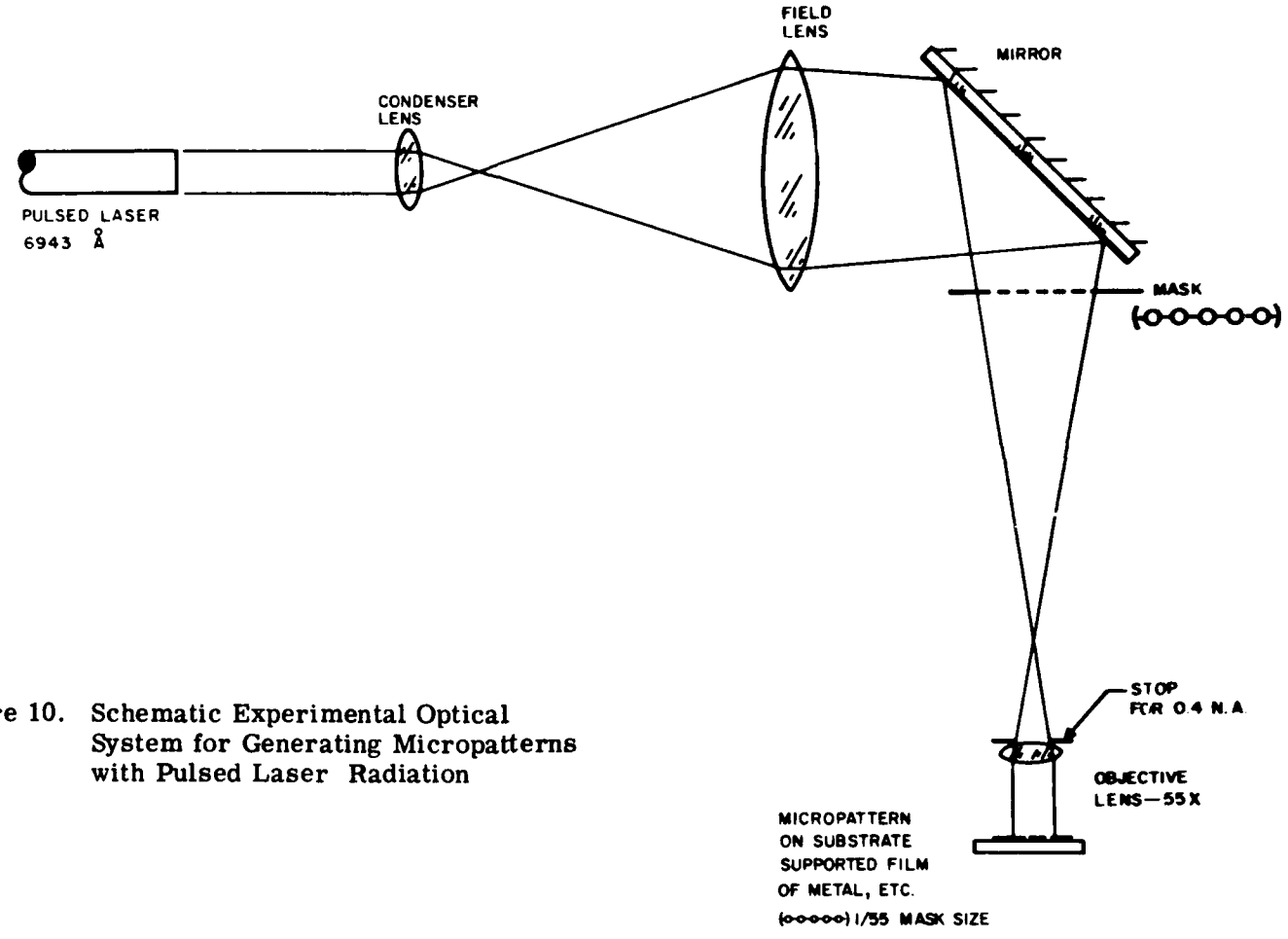


Figure 10. Schematic Experimental Optical System for Generating Micropatterns with Pulsed Laser Radiation

2. Films and Their Preparation

Previous work had shown that nickel films possessed good recording characteristics insofar as resolution and uniformity were concerned. Little was known of their refractory properties in the thickness range that produced ideal recordings, which was below 200 Å. At thicknesses above 200 Å, the films exhibited rapidly declining resolution, erratic recording properties and non-uniformity. After some unsuccessful experiments with glass, nickel was evaporated on sapphire and quartz. The resolution and uniformity in the laser-etched recordings peaked at film thicknesses, of between 50 and 100 Å and the results were, essentially, the same as for nickel films on glass substrates.

The films were prepared by vacuum evaporation at a pressure of 10^{-5} torr. Before evaporation, the substrate was first cleaned by ion bombardment. This procedure was very important because it enhanced the adhesive and durable properties of the films. Temperature tests, in which the nickel films were exposed to 2000°F (and, in a few cases, even higher temperatures) for one to thirty minutes yielded disappointing results. That is, while a recording still existed, it was in a transparent nickel oxide film of poor durability. The recording could be observed with a phase-contrast microscope. There was no distortion of the recording, but the contrast was reduced and, basically, only a surface thickness difference remained. This difference could be best observed using an optical interference technique such as phase contrast. The optical transmittance of the film changed from typical values of 25 to 50% to 4% or less after heating.

Most disappointing, however, was the durability of the remaining recording. Before heating, it was abrasion resistant, but after exposure to 2000°F it could be removed or made illegible by light rubbing with a pencil eraser.

A search for a superior material with good resolution and superior refractory properties resulted in experimentation with chromium film. These were prepared the same way as the nickel films. Experiments with different thicknesses of chromium films showed 100 Å to be the optimum thickness for resolution. The resolution approached that of nickel films, yielding an estimated 600 line-pairs per millimeter of laser etching. The failure of resolution, particularly toward the thicker films, was not as abrupt as with thick nickel films. It is believed that this is because the thicker chromium films adhere to the substrate more uniformly. Films as thick as 300 Å were successfully laser-etched.

The thickness of the chromium films was measured by monitoring their optical transmittance. These results were calibrated by interference microscope measurements. The basic data on film thickness was obtained from Bond[13], but we found chromium films to be 20% thicker for a given light transmittance than that data indicates.

3. Temperature Tests of Chromium Films

Temperature testing of the chromium films was performed in the same manner as the tests of the nickel films; but more extensive tests were made because the results were far more positive. When heated to 2000°F the etched chromium films became more transparent and slightly yellowish in color. There was, however, adequate contrast to permit the laser etched recordings to be interpreted with a conventional bright field microscope. In fact, the contrast was impaired very little by temperature tests in which the samples were subjected to 2000°F from 5 minutes to 1/2 hour.

Most important, the laser-etched recordings were durable after temperature tests. They withstood considerable rubbing from a pencil eraser and could be rubbed with a pointed scriber without damage. Heavy pressure on the scriber point marred the film in a way that indicated the film was ductile. Only deliberate rubbing with the scriber point could obliterate the record by interfering with legibility.

Temperature tests were conducted in various ways to vary heat input rates. The laser-etched samples were brought up to temperature over a period of several hours by starting with a cold furnace. In other tests they were plunged into a furnace already at 2000°F. The results showed little difference, except that the sapphire substrates sometimes cracked when they were removed from a hot furnace and placed on a cold surface.

4. Experimental Results

Photomicrographs of the results are presented as Figures 11 thru 18. All of these are of 100 Å thick chromium films on quartz substrates. This combination of materials yielded optimum results, in terms of demonstrating feasibility, and it is recommended that it be used as a basis in any subsequent work directed toward designing and producing practical microdots on a large scale.

More energy was required to evaporate chromium films than for nickel films. A value of 0.4 joules mm^2 was required to uniformly evaporate a pattern in a 100 Å nickel film. This contrasts with a value of 0.2 joule mm^2 for optimum nickel films.

Recognizable line drawings of faces 70 microns high were laser etched in 40 and 100 Å chromium films. A very recognizable photomicrograph of one of these, which was increased in a furnace at 2000°F for 5 minutes, has been included. Letters and numerals 5 to 10 microns high were also successfully laser-etched in these films. A lesser degree of success was obtained with fingerprints. It is apparent that, because of the importance of fine detail in a fingerprint, each print must be at least 50, and possibly 100, microns high to be adequate. It is recommended that complete identification data occupy both sides of a 0.5 mm diameter microdot or one side of a 1 mm diameter microdot. This represents a safe data density based on the techniques and results developed in this investigation.

A further effort at recording laser-etched data was attempted but was not successful. The film coated substrate was irradiated with much more energy than needed to evaporate the metal film in an attempt to infuse the metal or its oxide into the substrate. This did produce a change in the substrate, but examination indicated that only etching had occurred and not infusion. The resolution was poor. This experiment was performed with both nickel and chromium.

Figure 11.

Photomicrograph of laser-etched portrait before temperature test. Head is 133 microns high from top to jaw. Chromium film on fused quartz is 100 angstroms thick. (Sample A)

Figure 12.

Same as before after 10 minute exposure to 2000 degrees fahrenheit.
Both photomicrographs 263X reflected light bright-field. (Sample A)

Figure 13.

Photomicrograph of laser-etched portrait before temperature test. Head is 133 microns high from top to jaw. Chromium film on fused quartz is 100 angstroms thick. (Sample B)

Figure 14.

Same as before after 10 minute exposure to 2000 degrees fahrenheit.
Both photomicrographs 263X reflected light bright-field. (Sample B)

Figure 15.

Photomicrograph of laser-etched characters $6\frac{1}{2}$, 8 and 13 microns high before temperature test. Chromium film on fused quartz is 100 angstroms thick.

Figure 16.

Laser-etched characters after 10 minute exposure to 2000 degrees fahrenheit.
Both photomicrographs 263X reflected light bright-field.

Figure 17.

Photomicrograph of laser-etched fingerprints before temperature test. Chromium film on fused quartz is 100 angstroms thick.

Figure 18.

Laser-etched fingerprints after 10 minute exposure to 2000 degrees fahrenheit.
Both photomicrographs 263X reflected light bright-field.

III. CONCLUSION AND RECOMMENDATIONS

Investigations carried out on this program have resulted in the following conclusions and recommendations:

- A tag base material of Nickel 200 or Nickel 440 (NiBe) provides better high temperature scaling resistance than any other metals investigated. The former is the recommended material. It can be chemically or laser etched, but its grain size must be controlled.

- Plastic coating of the tag with cellulose propionate gives a clear coating that is incorrodible, quiet, and leaves no residue when exposed to a temperature of 2000^{O}F.

- A standard stainless steel necklace with polyurethane or nylon coating is comfortable to the wearer and is quiet. The steel survives the high temperature, but exhibits some scaling.

- A hardened stainless steel clasp has been designed and tested as a safety release in the necklace. To keep its crevices clear it is overcoated with heat shrinkable polyolefin tubing after assembly to the tag. The tubing must be carefully selected to prevent any undue increase of the parting load of the clasp. Raychem RT 876 is recommended.

- Coding techniques recommended for the tag are 1) use of additional characters, and 2) modification of the half-tone structure of the image. The latter technique is quite novel and has not been included in the tags.

- The chemically etched tags with positive images provide a clear and prominent display of the recorded facial image, fingerprints and alphanumeric characters.

  After exposure to 2000^{O}F the oxide film partially obscures the image and fingerprints. Etch depth must be controlled to minimize this.

- The laser etched tags exhibit line drawing representations of the facial image and fingerprints. As such, the display is less prominent. This was necessitated by practical limitations on the programming of the existing laser facility. The same oxidation occurs with these tags, but the permanence of the information is better. Deeper etching is possible with the laser technique.

- The chemical approach has advantages in terms of fabrication rate, because of its compatibility with semi-batch processing, and would lead to a quicker realization of a suitable tag. In contrast, the laser offers a shorter fabrication time, ease of automation, and simpler logistic support. In addition there is no problem with disposal of spent chemicals. Furthermore, the laser technology is in a very active growth state and improvements in the (conservative) etching times can be expected. On balance, the laser etching approach is recommended for further development.

- To reduce the overall tag fabrication time, some efforts should be expended to shorten the lamination process.

- The basic feasibility of laser recording of the information on the microdots has been established. These have proven durable at 2000°F, especially if the exposure energy is well controlled.

REFERENCES

1. Chun, M. K. and K. Rose, "Interaction of High-Intensity Laser Beams with Metals", J. Appl. Phys., Vol. 41, No. 2 pp. 614-620, Feb. 1970.

2. Ready, J. F., "Effects Due to Absorption of Laser Radiation", J. Appl. Phys., Vol. 36, No. 2, pp. 462-468, Feb. 1965.

3. Ready, J. F., Effects of High-Power Laser Radiation, New York, Academic Press, 1971.

4. Gagliano, F. P., R. M. Lumley, L. S. Watkins, "Lasers in Industry", Proc. IEEE, Vol. 57, No. 2, pp. 114-147, Feb. 1969.

5. Dabby, F. W. and U. C. Paek, "High-Intensity Laser-Induced Vaporization and Explosion of Solid Material", IEEE J. Quant. Electr., Vol. QE-8, No. 2, pp. 106-111, Feb. 1972.

6. U.C. Paek and F. P. Gagliano, "Thermal Analysis of Laser Drilling Process", IEEE J. Quant. Electr., Vol. QE-8, No. 2, pp. 112-119, Feb. 1972.

7. Bernel G, E. and J. F. Ready, "Lasers in High Temperature Research", pp. 133-157 in Proc. 3rd International Conference on High Temperature Technology, September 1967.

8. Nichols, K. G., "Review of Laser Microwelding and Micromachining", Proc. IEE Vol. 116, No. 12, p. 2093-2100, December 1969.

9. Ready, J. F., "Using the Laser in Production — The Design Implications Mechanical Engineering, Vol. 92, No. 9, p. 18-24, September 1970.

10. Bastien, J., "Applications des Lasers au Travail des Materiaux", Journées d'Etude des Applications Industrielles des Lasers, Paris, 19-20 November 1970, 8p.

11. Eleccion, M. "Materials Processing with Lasers", IEEE Spectrum, Vol. 9, No. 4, pp 62-72, April 1972.

12. Daniels, G. S., H. C. Meyers, and E. Churchill, Anthropometry of Male Basic Trainees, WADC Technical Report 53-49, Wright Air Development Center, July 1953.

13. Bond, W. L., "Notes on Solution of Problems in Odd Job Vapor Coating", JOSA, Vol. 44, No. 6, June 1954, pp. 429-458.

APPENDIX A

PROPERTIES OF MATERIALS CONSIDERED

Property	ASTM Method	ABS (Med Impact)	CAB (All Grades)	CP	PC	PP	PVC	PTP
Specific Gravity	D792	1.05-1.07	1.15 - 1.22	1.19 - 1.22	1.20	0.90 - 0.91	1.20 - 1.55	1.28
Coef. of Thermal Expansion (10^{-5} per ^{o}F)	D696	3.2 - 4.8	6 - 9	6 - 9	3.75	3.8 - 5.8	-	
Water Absorption (24 hrs, %)	D570	0.2 - 0.4	0.9 - 2	1.3 - 2	0.15	< 0.01 - 0.03	0.2 - 1.0	
Flammability (in /min)	D635	1.0 - 1.6	0.5 - 1.5	0.5 - 1.5	SE	0.7 - 1	SE	
Luminous Transmittance %	D791							
Transparency (visible light, %)		-	75 - 95	80 - 92	75 - 85	Translucent	-	75
Refractive Index	D1746	-	1.46 - 1.49	1.46 - 1.49	1.586	-	-	
Flexure Strength (1000 psi)	D790	9.9 - 11.8	2.5 - 9.0	5.6 - 7.9	13.5	6.7	-	
Hardness (Rockwell)	D785	R10^{a} - 115	R23 - 114		M70	R80 - 100		
(Shore)							A50 - 100	
Cold Flex. Temp. (F)	D1043	-					-70 - 0	
Cold Bend Temp. (F)		-					-40 - -4	
Max. Rec. Ser. Temp. (F)		-					150 - 220	
Heat Dist. Temp	D648							
66 psi (^{o}F)		-	136 - 222	163 - 201	-	205 - 230		338
264 psi (^{o}F)		185 - 223	118 - 196	129 - 173	270	135 - 140		(D1637)

TECHNICAL SERVICES LABORATORY
of the ELECTRONICS LABORATORY • ELECTRONICS PARK • SYRACUSE • NEW YORK • 13201

P4A-GP2 REV E
SHEET NO 1 of 3
CODE IDENT 13688

Issued November 6, 1968
Reissued October 27, 1971

PICKLING AND PASSIVATION OF STAINLESS STEEL

1. Scope

This process outlines the procedure for pickling and passivating of chrome-iron and chrome-nickel-iron alloys containing 12% or more of chromium.

2. Government Specification

2.1 MIL-F-14072 QQ-P-35
MIL-STD-171A

3. Materials

3.1	Alkaline Cleaner	-	See approved list
	Ferrifloc	-	Tennessee Corp. - Dist. by Faesy & Bestoff, N. Y., N. Y.
	Oakite #31	-	Oakite Products
	Hydrochloric acid	-	Commercial grade
	Nitric acid	-	"
	Hydrofluoric acid	-	"
	Sodium dichromate	-	"
	Chromic acid	-	"

3.2 Pickling (Part I)

This operation shall be performed only after heat treatment to remove scale and shall never be done after final machining. Pickling shall always be followed by immediate passivation.

1. Immerse parts in an approved alkaline cleaner made up at 8 oz/gal and operated at 180-200°F until all organic impurities are removed.

2. Rinse in running water.

3. Pickle in one of following until heat scale is removed.

a.	Ferrifloc	-	8 oz/gal
	Hydrofluoric acid	-	1.5-2.0 fl. oz/gal
	Temperature	-	140° - 180°F
b.	Nitric Acid	-	13 fl. oz/gal
	Hydrofluoric acid	-	3 fl. oz/gal
	Hydrochloric acid	-	1.4 fl. oz/gal
	Temperature	-	130° - 140°F
c.	Oakite #31	-	50%
	Water	-	50%
	Temperature	-	160° - 200°F

4. Rinse in running water.

5. Rinse in hot water.

6. Passivater per Step 3 in Part II.

3.3 Passivation (Part II)

This treatment should be performed immediately after final machining or pickling.

1. Immerse parts in an approved alkaline cleaner made up at 8 oz/gal and operated at 180°-200°F until all organic impurities are removed.

2. Rinse in running water.

3. Passivate in the following solution:

a. Nitric Acid	-	20% by volume
Water	-	80% by volume
Sodium dichromate	-	2-4 oz/gal

300 series steels may be processed through this solution for 20 minutes minimum at 120-130°F or for 30 minutes minimum at 70-90°F.

400 series steels may only be processed through this solution at 120-130°F for 20 minutes minimum.

4. Rinse in clean hot water.

5. Immerse for one hour in a 5% solution of sodium dichromate heated to 150°F.

6. Rinse in clean hot water.

7. Rinse in a Hot (170°F) rinse composed of enough chromic acid to reduce the acidity to pH 3-5.

8. Dry using filtered air blast.

*Note:
Steps 5-7 are required to comply with MIL-STD 171A and QQ-P-35 with any other specification it is permissible to eliminate these steps and proceed to Step 8.

4. Quality Control Assurance

4.1 The effectiveness of the passivation can be determined by putting a drop of the following solution on the part.

Copper sulfate	-	100 grams per liter of water
Sulfuric acid	-	15 cc. per liter of water

If the surface is not passivated, a copper film will appear under the drop.

4.2 The referee method of testing for proper passivation is according to paragraph 4.3.2. of QQ-P-35.

Clean part or parts with acetone to remove finger marks and expose to 100% humidity at 100°F for four to five hours in a humidity cabinet. There should be no sign of rust at the conclusion of the test.

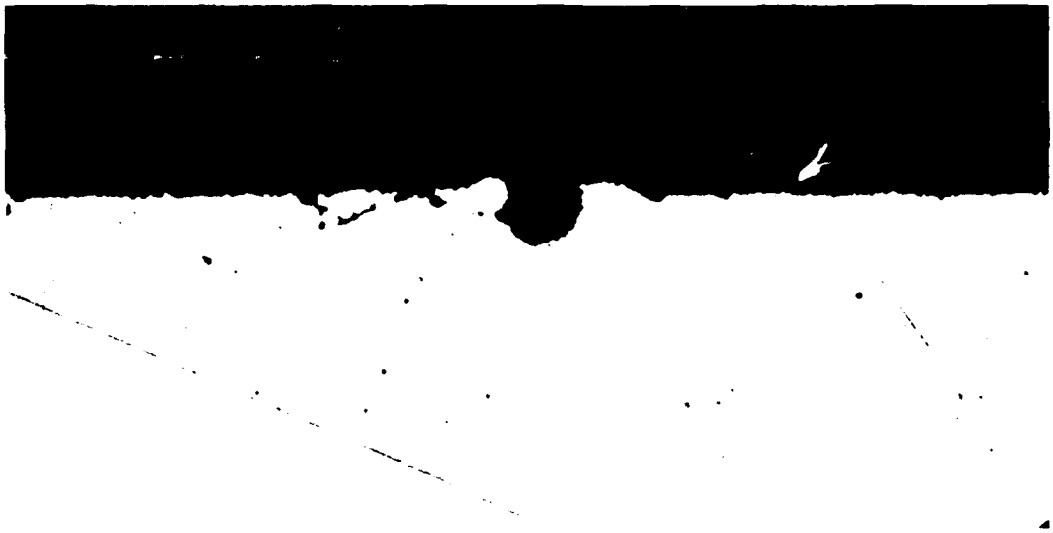
PREPARED BY

APPROVED BY:

SUPERSEDES ISSUE OF:

APPENDIX C

TECHNICAL SERVICES LABORATORY
METALLOGRAPHIC SECTION
Room 108, Building 3
Electronics Park
Ext. 2022



NEG. # 333 LAB. # MAG. X 400

MATERIAL Nickel identification tag.

ETCH REAGENT Laser etched

PHOTOMICROGRAPH SHOWS Cross section of an etched line. Depicts melted area in surface after nameplate has been oxidized. Void area that now forms the detail is .0006" wide and .00087" deep.