

MIL-STD-867A(USAF)

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MILITARY STANDARD

TEMPER ETCH INSPECTION



FSC MISC

MIL-STD-867A(USAF)

DEPARTMENT OF THE AIR FORCE

Washington DC 20330

TEMPER ETCH INSPECTION

MIL-STD-867A(USAF)

1. This Military Standard is approved for use by HQ AFLC CASO/LODS, Department of the Air Force, and is available for use by all Departments and Agencies of the Department of Defense.

2. Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: HQ AFLC CASO/LODS, Federal Center, Battle Creek, MI 49016, by using the self-addressed Standardization Document Improvement Proposal (Form DD 1426) appearing at the end of this document or by letter.

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TEMPER ETCH INSPECTION

1. SCOPE

1.1 Scope. This standard establishes requirements and tests for performing temper etch inspection of carburized steels and tool and low alloy steels which are heat treated above 1.24×10^9 Pascal (PA) [1.26×10^2 kilogram-force/square millimeter (Kgf/mm²) 1.8×10^5 pound-force/square inch (psi)].

1.2 Application. This standard applies to aircraft and missiles structural and propulsion system components when MIL-I-6870 and/or MIL-STD-866 are referenced as an item specification in the contract or order.

1.2.1 Groups. Temper etch inspection is typically performed on steels from the following groups:

Group A - Low alloy steels - SAE 52100, SAE 4140, SAE 4330, SAE 4340, 300M, D6AC, 440C, etc.

Carburizing Steels - SAE 4620, 9310, AMS 6260, etc.

Group B - Tool steels - H-11, M-50, M-2, H-13, etc.

1.2.2 Case hardened steels. This standard is not applicable to surface hardened steels produced by nitriding or carbonitriding.

1.3 Purpose. This inspection is to be conducted to determine if heat has been induced in a component, subsequent to final heat treat, by machining, grinding, or other means which will adversely affect the required properties of that component.

1.3.1 Localized discontinuous carburization. To detect localized discontinuous carburization due to an inadvertent carburize stop-off and to determine the presence of or lack of total carburization in specified locations.

1.3.2 Decarburized surface layer. To detect a decarburized surface layer.

2. REFERENCED DOCUMENTS

2.1 Issues of documents. The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this standard to the extent specified herein.

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SPECIFICATIONS

Federal

O-A-51	Acetone, technical
O-E-760	Ethyl Alcohol (Ethanol); Denatured Alcohol; and Proprietary Solvent
O-H-765	Hydrochloric Acid, Technical
O-N-350	Nitric Acid, Technical
O-S-598	Sodium Hydroxide, Technical

Military

MIL-I-6868	Inspection Process, Magnetic Particle
MIL-H-6875	Heat Treatment of Steels (Aircraft), Process For
MIL-C-16173	Corrosion Preventive Compound Solvent Cutback, Cold Application
MIL-C-38736	Compounds, Solvent for use in Fuel Tanks

(Copies of specifications, standards, drawings and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

3. DEFINITIONS (Not Applicable)

4. GENERAL STATEMENTS OF REQUIREMENTS

4.1 Materials and equipment.

4.1.1 Materials. The use of reclaimed materials shall be encouraged to the maximum extent possible. Materials required are as follows:

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- a. Methyl or ethyl alcohol [$\text{CH}_3\text{OH}-\text{C}_2\text{H}_5\text{OH}$], (Federal O-E-760)
- b. Hydrochloric Acid [HCL], (Federal O-H-765)
- c. Nitric Acid [HNO_3], (Federal O-N-350)
- d. Sodium Hydroxide [NaOH], (Federal O-S-598)
- e. In lieu of (d) above, any common alkali solution with a pH greater than or equal to 10 may be used.
- f. Ammonium Peroxydisulfate (Ammonium Persulfate) $(\text{NH}_4)_2\text{S}_2\text{O}_8$
Commercial

4.1.2 Equipment

- a. A commercial industrial vapor degreaser or a hot alkaline cleaning system.
- b. Containers used for acid solutions shall be non-reactive to acid and alkali solutions, preferably made of polyethylene or polyvinyl chloride material.
- c. Water rinse tanks shall be equipped with a constant overflow or skimming device.
- d. The following auxiliary equipment shall be provided:
 - (1) Clock or timer with sweep second hand.
 - (2) Light capable of insuring 2152.8 Lux. (200 ft. Candles) at inspection level.
 - (3) Burette for titrating solutions for acid concentrations.

4.1.3 Company process specifications. Company process specifications may be prepared incorporating the applicable requirements of this standard and in addition supplying the detailed information necessary to meet or exceed this standard using the particular equipment, process, personnel and test facilities required to meet the reliability requirements of the product. Personnel training, qualification and certification procedures shall be documented.

4.1.3.1 Standardization. The company process specification shall reflect procedures and records to assure adequate Quality Assurance measures are being enforced to keep the NDT process in control. Applicable drawings or other documents shall

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specify the allowable severity and concentration of allowable temper etch conditions thru use of photographs or wording as to allowable grinding burn severities by color, size and/or hardness differential. The component drawing or applicable document shall also show:

- a. The defined etch process
- b. Frequency of inspection
- c. Zone classifications according to the structural integrity requirements of the components.

4.1.3.2 Detail process procedures. Written detail process, procedures shall be maintained in the process area and shall contain at least the following information:

- a. Process name
- b. Process step number and operation name
- c. Processing time for each step
- d. Processing temperature for each step
- e. Materials used in each processing tank

4.2 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. The supplier may utilize his own facilities or any other commercial laboratory acceptable to the customer.

4.3 Manufacturing process cycle. The temper etch process shall be used after final grinding or machining operations but prior to any processing which may interfere with this inspection, such as plating or shot peening. Parts finished to close tolerances shall be dimensionally inspected after etch inspection as the etch process may remove from 0.0013 to 0.0051 millimeters (0.00005 to 0.0002 inch) of material from the surface of the part.

4.4 Record of inspection. The results of the etch inspection shall be appropriately recorded and filed.

4.5 Qualification and certification. Before performing temper etch inspection on production parts inspection personnel shall be qualified and certified in accordance with this standard.

4.5.1 Initial qualification. At qualification inspection personnel shall pass physical, written and practical tests.

4.5.1.1 Physical test. The inspection applicant must pass a vision test administered by medically qualified personnel, with a requirement of reading Jaeger J-2 or Ortho-Rater 8 letters, at 0.305 meters (12 inches) in at least one eye.

4.5.1.2 Written test. Twenty to thirty written questions shall be administered. Questions shall cover cleaning, operating procedures, and inspection techniques that the applicant would encounter during inspection.

4.5.1.3 Practical test. At least three selected specimens representative of actual products will be tested. During these tests, at least ten different check points shall be graded showing an understanding of test variables and an ability to perform temper etch inspection and correctly interpret its results.

4.5.1.4 Re-examine for qualification. Personnel examined and not meeting qualification standards must wait 30 days and show evidence of having taken suitable corrective action, additional training or self-study before re-examination.

4.5.1.5 Recertification. Personnel performing temper etch inspection shall be requalified and certified as specified in 4.5.1.2 and 4.5.1.3 at intervals not to exceed 3 years. Vision tests are required yearly (see 4.5.1.1).

4.5.2 Certification. Records for all qualified personnel shall be maintained and include: Date of qualification; results of physical, written and practical tests; and experience as a temper etch inspector.

5. DETAILED STATEMENTS OF REQUIREMENTS

5.1 Process. Temper etch inspection may be accomplished utilizing a process procedure as listed in Tables I, II, III or IV for the type of steel to be inspected. The entire surface of the feature in question shall be etched and inspected.

5.2 Cleaning. Parts shall be cleaned to a water break free condition just prior to the etch process by vapor degreasing, hot ultrasonic cleaning or nitric acid etch. To obtain a uniform surface for optimum temper etch on rough surfaces, grit blasting or liquid honing is recommended.

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TABLE I

Table I may be used to etch low alloy steels (Group A).

STEP	PROCESS	SOLUTION	TIME	TEMP	REMARKS
1	Nitric Acid Etch	Nitric Acid 3%-5% (By volume) in alcohol or Nitric Acid 3%-5% (by volume) in water	15-60 Seconds - - - - 5-15 Seconds	Ambient	Agitate parts when immersed. Exact time may vary to produce desired black oxide film
2	Rinse	Circulating Water	Rinse Only	Ambient	To remove acid. Total immersion required.
3	Hydrochloric Acid Dip	Hydrochloric Acid 4%-6% (By volume) in alcohol or water Hydrochloric Acid 1%-3% (by volume) in alcohol	30-60 Seconds 5-15 Seconds	Ambient Part immersed hot directly from a 82.2°C (180°F) min vapor degreaser	Agitate parts when immersed to remove black oxide film and provide uniform brownish grey surface
4	Rinse	Running Water	Rinse Only	Ambient	Agitate parts when immersed
5	Neutralize	Any Alkali Solution with pH of 10 minimum	15 Seconds Minimum	15.5-82.2°C (60-180°F)	Agitate parts when immersed
6	Rinse	Circulating Water	Rinse Only	Ambient	To remove Caustic.
*7	Rinse	Alcohol	Rinse Only	Ambient	To remove water
8	Oil	Rust Preventative Oil	Dip Only	Ambient	To prevent Corrosion & Aid to Color Contrast of Burns
9	Inspect and evaluate per appropriate criteria.				
*Operational procedure: Hot water rinse [65.5°C Minimum (150°F)], followed by a dry air blast may be used in lieu of this rinse.					
Precleaning, water rinse and steps 1 thru 8 shall be one continuous operation.					

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TABLE II

Table II may be used to acid etch tool steels, (Group B).

STEP	PROCESS	SOLUTION	TIME	TEMP	REMARKS
1	Hydro-chloric Acid Clean	Hydro-chloric Acid 4.0%-6.0% (by volume) in alcohol or water	1-1/2 to 3-1/2 Min.	Ambient	To deoxidize part. Agitate part during entire immersion time.
2	Rinse	Circulating Water	Rinse Only	Ambient	To remove acid. Total immersion required.
3	Nitric Acid	Nitric Acid 2.5% to 3.5% (by volume) in alcohol or water	1-1/2 to 2-1/2 Minutes	Ambient	Agitate parts during entire immersion time. Exact time may vary to produce desired black oxide film.
4	Rinse	Circulating Water	Rinse Only	Ambient	To remove acid. Total immersion required.
5	Hydro-chloric Acid dip	Hydro-chloric Acid 4.0%-6.0% (by volume) in alcohol or water	1-1/2 to 3-1/2 Minutes	Ambient	Agitate parts during entire immersion time. To remove black oxide film and produce a uniform brownish/gray surface.
6	Rinse	Circulating Water	Rinse Only	Ambient	Agitate parts when immersed.
7	Neutralize	Any alkali solution with pH of 10 Min	15 Seconds Minimum	Ambient	Agitate parts when immersed.
8	Rinse	Running Water	Rinse Only	Ambient	To remove caustic
*9	Rinse	Alcohol	Rinse Only	Ambient	To remove water
10	Oil	Rust Preventative Oil	Dip Only	Ambient	To prevent corrosion and aid in color contrast of burns.
11	Inspect and evaluate per appropriate criteria.				
*Optional Procedure: Hot water rinse [65.5°C Minimum (150°F)], followed by a dry air blast may be used in lieu of this rinse.					
Precogning, water rinse and steps 1 thru 10 shall be one continuous operation.					

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TABLE III

(Limited Access or Swab Etch Techniques)

STEP	PROCESS	SOLUTION	TIME	TEMP	REMARKS
1	Solvent Clean	Cleaner MIL-C-38736 or Acetone Fed 0-A-51	As necessary	Ambient	Wipe surface with commercial cheese cloth saturated with solvent. Wipe dry with cheese cloth.
2	Nitric Acid Etch	Nitric Acid (Fed-0-N-350) 4% to 10% by volume in Alcohol (Fed 0-E-760)	Until a Black Smutty Etch is produced.	Ambient	Swab the surface with commercial grade cheese cloth saturated with Nitric solution. The more concentrate solution reduces time.
3	Rinse	Acetone (Fed-0-A-51)	To remove Nital	Ambient	Rinse immediately.
4	Acid Clean	Hydro chloric Acid (Fed-0-H- 765) 6 to 10% by volume in water.	To remove Black smut.	Ambient	Swab with commercial cheese cloth saturated with HCL solution.
5	Rinse	Clean Water	To remove HCL	Ambient	Swab with commercial cheese cloth saturated with water.
6	Neutral- izer	Any Alkali solution with pH of 10 min.	To neutralize HCL.	Ambient	Swab with commercial cheese cloth saturated with alkali solution.
7	Rinse	Clean water	To remove caustic	Ambient	Swab with commercial cheese cloth saturated with water.
8	Rinse	Alcohol (Fed 0-E-760)	To remove water.	Ambient	Swab with commercial cheese cloth saturated with alcohol and wipe dry.
9	Oil	Rust Prevent- ative MIL-C- 16173	Swab Only	Ambient	To prevent corrosion.
10	Inspect and evaluate per appropriate criteria				
Steps 1 through 10 should be completed without interruption.					

*TABLE IV
Ammonium Peroxydisulfate (Ammonium Persulfate) swab etch technique

STEP	PROCESS	SOLUTION	TIME	TEMP	REMARKS
1	Solvent Clean	Acetone (Fed O-A-51) or Cleaner (MIL-C-38736)	As necessary	Ambient	Wipe surface with commercial cheese cloth saturated with solvent. Wipe dry with cheese cloth.
2	Etch	Ammonium Peroxy- disulfate 10% by weight in water.	15-60 seconds	Ambient	Make solution just before using swab area with commer- cial cheese cloth saturated with solution.
3	Rinse	Clear Water	As required to remove Alkali Sol.	Ambient	Swab area with commercial cheese cloth saturated with water.
4	Neutral- ization	Any Alkali solution with a pH of 10 min.	As required to neutralize etchant.	Ambient	Swab area with commercial cheese cloth saturated with Alkali solution.
5	Rinse	Clear Water	As required to remove Alkali Sol.	Ambient	Swab area with commercial cheese cloth saturated with water.
6	Rinse	Alcohol (Fed O-E-760)	To remove water.	Ambient	Swab with commer- cial cheese cloth saturated with alcohol. Wipe Dry.
7	Oil	Rust preventative MIL-C-16173	Swab Only	Ambient	To prevent corrosion.

8 Inspect and evaluate per appropriate criteria.

* This process may also be used as an immersion technique,
provided precleaning is in accordance with 5.2.

Steps 1 through 8 should be completed without interruption.

NOTE: No baking is required after etching - this process

NOTE: Make up solution just before using as it becomes inactive
after 72 hours.

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5.3 Acid etch visual inspection. Inspection of etched surfaces shall be performed under light resulting in a minimum of 2152,8 Lux (200 foot candles) at part surface.

5.3.1 Rejection criterion. The presence of the following indications shall be cause for rejection.

a. Untempered martensite as evidenced by light gray to white areas surrounded by a light brown to black border.

b. Overtempered areas as evidenced by light brown to black indications darker than surrounding areas.

5.3.2 Acceptance/rejection standards. The presence of the following indications shall be cause for review to established acceptance/rejection standards (see 4.1.3.1).

a. Noncarburized/Decarburized Areas - Non-grinding related. These areas will appear unusually lighter in color than adjacent carburized areas.

b. Improper Carburization - Non-grinding related. These areas are isolated or complete areas of carburization not required by blueprint.

5.3.3 Acceptable color. If no damage was produced by the preproduction operations, the surface will be a uniform gray. This discoloration is not detrimental to part usage.

5.4 Baking. After the final etch, accepted parts processed by etch methods Table I, II and III shall be baked at $191 \pm 14^{\circ}\text{C}$ ($375 \pm 25^{\circ}\text{F}$) or $10 \pm 13.88^{\circ}\text{C}$ ($50 \pm 25^{\circ}\text{F}$) below the final tempering temperature of the part for four hours. Parts with tempering temperature exceeding 398.88°C (750°F) may be baked at $371.11 \pm 13.89^{\circ}\text{C}$ ($700 \pm 25^{\circ}\text{F}$) for four hours. Such baking to be initiated within eight (8) hours of the final visual inspection and before any plating operations. Parts processed by etch methods in Table IV will not require a bake. All furnaces used shall be certified to MIL-H-6875.

5.5 In-service quality checks.

5.5.1 Acid concentrations. Acid solutions shall be analyzed at a frequency determined to assure concentration limits.

5.5.2 Sensitivity. A standard test piece which contains representative grinding burns will be processed each day that the etch is operated by production inspection personnel to verify that the etching solutions and techniques are adequate to detect grinding burns.

5.5.3 Magnetic particle inspection. All parts shall be magnetic particle inspected in accordance with MIL-I-6868 after baking and prior to other processing which might hinder detection of a discontinuity.

5.5.4 Records. Historical records shall be maintained on all chemical tests and on additions made to the solution.

6. NOTES

6.1 Re-etching. A part may be etched two or more times if dimensions and surface finishes allow, in order to distinguish burn indications from irrelevant indications such as smear or smutty finishes. Aluminum oxide grit-blast between re-etch is recommended.

6.2 Seeking competent advice. A metallurgist shall be consulted in instances where the results of the etching appear inconclusive and where further interpretation is required. When results of etching are inconclusive or interpretation is questionable, a representative part may be sectioned and prepared for metallographic examination. Microhardness and structural characteristics should be determined.

6.3 Hardness testing. If part size and configuration permits, hardness testing in accordance with ASTM-E-18 using 15/N Rockwell test equipment may be used as an evaluation of hardness in moderate to heavy overtempered areas. A reduction of two (2) Rockwell 15/N points or more from the areas directly adjacent to the burn is indicative of an overstressed and crack susceptible condition and should not be accepted in high stress areas. In lieu of Rockwell 15/N hardness testing, 1000 gram maximum load micro hardness testing may be employed.

6.4 Rework/salvage. If sufficient stock remains for rework within the specified dimensions, the parts may be reworked to remove indications of overtempering or rehardening. Parts subjected to this rework shall be re-acid etch inspected.

6.5 Acid etch removal. Discoloration from the acid etch inspection process shall be acceptable unless otherwise specified by the drawing or customer requirements. The discoloration may be removed by polishing, honing, vapor grit blasting, or electrolytic alkaline cleaning methods providing dimensional changes do not exceed drawing requirements and customer approval is obtained.

6.6 Marking. Parts which have met the etch inspection requirements shall be marked in accordance with the applicable drawing, specification, purchase order or contract. Marking, as specified, shall be applied in such manner and location as to be

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harmless to the part and which will preclude removal smearing, or obliterating by subsequent handling. A symbol N or NE may be used.

6.7 Toxic and hazardous substances. This document specifies use of certain materials which have been listed in subpart Z, 29 CFR 1910 (OSHA Standards), "Toxic and Hazardous Substances". Personnel exposure to these materials must be limited to the values specified in applicable portions of 29 CFR 1910.1000.

Custodian:
Air Force - 99

Preparing activity:
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SPECIFICATION ANALYSIS SHEET		Form Approved Budget Bureau No. 22-R255
<p>INSTRUCTIONS: This sheet is to be filled out by personnel, either Government or contractor, involved in the use of the specification in procurement of products for ultimate use by the Department of Defense. This sheet is provided for obtaining information on the use of this specification which will insure that suitable products can be procured with a minimum amount of delay and at the least cost. Comments and the return of this form will be appreciated. Fold on lines on reverse side, staple in corner, and send to preparing activity. Comments and suggestions submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or serve to amend contractual requirements.</p>		
SPECIFICATION		
<i>MIL-STD - 867 A</i>		
ORGANIZATION		
CITY AND STATE		CONTRACT NUMBER
MATERIAL PROCURED UNDER A		
<input type="checkbox"/> DIRECT GOVERNMENT CONTRACT <input type="checkbox"/> SUBCONTRACT		
<p>1. HAS ANY PART OF THE SPECIFICATION CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE?</p> <p>A. GIVE PARAGRAPH NUMBER AND WORDING.</p>		
<p>B. RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES</p>		
2. COMMENTS ON ANY SPECIFICATION REQUIREMENT CONSIDERED TOO RIGID		
<p>3. IS THE SPECIFICATION RESTRICTIVE?</p> <p><input type="checkbox"/> YES <input type="checkbox"/> NO (If "yes", in what way?)</p>		
<p>4. REMARKS (Attach any pertinent data which may be of use in improving this specification. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity)</p>		
SUBMITTED BY (Printed or typed name and activity - Optional)		DATE

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