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Destructive tests on welds in metallic materials — Etchants for macroscopic and microscopic examination

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Contents

National foreword

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REPORT

RAPPORT

BERICHT

CR 12361

April 1996

Incorporates corrigendum August 1997

English version

Destructive tests on welds in metallic materials — Etchants for macroscopic and microscopic examination

Essais destructifs des soudures sur matériaux métalliques — Réactifs pour examen macroscopique et microscopique

Zerstörende Prüfung von Schweißnähten an metallischen Werkstoffen — Ätzungen für die makroskopische und mikroskopische Untersuchung

This CEN REPORT has been prepared by Technical Committee CEN/TC 121 'Welding' and has been approved by CEN on 1996-03-14.

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Foreword

This technical report was prepared by the technical committee CEN/TC 121, Welding, of which the secretariat is held by DS.

The technical committee agreed to publish this technical report.

Contents

1 Scope

This technical report gives a non-exhaustive list of etchants which can be used for the macroscopic and microscopic examination of welds in accordance with EN 1321 for the following materials:

– carbon and low alloy steels;
– stainless steels;
– nickel and nickel alloys;
– titanium and titanium alloys;
– copper and copper alloys;
– aluminium and aluminium alloys.

2 Normative references

This European technical report incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European technical report only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 1321 *Destructive tests on welds in metallic materials — Macroscopic and microscopic examination of welds*

3 General

Where details of concentration or waters of crystallization of reagents are not defined in the annexes, table 1 applies. These should be confirmed by the suppliers of each etchant.

Table 1. Characteristics of components

Components	**Characteristics**			
	Specific gravity g/cm^3	**Concentration** %	**Hydrate**	**Remarks**
HCl	1,18 1,16	35 to 38 31,5 to 33	—	
HF	1,13	40	—	
HNO_3	1,42	69	—	
H_2SO_4	1,84	98	—	
H_2O_2	—	6 % W/V[1)]	—	Usually 20 volumes (i.e. 20 volumes of available O_2)
H_3PO_4	1,70	85	—	
CH_3COOH	1,05	99,1	—	Glacial
HBF_4	1,23	35	—	
$C_2H_2O_4$	—	—	2	
$FeCl_3$	—	—	6	
$CuCl_2$	—	—	2	
$MgCl_2$	—	—	6	
$Fe(NO_3)_3$	—	—	9	

[1)] W/V means weight by volume.

4 Etchants for carbon and low alloy steels

The etchants for carbon and low alloy steels are given in annex A.

5 Etchants for stainless steels

The etchants for stainless steels are given in annex B.

6 Etchants for nickel and nickel alloys

The etchants for nickel and nickel alloys are given in annex C.

7 Etchants for titanium and titanium alloys

The etchants for titanium and titanium alloys are given in annex D.

8 Etchants for copper and copper alloys

The etchants for copper and copper alloys are given in annex E.

9 Etchants for aluminium and aluminium alloys

The etchants for aluminium and aluminium alloys are given in annex F.

10 Designation

Etchants should be designated either by names or by numbers of tables in accordance with annex G.

Annex A (informative)

Etchants for carbon and low alloy steels

See tables A.1 to A.13

Table A.1 Nital
Type of etchant Macroscopic and microscopic etchant
Composition in volume and in order of mixing 99 ml to 95 ml industrial methylated spirits* 1 ml to 5 ml nitric acid (HNO_3) * ethyl alcohol (C_2H_5OH), denatured with methyl alcohol (CH_3OH) Also methyl alcohol or isoamyl alcohol [$(CH_3)_2CH(CH_2)_2OH$]
Safe shelf life Indefinite
Surface preparation 600 grit or finer (macroetching ≈ 5 % of nitric acid) 3 μm diamond or finer (microetching ≈ 2 % of nitric acid)
Etching temperature Ambient
Etching time Few seconds, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Can increase strength to 15 % for macroetching on ground surface; reveals ferrite boundaries; differentiates ferrite from martensite. Good general purpose etchant. Amyl alcohol is preferable for galvanized steel

Table A.2 Picral (4 %)
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 100 ml ethyl alcohol (C_2H_5OH) 4 g picric acid [$C_6H_2OH(NO_2)_3$] [+ wetting agent (sodium dodecyl benzene sulfate) ($C_{18}H_{29}NaSO_3$) if required]
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time Few seconds, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Little attack prior austenite boundaries; detects carbides; good resolution with fine pearlite, martensite, tempered martensite and bainitic structures

Table A.3 Picric acid solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 1 l saturated aqueous picric acid [$C_6H_2OH(NO_2)_3$] 10 ml wetting agent (sodium dodecyl benzene sulfate) ($C_{18}H_{29}NaSO_3$)
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature 55 °C to 60 °C
Etching time Few seconds, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Reveals prior grain boundaries and segregation

Table A.4 Picral (15 %)
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 100 ml ethyl alcohol* (C_2H_5OH) 15 g picric acid [$C_6H_2OH(NO_2)_3$] * Also methyl alcohol (CH_3OH)
Safe shelf life Indefinite
Surface preparation 2 μm diamond or finer
Etching temperature Ambient
Etching time Few seconds to a minute, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Reveals general structure. Composition given will saturate the solution with picric acid

Table A.5 Hydrochloric picric acid solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 100 ml ethyl alcohol (C_2H_5OH) 1 ml hydrochloric acid (HCl) 4 g picric acid [$C_6H_2OH(NO_2)_3$]
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time 10 s to a few minutes
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Microstructural characterization of HAZ, weld and parent metal. Especially effective for very fine structures. Less effective than Nital for the ferrite grain boundaries

Table A.6 Ammonium peroxodisulfate solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 100 ml water 10 g ammonium peroxodisulfate [$(NH_4)_2S_2O_8$]
Safe shelf life Limited
Surface preparation 6 μm diamond or finer
Etching temperature Ambient
Etching time 2 min to 3 min
Additional precautions/requirements
Comments Reveals extent of HAZ and microscopic features of multipass welds

Table A.7 Alcoholic hydrochloric solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 100 ml ethyl alcohol (C_2H_5OH) 1 ml to 5 ml hydrochloric acid (HCl)
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature 40 °C to 50 °C
Etching time A few seconds to 1 min
Additional precautions/requirements Usual precautions for handling and disposal of acids. Add HCl to C_2H_5OH
Comments

Table A.8 120 / 10 / 30 etchant
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 120 ml ethyl alcohol (C_2H_5OH) or methyl alcohol (CH_3OH) 10 ml iron (III) chloride ($FeCl_3$) (60 % W/V) 30 ml hydrochloric acid (HCl)
Safe shelf life Indefinite
Surface preparation 2 μm diamond or finer
Etching temperature Ambient
Etching time Few seconds by immersion, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Good general purpose etchant

Table A.9 Cuprochloric solution 1
Type of etchant Macroscopic etchant
Composition in volume and in order of mixing 30 ml water (H_2O) 25 ml ethyl alcohol (C_2H_5OH) 40 ml hydrochloric acid (HCl) 5 g copper (II) chloride ($CuCl_2$)
Safe shelf life 2 h
Surface preparation 1000 grit or finer
Etching temperature Ambient
Etching time 10 s to 20 s
Additional precautions/requirements After the etching the specimen should be washed in order to remove copper deposits. Usual precautions for handling and disposal of acids
Comments Cold working strain lines

Table A.10 Magneso cuprochloric solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 100 ml ethyl alcohol (C_2H_5OH) 20 ml water (H_2O) 1 ml hydrochloric acid (HCl) 4 g magnesium (II) chloride ($MgCl_2$) 1 g copper (II) chloride ($CuCl_2$)
Safe shelf life 2 h
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time 60 s
Additional precautions/requirements Salts should be dissolved in the smallest amount of hot water, then diluted with ethyl alcohol. A slight polishing (1 μm) after the etching provides the best contrast
Comments Phosphorus and related segregations. The copper deposits first of all on the poorest concentrations of phosphorus

Table A.11 Adler's etchant
Type of etchant Macroscopic etchant
Composition in volume and in order of mixing 25 ml water (H_2O) 3 g ammonium tetrachlorodiaquocuprate (II) [$(NH_4)_2CuCl_4 \cdot 2H_2O$] 50 ml hydrochloric acid (HCl) 15 g iron (III) chloride ($FeCl_3$)
Safe shelf life Months
Surface preparation 320 grit or finer
Etching temperature Ambient
Etching time 5 s to 10 s
Additional precautions/requirements Add [$(NH_4)_2CuCl_4 \cdot 2H_2O$] to H_2O (a), add $FeCl_3$ to HCl (b). Mix both and add (b) to (a) afterwards
Comments

Table A.12 Heyn's etchant
Type of etchant Macroscopic etchant
Composition in volume and in order of mixing 120 ml water (H_2O) 10 g ammonium tetrachlorodiaquocuprate II [$(NH_4)_2CuCl_4 \cdot 2H_2O$]
Safe shelf life Months
Surface preparation 240 grit or finer
Etching temperature Ambient
Etching time 10 s to 1 min
Additional precautions/requirements Add [$(NH_4)_2CuCl_4 \cdot 2H_2O$] to H_2O
Comments Copper deposit shall be removed

Table A.13 Ferric chloride solution
Type of etchant Macroscopic etchant
Composition in volume and in order of mixing 70 ml water (H_2O) 30 ml iron (III) chloride ($FeCl_3$) (60 % W/V)
Safe shelf life Indefinite
Surface preparation 1000 grit or finer
Etching temperature Ambient
Etching time Few seconds
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments 1) swab the surface; 2) rinse with water; 3) swab the surface again; 4) after etching, water rinse, alcohol rinse, dry

Annex B (informative)
Etchants for stainless steels

See tables B.1 to B.12.

Table B.1 Oxalic acid solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 100 ml water (H_2O) 10 g oxalic acid (ethanedioic acid) ($C_2H_2O_4$)
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time Few seconds, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Electrolytic 2 V to 6 V; can reveal sensitivity to inter-crystalline corrosion; reveals general structure; reveals carbides at the grain boundaries

Table B.2 Thiocyanate solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 80 ml water (H_2O) 20 ml sulfuric acid (H_2SO_4) 10 g ammonium thiocyanate (NH_4SCN)
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time Few seconds, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Electrolytic 1,5 V to 2,0 V; good general purpose etchant

Table B.3 Acidified ferric chloride solution
Type of etchant Macroscopic etchant
Composition in volume and in order of mixing 480 ml water (H_2O) 120 ml hydrochloric acid (HCl) 50 g iron (III) chloride ($FeCl_3$)
Safe shelf life Indefinite
Surface preparation 600 grit or finer
Etching temperature Ambient
Etching time Few seconds to a minute, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Good general purpose macroscopic etchant for austenitic, chromium–nickel and other stainless steels

Table B.4 Modified Murakami's etchant
Type of etchant Macroscopic etchant
Composition in volume and in order of mixing 60 ml water (H_2O) 30 g potassium ferricyanide [$K_3Fe(CN)_6$] 30 g potassium hydroxide (KOH)
Safe shelf life Fresh solution
Surface preparation 1 μm diamond or finer
Etching temperature Temperature of freshly prepared solution (hot)
Etching time 20 s to 40 s
Additional precautions/requirements Usual precautions for handling and disposal of caustic solutions
Comments Reveals σ phase from δ ferrite and carbides; austenite matrix is not revealed

Table B.5 Cuprochloric solution 2
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 100 ml water (H_2O) 100 ml ethyl alcohol (C_2H_5OH) 100 ml hydrochloric acid (HCl) 5 g copper (II) chloride ($CuCl_2$)
Safe shelf life 2 h
Surface preparation 1 μm diamond or finer
Etching temperature Ambient
Etching time 10 s to few minutes
Additional precautions/requirements After the etching the test specimen should be washed in order to remove copper deposits. Usual precautions for handling and disposal of acids
Comments Especially for ferritic and martensitic stainless steels, as an alternative to electrolytic etching. Less effective for austenitic grain boundaries that can be attacked using longer etching times. Not effective for carbides

Table B.6 Chromic acid solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 100 ml water (H_2O) 10 g chromium (VI) oxyde (CrO_3)
Safe shelf life Few days
Surface preparation 1 μm diamond or finer
Etching temperature Ambient
Etching time 10 s to 1 min
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Austenitic grain boundaries; distinguishes σ phase and carbides. Electrolytic etching (3 V) for general purpose etchings. For the analysis of carbides, the etching will be carried out in two steps: first with 1 V and afterwards with 3 V

Table B.7 Alcoholic hydrochloric solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 100 ml ethyl alcohol (C_2H_5OH) 1 ml to 5 ml hydrochloric acid (HCl)
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature 40 °C to 50° C
Etching time Few seconds to one minute
Additional precautions/requirements Add HCl to C_2H_5OH. Usual precautions for handling and disposal of acids
Comments

Table B.8 Hydrochloric nitric acid solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 39 ml water (H_2O) 52 ml hydrochloric acid (HCl) 9 ml nitric acid (HNO_3) (concentration 65 %)
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time Few seconds to minutes
Additional precautions/requirements Always add HCl and HNO_3 to H_2O. Usual precautions for handling and disposal of acids
Comments

Table B.9 Adler's etchant
Type of etchant Macroscopic etchant
Composition in volume and in order of mixing 25 ml water (H_2O) 3 g ammonium tetrachlorodiaquocuprate (II) [$(NH_4)_2CuCl_4 \cdot 2H_2O$] 50 ml hydrochloric acid (HCl) 15 g iron (III) chloride ($FeCl_3$)
Safe shelf life Months
Surface preparation 320 grit or finer
Etching temperature Ambient
Etching time 5 s to 10 s
Additional precautions/requirements Add [$(NH_4)_2$ $CuCl_4 \cdot 2H_2O$ to H_2O] (a), add $FeCl_3$ to HCl (b), mix both and add (b) to (a) afterwards
Comments

Table B.10 Fluoronitric acid solution 1
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 50 ml nitric acid (HNO_3) 50 ml hydrofluoric acid (HF)
Safe shelf life Do not store after use
Surface preparation 2 μm diamond or finer
Etching temperature Ambient
Etching time 5 min to 30 min by immersion
Additional precautions/requirements Caution: HF! Wear hand and eye protection. In the event of spillage wash off skin immediately and seek medical advice. Use plastics receptacles. Usual precautions for handling and disposal of acids
Comments Reveals general structure

Table B.11 Fluoronitric acid solution 2
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 100 ml water (H_2O) 100 ml nitric acid (HNO_3) 100 ml hydrofluoric acid (HF)
Safe shelf life Do not store after use
Surface preparation 2 μm diamond or finer
Etching temperature Ambient
Etching time 1 min to 15 min by immersion
Additional precautions/requirements Caution: HF! Wear hand and eye protection. In the event of spillage wash off skin immediately and seek medical advice. Use plastics receptacles. Usual precautions for handling and disposal of acids
Comments Reveals grain boundaries

Table B.12 Nitric acid solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 50 ml water (H_2O) 50 ml nitric acid (HNO_3)
Safe shelf life Indefinite
Surface preparation 2 μm diamond or finer
Etching temperature Ambient
Etching time A few seconds
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Electrolytic at 3 V to 6 V. Rinse in the solution to remove the film present on the surface

Annex C (informative)
Etchants for nickel and nickel alloys

See tables C.1 to C.4.

Table C.1 Alcoholic hydrochloric acid solution with hydrogen peroxide
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 65 ml ethyl alcohol (C_2H_5OH) 35 ml hydrochloric acid (HCl) 4 ml hydrogen peroxide (H_2O_2) 20 volumes
Safe shelf life Do not store after use
Surface preparation 2 μm diamond or finer
Etching temperature Ambient
Etching time Few seconds, check by eye
Additional precautions/requirements Add H_2O_2 just before using. Usual precautions for handling and disposal of acids
Comments Good general purpose etchant

Table C.2 Thiocynate solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 80 ml water (H_2O) 20 ml sulfuric acid (H_2SO_4) 10 g ammonium thiocyanate (NH_4SCN)
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time A few seconds, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Electrolytic 1,5 V to 2,0 V. Good general purpose etchant.

Table C.3 Nitric acetic acid solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 30 ml acetone [$(CH_3)_2CO$] 30 ml nitric acid (HNO_3) 30 ml acetic acid (CH_3COOH)
Safe shelf life Do not store. Discard after use
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time Few seconds, check by eye
Additional precautions/requirements Keep cool. Nitrous oxides given off. Usual precautions for handling and disposal of acids
Comments Good general purpose etchant

Table C.4 Adler's etchant
Type of etchant Macroscopic etchant
Composition in volume and in order of mixing 25 ml water (H_2O) 3 g ammonium tetrachlorodiaquocuprate (II) [$(NH_4)_2CuCl_4 \cdot 2H_2O$] 50 ml hydrochloric acid (HCl) 15 g iron (III) chloride ($FeCl_3$)
Safe shelf life Months
Surface preparation 320 grit or finer
Etching temperature Ambient
Etching time 5 s to 10 s
Additional precautions/requirements Add [$(NH_4)_2CuCl_4 \cdot 2H_2O$] to H_2O (a), add $FeCl_3$ to HCl (b). Mix both and add (b) to (a) afterwards
Comments

Annex D (informative)
Etchants for titanium and titanium alloys

See tables D.1 and D.2.

Table D.1 Keller's etchant
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 950 ml water (H_2O) 25 ml nitric acid (HNO_3) 15 ml hydrochloric acid (HCl) 10 ml hydrofluoric acid (HF)
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time Few seconds, check by eye
Additional precautions/requirements Caution: HF! Wear hand and eye protection. In the event of spillage wash off skin immediately and seek medical advice. Use plastics receptacles. Usual precautions for handling and disposal of acids
Comments Good general purpose etchant

Table D.2 Fluoronitric acid solution 3
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 880 ml water (H_2O) 100 ml nitric acid (HNO_3) 20 ml hydrofluoric acid (HF)
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time Few seconds, check by eye
Additional precautions/requirements Caution: HF! Wear hand and eye protection. In the event of spillage wash off skin immediately and seek medical advice. Use plastics receptacles. Usual precautions for handling and disposal of acids
Comments Good general purpose etchant

Annex E (informative)

Etchants for copper and copper alloys

See tables E.1 to E.3.

Table E.1 Alcoholic acidified ferric chloride solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 960 ml industrial methylated spirits* 20 ml hydrochloric acid (HCl) 50 g iron (III) chloride ($FeCl_3$) *ethyl alcohol (C_2H_5OH) denatured with methyl alcohol (CH_3OH)
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time Few seconds, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Good general purpose etchant, especially for high copper alloys

Table E.2 Ammonium peroxodisulfate solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 90 ml water (H_2O) 10 mg ammonium peroxodisulfate [$(NH_4)_2S_2O_8$] 10 ml ammonium hydroxide (ammonia solution) (NH_3 in H_2O) specific gravity 0,880 g/cm^3
Safe shelf life Use fresh. Do not store after use
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time Few seconds, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of caustic solutions
Comments Good general purpose etchant

Table E.3 Nitric acid with ammonium and ferric nitrate solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 500 ml water (H_2O) 2 ml nitric acid (HNO_3) 2 g ammonium nitrate (NH_4NO_3) 20 g iron (III) nitrate [$Fe(NO_3)_3$]
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time Few seconds, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Good general purpose etchant

Annex F (informative)

Etchants for aluminium and aluminium alloys

See tables F.1 to F.7.

Table F.1 Sodium hydroxide solution
Type of etchant Macroscopic etchant
Composition in volume and in order of mixing 100 ml water (H_2O) 15 g sodium hydroxide (NaOH)
Safe shelf life Do not store. Discard after use
Surface preparation 600 grit or finer
Etching temperature Ambient
Etching time Few seconds, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of caustic solutions. Use in fume cupboard
Comments Good general purpose etchant. Can be used in various dilutions

Table F.2 Keller's etchant
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 950 ml water (H_2O) 25 ml nitric acid (HNO_3) 15 ml hydrochloric acid (HCl) 10 ml hydrofluoric acid (HF)
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time Few seconds, check by eye
Additional precautions/requirements Caution: HF! Wear hand and eye protection. In the event of spillage wash off skin immediately and seek medical advice. Use plastics receptacles. Usual precautions for handling and disposal of acids
Comments Good general purpose etchant. Warning: grain boundary attack may look like cracks

Table F.3 Hydrochloric nitric hydrofluoric acid solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 350 ml water (H_2O) 300 ml hydrochloric acid (HCl) 300 ml nitric acid (HNO_3) 50 ml hydrofluoric acid (HF)
Safe shelf life Is indicated by a change of colour to greenish brown and sluggish reaction
Surface preparation 3 μ diamond or finer
Etching temperature Ambient
Etching time 30 s to 60 s after start of reaction
Additional precautions/requirements Caution: HF! Wear hand and eye protection. In the event of spillage wash off skin immediately and seek medical advice. Use plastics receptacles. Usual precautions for handling and disposal of acids
Comments Warning: grain boundary attack may look like cracks

Table F.4 Hydrochloric nitric orthophosphoric acid solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 480 ml water (H_2O) 200 ml hydrochloric acid (HCl) 200 ml nitric acid (HNO_3) 120 ml orthophosphoric acid (H_3PO_4)
Safe shelf life Is indicated by a change of colour to greenish brown and sluggish reaction
Surface preparation 3 μm diamond or finer
Etching temperature 30 °C to 40 °C
Etching time 60 s to 120 s after start of reaction
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Warning: grain boundary attack may look like cracks

Table F.5 Barker's etchant
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 940 ml water (H_2O) 60 ml fluorobaric acid (HBF_4)
Safe shelf life Is stable normally for an unlimited period
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time 40 s to 60 s
Additional precautions/requirements See **F.1** for a description of the Barker procedure
Comments Warning: grain boundary attack may look like cracks For examination of grain and/or fibre structures of aluminium and aluminium alloys, the examination of the 'Barker' anodized samples under polarized light proved to be successful and produced the required result

Table F.6 Poulton's etchant
Type of etchant Macroscopic etchant
Composition in volume and in order of mixing 50 ml water (H_2O) 600 ml hydrochloric acid (HCl) 300 ml nitric acid (HNO_3) 50 ml hydrofluoric acid (HF)
Safe shelf life Do not store
Surface preparation 1000 grit or finer
Etching temperature Ambient
Etching time Few seconds, check by eye
Additional precautions/requirements Caution: HF! Wear hand and eye protection. In the event of spillage wash off skin immediately and seek medical advice. Use plastics receptacles. Usual precautions for handling and disposal of acids
Comments Reveal general macroscopic structure

Table F.7 Kroll's etchant
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 960 ml water (H_2O) 30 ml nitric acid (HNO_3) 10 ml hydrofluoric acid (HF)
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time Less than 5 s
Additional precautions/requirements Caution: HF! Wear hand and eye protection. In the event of spillage, wash off skin immediately and seek medical advice. Use plastics receptacles. Usual precautions for handling and disposal of acids
Comments Warning: grain boundary attack may look like cracks

F.1 Description of the Barker procedure

The barker anodic oxidation procedure will be used (see table F.5).

A schematic presentation of the method is given below.

Apparatus:

- *electrolyte* 6 volume % HBF_4 (35 weight %) in distilled water;
- *container* plastics;
- *anode* test specimen connected to an electrode by a good electrical conductor, e.g. aluminium wire;
- *cathode* pure aluminium (sheet);
- *voltage* 25 V to 30 V (d.c.) ;
- *time* 40 s to 60 s;
- *temperature* maximum ambient temperature (stirring or cooling of electrolyte bath).

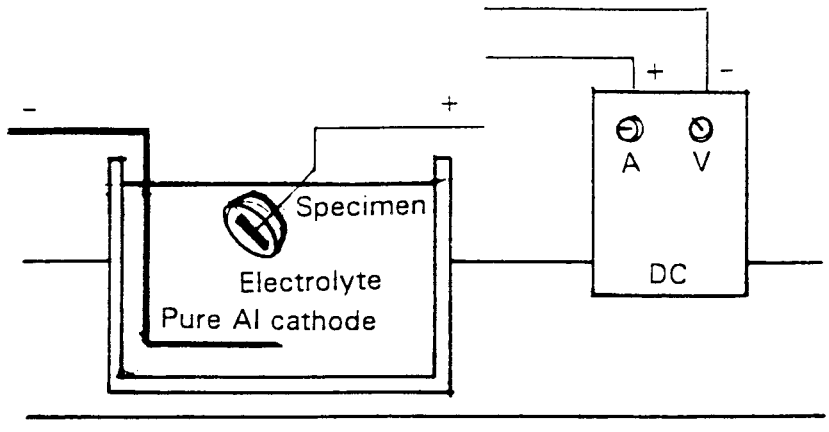
Treatment

After the anodic oxidation, wash the test specimen under running water and remove the water using alcohol. Dry in hot air.

Do not apply ultrasonic treatments.

Examination

Observe the oxidized surface with polarized light using a light microscope.



Annex G (informative)
List of etchants

See tables G.1 and G.2.

Table G.1 List of etchants classified by groups of materials

Name	Table	Type of etchant	Material
Adler's etchant	A.11	Macroscopic	Carbon and low alloy steels
Alcoholic hydrochloric solution	A.7	Microscopic	Carbon and low alloy steels
Ammonium peroxodisulfate solution	A.6	Microscopic	Carbon and low alloy steels
Cuprochloric solution 1	A.9	Macroscopic	Carbon and low alloy steels
Ferric chloride solution	A.13	Macroscopic	Carbon and low alloy steels
Heyn's etchant	A.12	Macroscopic	Carbon and low alloy steels
Hydrochloric picric solution	A.5	Microscopic	Carbon and low alloy steels
Magneso cuprochloric solution	A.10	Microscopic	Carbon and low alloy steels
Nital	A.1	Macroscopic and microscopic	Carbon and low alloy steels
Picral (4 %)	A.2	Microscopic	Carbon and low alloy steels
Picral (15 %)	A.4	Microscopic	Carbon and low alloy steels
Picric acid solution	A.3	Microscopic	Carbon and low alloy steels
120/10/30 etchant	A.8	Microscopic	Carbon and low alloy steels
Acidified ferric chloric solution	B.3	Macroscopic	Stainless steels
Adler's etchant	B.9	Macroscopic	Stainless steels
Alcoholic hydrochloric solution	B.7	Microscopic	Stainless steels
Chromic acid solution	B.6	Microscopic	Stainless steels
Cuprochloric solution 2	B.5	Microscopic	Stainless steels
Fluoronitric acid solution 1	B.10	Microscopic	Stainless steels
Fluoronitric acid solution 2	B.11	Microscopic	Stainless steels
Hydrochloric nitric acid solution	B.8	Microscopic	Stainless steels
Modified Murakami's etchant	B.4	Macroscopic	Stainless steels
Nitric acid solution	B.12	Microscopic	Stainless steels
Oxalic acid solution	B.1	Microscopic	Stainless steels
Thiocyanate solution	B.2	Microscopic	Stainless steels

Table G.1 List of etchants classified by groups of materials *(continued)*			
Adler's etchant	C.4	Macroscopic	Nickel and nickel alloys
Alcoholic hydrochloric with hydrogen peroxide	C.1	Microscopic	Nickel and nickel alloys
Nitric acetic acid solution	C.3	Microscopic	Nickel and nickel alloys
Thiocyanate solution	C.2	Microscopic	Nickel and nickel alloys
Keller's etchant	D.1	Microscopic	Titanium and titanium alloys
Fluoronitric solution 3	D.2	Microscopic	Titanium and titanium alloys
Alcoholic acidified ferric chloride solution	E.1	Microscopic	Copper and copper alloys
Ammonium peroxodisulfate solution	E.2	Microscopic	Copper and copper alloys
Nitric acid with ammonium and ferric nitrate	E.3	Microscopic	Copper and copper alloys
Barker's etchant	F.5	Microscopic	Aluminium and aluminium alloys
Hydrochloric nitric hydrofluoric acid solution	F.3	Microscopic	Aluminium and aluminium alloys
Hydrochloric nitric orthophosphoric acid solution	F.4	Microscopic	Aluminium and aluminium alloys
Keller's etchant	F.2	Microscopic	Aluminium and aluminium alloys
Kroll's etchant	F.7	Microscopic	Aluminium and aluminium alloys
Poulton's etchant	F.6	Macroscopic	Aluminium and aluminium alloys
Sodium hydroxide solution	F.1	Macroscopic	Aluminium and aluminium aloys

Table G.2 List of etchants by English alphabetical order

Name	**Table**	**Type of etchant** [1)]	**Material**
Acidified ferric chloride solution	B.3	A	Stainless steels
Adler's etchant	A.11	A	Carbon and low alloy steels
	B.9		Stainless steels
	C.4		Nickel and nickel alloys
Alcoholic acidified ferric chloride solution	E.1	I	Copper and copper alloys
Alcoholic hydrochloric acid with hydrogen peroxide	C.1	I	Nickel and nickel alloys
Alcoholic hydrochloric solution	A.7	I	Carbon and low alloy steels
	B.7		Stainless steels
Ammonium peroxodisulfate solution	E.2	I	Copper and copper alloys
Ammonium peroxodisulfate solution	A.6	I	Carbon and low alloy steels
Barker's etchant	F.5	I	Aluminium and aluminium alloys
Chromic acid solution	B.6	I	Stainless steels
Cuprochloric solution 1	A.9	A	Carbon and low alloy steels
Cuprochloric solution 2	B.5	I	Stainless steels
Ferric chloride solution	A.13	A	Carbon and low alloy steels
Fluoronitric acid solution 1	B.10	I	Stainless steels
Fluoronitric acid solution 2	B.11	I	Stainless steels
Fluoronitric acid solution 3	D.2	I	Titanium and titanium alloys

[1)] 'A' indicates macroscopic etching and 'I' indicates microscopic etching, in accordance with EN 1321.

Table G.2 List of etchants by English alphabetical order *(continued)*

Name	Table	Type of etchant [1)]	Material
Heyn's etchant	A.12	A	Carbon and low alloy steels
Hydrochloric nitric acid solution	B.8	I	Stainless steels
Hydrochloric nitric hydrofluoric acid solution	F.3	I	Aluminium and aluminium alloys
Hydrochloric nitric orthophosphoric acid solution	F.4	I	Aluminium and aluminium alloys
Hydrochloric picric acid	A.5	I	Carbon and low alloy steels
Keller's etchant	D.1	I	Titanium and titanium alloys
	F.2		Aluminium and aluminium alloys
Kroll's etchant	F.7	I	Aluminium and aluminium alloys
Magneso cuprochloric solution	A.10	I	Carbon and low alloy steels
Modified Murakami's etchant	B.4	A	Stainless steels
Nital	A.1	A and I	Carbon and low alloy steels
Nitric acid solution	B.12	I	Stainless steels
Nitric acetic acid solution	C.3	I	Nickel and nickel alloys
Nitric acid with ammonium and ferric nitrate	E.3	I	Copper and copper alloys
Oxalic acid solution	B.1	I	Stainless steels
Picral (4 %)	A.2	I	Carbon and low alloy steels
Picral (15 %)	A.4	I	Carbon and low alloy steels
Picric acid solution	A.3	I	Carbon and low alloy steels
Poulton's etchant	F.6	A	Aluminium and aluminium alloys
Sodium hydroxide	F.1	A	Aluminium and aluminium alloys
Thiocyanate solution	B.2 C.2	I	Stainless steels Nickel and nickel alloys
120/10/30 etchant	A.8	I	Carbon and low alloy steels

[1)] 'A' indicates macroscopic etching and 'I' indicates microscopic etching, in accordance with EN 1321.

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