INTERNATIONAL STANDARD

ISO 14104

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Gears — Surface temper etch inspection after grinding

Engrenages — Contrôle par attaque chimique des zones revenues lors de la rectification

Reference number
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Contents

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 14104 was prepared by The American Gear Manufacturers Association (AFMA) (as ANSI/AGMA 2007-B92) and was adopted, under a special "fast-track procedure", by Technical Committee ISO/TC 60, *Gears*, in parallel with its approval by the ISO member bodies.

The AGMA metallurgic committee undertook a revision of the former temper etch procedure (AGMA 230.01-1967) in 1989. It was renamed "Surface temper etch inspection after grinding" for better definition, the contents reorganized, and obsolete procedures deleted. The revision AGMA 230.01 was redesignated AGMA 2007-B92, and was approved by the AGMA membership in February 1992.

An industry-wide survey was conducted to establish common solutions in time that were acceptable to the greatest number of users. The safety and environmental precautions were included therein for those not familiar with storage, handling, use and disposal of concentrated acids, alkalis and solvents. These precautions, however, do not supersede the latest applicable requirements.

Introduction

This International Standard explains the materials and procedures necessary to determine, evaluate and describe localized overheating on ground surfaces. A system to describe and classify the indications produced during this inspection is included. However, specific acceptance or rejection criteria are not contained.

Gears — Surface temper etch inspection after grinding

1 Scope

This International Standard specifies standard procedures and requirements for the detection and classification of localized overheating on ground surfaces by chemical etch methods.

Some methods which have been used in the past are no longer recommended. Specifications should be changed to use the methods in this International Standard. These etching methods are more sensitive to changes in surface hardness than most hardness testing methods.

This International Standard applies to steel parts such as gears, shafts, splines and bearings, but is not applicable to nitrided parts and stainless steels.

NOTE 1 This process, although at times called "nital etch", should not be confused with other processes also known as "nital etch".

The surface temper etch procedure is to be performed after grinding and before additional finishing operations.

2 Equipment

2.1 Container materials

Container materials shall not react with the solutions contained, nor damage the parts to be processed. All containers shall be labelled with the solution contained and covered when not in use.

2.2 Inspection area

The area to be inspected shall be sufficiently illuminated to be free of shadows and reflections. Light capable of ensuring 3 200 lux (300 foot candles) at the inspection level is recommended.

2.3 Timing device

A suitable timing device shall be used for the uniform processing of all parts in a group.

2.4 Cleaner

An alkaline cleaner, vapour degreaser, solvent wash, or equivalent cleaning process should be used.

3 Reagents

All chemicals shall be technical grade or better.

3.1 Cleaning materials

Cleaning materials shall be used which are capable of ensuring removal of all dirt, grit, grease and oil, to obtain a "water break" free surface. A "water break" free surface is one which maintains a continuous water film for a minimum period of 15 s after having been rinsed in clean water at a temperature below 40 °C.

3.2 Nitric acid, ρ = 1,42 g/ml.

3.3 Hydrochloric acid, ρ = 1,19 g/ml.

3.4 Alcohol

Methanol or denaturated ethanol, clean and free of contaminants such as oil, should be used.

3.5 Water, clean and free of contaminants.

3.6 Alkaline solution

A solution such as 4 % to 6 % sodium hydroxide in water with a minimum pH value of 10, or 13 % to 17 % ammonium hydroxide in alcohol, should be used.

3.7 Rust-preventive oil, which does not mask the results of etching.

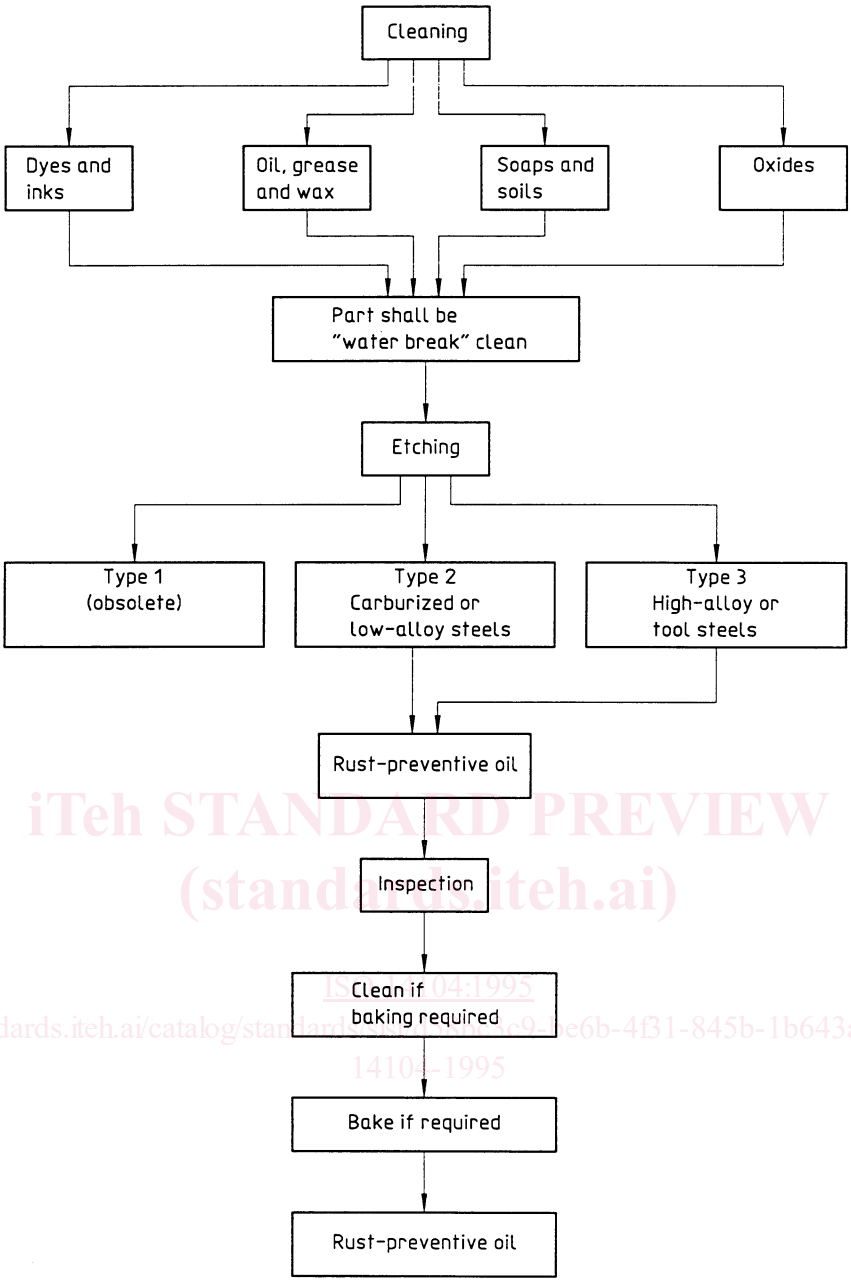

Figure 1 — Procedure flowchart

4 Procedure

As shown in figure 1, first clean the part (see 4.1 and table 1) then etch it using one of the procedures shown in either table 2 or 3, as appropriate for the type of steel being inspected. Unless otherwise specified, selection of the specific procedure shall be at the supplier's option.

4.1 Cleaning

Proper cleaning is mandatory for parts to be etched and inspected. Satisfactory cleaning will be evidenced by the absence of "water breaks" on the cleaned parts when rinsed in water. The cleaning procedure shall be chosen by the supplier. The exact method depends on the type of contaminant present. Table 1 lists contaminants and corresponding methods of cleaning in common use.

It should be noted that additional processes may be required to remove residues from the parts prior to etching. Thorough cleaning of parts prior to etching is imperative in order to obtain good results. Improper cleaning will result in non-uniform discoloration and staining, which may lead to difficult interpretation of etching results. Typical (recommended) cleaning procedure consists of:

a) vapour degreasing or solvent cleaning;

b) abrasive cleaning: select particle size and blasting procedure to maintain surface finish and dimensions; handle parts with clean white gloves;

c) alkaline or ultrasonic clean; examine for water breaks and repeat if water break occurs.

Table 1 — Examples of cleaning methods

Type of contaminant	Method of cleaning
Dyes and inks	Alcohol, methyl ethyl ketone, or equivalent
Oil and grease	Vapour degreasing
Soaps	Alkaline cleaner (60 °C to 80 °C) or ultrasonic cleaner

4.2 Etching

The following etching techniques are effective preparation for surface temper inspection. The type of etching should be chosen based on the material to be inspected and the ease of handling.

Type 2 etching (see table 2) is generally accepted as a good production inspection method. Type 2 is normally used for inspecting carburized steels and can also be used for surface-hardened areas of through-hardened steels. Type 3 (see table 3) is normally used for inspecting tool and high-alloy steels, and can be used in place of Type 2. When the Type 3 etching procedure is used, abrasive cleaning is not required.

Parts which cannot be immersed in tanks can be etched using cotton swabs, using the same materials and techniques described either in table 2 or 3. The swabbing technique is difficult to control and interpret, therefore, tank immersion is preferred where practical. Use of a swabbing technique should be agreed upon by the customer and supplier.

Inspect parts immediately following the complete etch procedure.

An elevated-temperature bake to relieve hydrogen embrittlement is optional. The maximum bake temperature shall be at least 14 °C below the final heat-treat tempering temperature. A baking time of 2 h to 4 h is recommended.

Table 2 — Type 2 etching

Step [1]	Process	Solution [2]	Recommended time [3]	Remarks
1	Nitric acid etch [4]	Nitric acid, 3 % to 5 % (by volume), in alcohol: ..... in water:	30 s to 60 s 10 s to 30 s	Exact time to develop black oxide film will vary; time should be established and reproduced
2	Rinse	Water	As required	To remove acid
3	Alcohol dip [5]	Alcohol	Dip and dry	To remove water
4	Bleach [3]	Hydrochloric acid, 4 % to 6 % (by volume), in alcohol: ..... in water:	30 s to 60 s	Part should be immersed for a sufficient time to cause a *uniform brownish-grey colour* on part; exact bleaching time should be established by test and reproduced
5	Rinse	Water	As required	To remove acid
6	Neutralize	Alkali solution with pH of 10 minimum	10 s to 60 s	Agitate parts while immersed
7	Rinse	Water	As required	To remove caustic solvents
8	Alcohol dip [5]	Alcohol	Dip and dry	To remove water
9	Oil	Rust preventive	Dip only	To prevent corrosion and aid in colour contrast

1) Uniform agitation of the parts while immersed in the respective baths and rinses is required to avoid a spotty etching condition as well as to accomplish complete neutralization.

2) All solutions are used at ambient temperature.

3) It is permissible to deviate from these recommended times.

4) Areas with close tolerances which do not require surface temper etch inspection should be suitably masked to avoid stock removal. Approximately 0,003 mm of stock per surface is removed by etching each time this process is performed.

5) Optional procedure: hot water rinse at 65 °C minimum, followed by a dry air blast, may be used in lieu of alcohol dip.

Table 3 — Type 3 etching

Step [1]	Process	Solution [2]	Recommended time [3]	Remarks
1	Hydrochloric acid clean [4]	Hydrochloric acid, 4 % to 6 % (by volume), in alcohol: ..... in water:	1,5 min to 3,5 min 30 s to 60 s	Exact time to be established by test
2	Rinse	Water	As required	To remove acid
3	Alcohol dip [5]	Alcohol	Dip and dry	To remove water
4	Nitric acid etch [4]	Nitric acid, 3 % to 5 % (by volume), in alcohol: ..... in water:	1,5 min to 3,5 min 30 s to 60 s	Exact time to develop black oxide film will vary; time should be established and reproduced
5	Rinse	Water	As required	To remove acid
6	Alcohol dip [5]	Alcohol	Dip and dry	To remove water
7	Bleach [3]	Hydrochloric acid, 4 % to 6 % (by volume), in alcohol: ..... in water:	1,5 min to 3,5 min 30 s to 60 s	Part should be immersed for a sufficient time to cause a *uniform brownish-grey colour* on the part; exact bleaching time should be established by test and reproduced
8	Rinse	Water	As required	To remove acid
9	Neutralize	Alkali solution with pH of 10 minimum	10 s to 60 s	Agitate parts while immersed
10	Rinse	Water	As required	To remove caustic solvents
11	Alcohol dip [5]	Alcohol	Dip and dry	To remove water
12	Oil	Rust preventive	Dip only	To prevent corrosion and aid in colour contrast

1) Uniform agitation of the parts while immersed in the respective baths and rinses is required to avoid a spotty etching condition as well as to accomplish complete neutralization.

2) All solutions are used at ambient temperature.

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4) Areas with close tolerances which do not require surface temper etch inspection should be suitably masked to avoid stock removal. Approximately 0,003 mm of stock per surface is removed by etching each time this process is performed.

5) Optional procedure: hot water rinse at 65 °C minimum, followed by a dry air blast, may be used in lieu of alcohol dip.

5 Inspection criteria

5.1 Visual appearance and classification

The appearances of tempering indications are described in table 4. Parts with no temper indications will be uniform grey in colour when properly cleaned and etched. Localized tempered areas appear as dark grey or black areas on the etched part. Generally, the severity of temper burns increases as the colour becomes darker. If sufficient heat is generated during grinding, rehardening may result. The rehardened area will contain an area of white or light-coloured untempered martensite, and may be surrounded by a black tempered area.

Any indications, especially rehardening burns (i.e. untempered martensite) may affect the durability of the part, but the part may still be functional.

It is good practice to inspect parts with temper indications by magnetic particle inspection, especially those with class D or E indications, which are more susceptible to cracking.

The classification system given in table 4 should be used to develop the acceptance or rejection criteria.

It is recommended that users of this International Standard set their own reference standards.

5.2 Surface hardness effects

These etching methods detect surface hardness changes more readily than most hardness testing. Experience warrants reduction in allowable stress number (contact), $\sigma_{H\,lim}$, values for carburized surfaces with localized tempering. When possible, inspection of tempered areas should be performed using microhardness testing methods. Because of differences in hardness testing methods, the equipment type, loads and conversion charts used should be reported. There are some portable microhardness testers available that will allow hardness testing without damaging parts. However, their proper use is essential to ensure accurate hardness readings. Some difficult-to-reach areas may also be inaccessible to this equipment.

Table 4 — Surface temper classification system

Prefix code		
F = Functional surfaces; includes flanks, ground roots, bearing journals and, if specified, other areas. N = Non-functional surfaces; includes all other ground surfaces.		
Class code [1]		
Class	**Description**	**Visual appearance, worst area**
A B (C) (Obsolete class) D E	No tempering Light tempering (Moderate tempering) Heavy tempering Rehardening Severe overheating	Uniform grey colour Norrow (light) indications (No longer used) Wide (dark) indications White area surrounded by black indications
Suffix code		
Level	**Maximum percentage of surface area affected** [1]	
1 2 3	10 % 25 % Unrestricted	
1) Measured on a single surface such as a tooth flank.		
NOTE — Sample classification callouts are as follows: **FA/NB2:** No tempering is allowed on any functional surface, but light tempering on up to 25 % of the area of the worst single non-functional surface, such as a shoulder, is permitted. **FB1/ND2** **FB2:** Implies no restrictions on non-functional surfaces. **FB3/FD2/ND3:** Light tempering of an unrestricted amount and heavy tempering on up to 25 % of the area of the worst single functional surface, such as a single tooth flank, is permitted. In addition, heavy tempering of an unrestricted amount is permitted on non-functional surfaces.		

False indications which may be caused by smears, stains, rust, or other differences in surface conditions shall require cleaning and retesting. Since repeated etching may result in appreciable metal removal, care shall be exercised to ensure that close tolerance dimensions are maintained. Stains may often be distinguished from actual temper indications since stains can be wiped off almost entirely. Temper indications will remain darker than the surrounding area even after wiping. It should be noted that these etching procedures may be used to discover other hardness differences such as those due to chemistry variation, spotty or non-carburized areas.

6 Rework of surface-tempered parts

If stock permits, finish ground parts found not to be acceptable by surface temper inspection may be reworked. Permission to rework parts may be required by the customer.

Magnetic particle inspection is required before and after rework operations.

Controlled shot peening may reduce some of the detrimental effects caused by abusive grinding. Use of shot peening on surface-tempered areas shall be agreed upon by the customer and supplier.

7 Temper etch removal

If desired, etch discoloration may be removed for cosmetic purposes by a standard electrolytic alkaline cleaner, vapour honing, polishing or glass-bead cleaning. Removal may cause associated stock removal or surface texture changes. Discoloration, however, has no deleterious effects upon operation.

8 Maintenance and control

To check solution performance, it is recommended that parts with known temper indications be etched, as required, dependent on solution usage, or when new solutions are made. After etching and inspection, the temper etch results shall be removed from the sample as indicated in clause 7. The sample should then be protected against rust. Such samples should be replaced periodically.

The solutions shall be checked for concentration and contamination at regular intervals, dependent on usage, and appropriately documented. The preferred method of inspecting acid solutions in alcohol is by alkali titration.

9 Safety and environmental precautions

The following are recommended safety and environmental precautions.

a) Concentrated and dilute acids and alkalis can be dangerous. If any contact with the body occurs, flood immediately with cold water. Seek physician for medical attention.

b) Avoid contact with solvents such as methyl ethyl ketone (MEK), as serious skin "burn" may result. Use with adequate ventilation.

c) Always add the acid to water or alcohol. Do not add water or alcohol to acid.

d) Keep tanks covered when not in use.

e) Protective gloves, face shield and apron should be worn at all times when mixing or working with acidic and alkaline chemicals.

f) Comply with occupational safety and health regulations covering training, material safety data sheets, storage and labelling of containers and tanks.

g) Comply with regulations covering permissible exposure limits for all acids, chemicals and by-products.

h) Ventilation should be in accordance with applicable regulations.

i) Comply with regulations on flammable and combustible liquids, insurance, and fire-protection standards covering storage and use of flammable solvents.

j) Government regulations for hazardous waste and transportation of chemicals should be observed.

k) Community right-to-know and reporting regulations shall be observed.