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Gears — Surface temper etch inspection after grinding, chemical method

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

Reference number
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Contents

Page

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 60, *Gears*, Subcommittee SC 2, *Gear capacity calculation.*

This third edition cancels and replaces the second edition (ISO 14104:2014), which has been technically revised. The changes of the corrected version have been incorporated as well as adoptions in the cleaning method of etching procedures shown in Tables 2 and 3.

Introduction

This document 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.

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.

Gears — Surface temper etch inspection after grinding, chemical method

1 Scope

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

The process described in this document is typically used on ground surfaces; however, it is also useful for the detection of surface anomalies that result from post-heat treatment machining such as hard turning, milling and edge breaking (deburring) processes. Surface metallurgical anomalies caused by carburization or decarburization are also readily detectable with this process.

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

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

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

The surface temper etch procedure is performed after grinding and before additional finishing operations such as superfinishing, shot peening and honing.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Apparatus

4.1 Container materials.

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

4.2 Inspection area.

The area to be inspected shall be sufficiently illuminated to be free of shadows and reflections. A minimum light intensity of 2 200 lx (~200 foot candles) at the inspection level is recommended.

4.3 Cleaner.

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

4.4 Timing device.

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

4.5 Grey scale reference.

A suitable grey scale reference should be used, such as Tiffen Color Separation Guide and Gray Scale Q13 (small) or Q14 (large)[1].

Figure 1 is an example of a suitable grey scale reference. Use of a sample part with known indications to exhibit surface tempering is also recommended.

Figure 1 is an approximate reproduction of a commercially available grey scale. The reproduction shown in this image is not accurate due to variance in graphic reproduction quality and is provided for reference only. It shall not be used for inspection in conformity with this document.

The grey scale card should be replaced at regular intervals.

Figure 1 — Gray scale card (reprinted with the permission of The Tiffen Company)

5 Reagents

All chemicals shall be technical grade or better.

5.1 Cleaning materials, which ensure 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.

5.2 Nitric acid.

See Tables 2 and 3.

5.3 Hydrochloric acid.

See Tables 2 and 3.

1) Tiffen Gray Scale is the trade name of a product supplied by Tiffen. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

5.4 Alcohol, methanol or denatured ethanol, clean and free of contaminants such as oil.

5.5 Water, clean and free of contaminants. Distilled water preferred, but not required.

5.6 Alkaline solution, a solution such as 4 % to 6 % sodium hydroxide in water with a minimum pH value of 10 or equivalent.

5.7 Rust-preventive oil, suitable for displacing water and which does not mask the results of etching.

6 Procedure

6.1 General

As shown in Figure 2, clean the part first (see 6.2 and Table 1) then etch it using one of the procedures shown in either Table 2 or Table 3, whichever is appropriate for the type of steel being inspected. Unless otherwise specified, selection of the specific procedure shall be at the supplier's option.

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

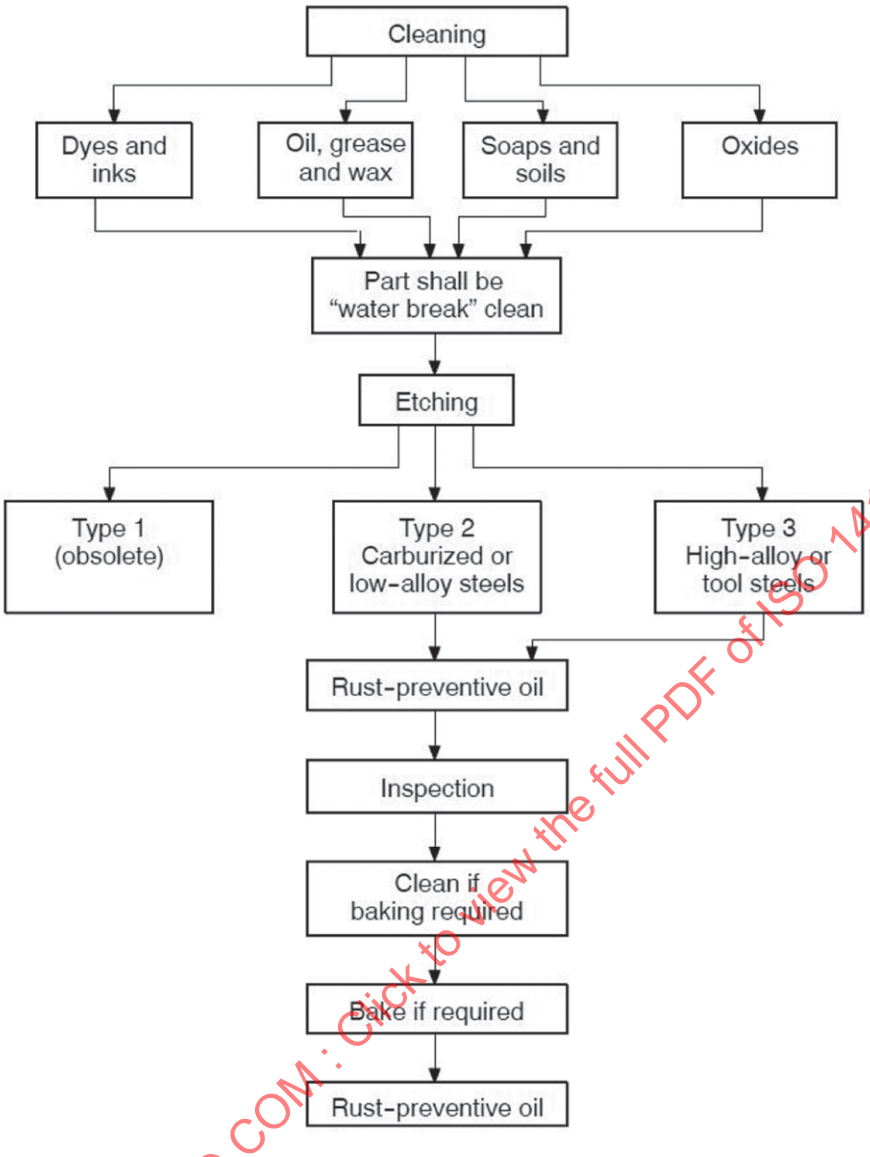


Figure 2 — Procedure flow chart

6.2 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 clean parts after immersion 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.

Additional processes can 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 can lead to difficult interpretation of etching results. A typical (recommended) cleaning procedure consists of the following:

— vapour degreasing or solvent cleaning;

— abrasive cleaning: select particle size, media and blasting procedure to maintain surface finish and dimensions; avoid damage and handle parts with clean white gloves;

— alkaline or ultrasonic clean;

— rinsing in water and examining for water breaks after cleaning. If water break occurs, re-clean and re-examine for water break until water break free.

Table 2 — Type 2 etching

Step[a]	Process	Solution[b]	Recommended time[c]	Remarks
1	Nitric acid etch[d] Grey scale Tank 7(M) to 11, Swab 7(M) to 15	Nitric acid, 1,5 % 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 or alcohol	As required	Removes acid
3	Alcohol dip[e]	Alcohol	Dip	Removes water
4	Bleach[d] Grey scale Tank 6 to 10, Swab 2 to 10	Hydrochloric acid, 2 % 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 the part; exact bleaching time should be established by test and reproduced.
5	Rinse	Water	As required	Removes acid
6	Neutralize	Alkali solution with pH of 10 minimum	10 s to 60 s	Agitate parts while immersed
7	Rinse	Water	As required	Removes caustic solvents
8	Dry[f,g]	Alcohol or hot water	Dip and dry	Removes water
9	Oil	Rust-preventive	As required	Prevents corrosion and aids in colour contrast

[a] 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. Multiple rinses can be used in steps 2, 5 and 7.

[b] All solutions are used at ambient temperature.

[c] It is permissible to deviate from these recommended times.

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

[e] Step 3 is needed only if step 2 is in water and step 4 will be in alcohol.

[f] Typical procedures include alcohol dip or hot water rinse at 65 °C minimum, followed by contaminant-free air blast.

[g] Step 8 is not mandatory if in step 9 a water-displacing oil is used.

Table 3 — Type 3 etching

Step[a]	Process	Solution[b]	Recommended time[c]	Remarks
1	Hydrochloric acid clean[d]	Hydrochloric acid, 2 % 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 or alcohol	As required	Removes acid
3	Alcohol dip[e]	Alcohol	Dip	Removes water
4	Nitric acid etch[d] Grey scale 11 to 15	Nitric acid, 1,5 % 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 or alcohol	As required	Removes acid

Table 3 *(continued)*

Step[a]	Process	Solution[b]	Recommended time[c]	Remarks
6	Alcohol dip[e]	Alcohol	Dip	Removes water
7	Bleach[d,e] Grey scale 2 to 6	Hydrochloric acid, 2 % 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	Removes acid
9	Neutralize	Alkali solution with pH of 10 minimum	10 s to 60 s	Agitate parts while immersed
10	Rinse	Water	As required	Removes caustic solvents
11	Dry[f,g]	Alcohol or hot water	Dip and dry	Removes water
12	Oil	Rust-preventive	As required	Prevents corrosion and aids in colour contrast

[a] 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. Multiple rinses can be used in steps 2, 5, 8 and 10.

[b] All solutions are used at ambient temperature.

[c] It is permissible to deviate from these recommended times.

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

[e] Step 3 is needed only if step 2 is in water and step 4 will be in alcohol. Likewise, step 6 is needed only if step 5 is in water and step 7 will be in alcohol.

[f] Typical procedures include alcohol dip or hot water rinse at 65 °C minimum, followed by contaminant-free air blast.

[g] Step 11 is not mandatory if in step 12 a water-displacing oil is used.

6.3 Etching

Use of a suitable grey scale reference is recommended to quantify the interpretation of temper indications. Figure 1 is an example of a commercially available grey scale reference. Use of a sample part with known indications to exhibit surface tempering is also recommended. The use of a grey scale card is required in order to achieve the goal of improved reproducibility of results over time and between inspectors or laboratories, independent of the specific processing variations allowed by this document.

The suggested ranges for chemical concentrations, dwell times and grey shades are not mandatory for conformance with this document provided that there is evidence that the same surface temper classification (see Table 4) results as if the recommended ranges were used. Ranges for the shades of grey are suggested rather than maximums and minimums in order to avoid processing that results in shades of grey that are too light or too dark.

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[a]		
Class	**Description**	**Visual appearance, worst area**
A	No tempering	Uniform grey colour
B	Light tempering	Narrow (light) indications
(C) (Obsolete class)	(Moderate tempering)	(No longer used)
D	Heavy tempering	Wide (dark) indications
E	Rehardening (Severe overheating)	White area surrounded by black indications
Suffix code[b]		
Level	**Maximum percentage of surface area affected**[a]	
1	10 %	
2	25 %	
3	Unrestricted	

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: Light tempering is allowed on up to 10 % of the area of the worst single functional surface. In addition, heavy tempering on up to 25 % of the area of the worst single non-functional surface, such as a shoulder, is permitted.

— FB2: Light tempering is allowed on up to 25 % of the area of the worst single functional surface. In addition, it 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.

[a] Measured on a single surface such as a tooth flank.

[b] Not applicable to Class A.

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.

The type 1 (hot bleach) etching method that is sometimes described in older publications is obsolete and not covered in this document.

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. Occasionally, a part might not respond to step 1, in which case, with engineering approval, consider pre-cleaning as per Table 3, steps 1 to 3.

Type 3 (see Table 3) is normally used for inspecting tool steels and alloy steels, which do not respond to simple nitric acid etching and require a pre-etch using hydrochloric acid, and can be used in place of type 2. Type 3 etching procedure should be used when grey scale level cannot be developed by nitric acid in step 1 of the type 2 procedure.

Parts which cannot be immersed in tanks can be etched using other techniques such as swabbing or spraying, using the procedures described in Tables 2 or 3. Use of a non-immersion technique should be agreed upon by the customer and supplier.

Inspect parts immediately following the complete etch procedure. When using the swab or spray technique, there is a risk of splashing adjacent surfaces such as teeth and critical bearing journals. Therefore, appropriate protective measures should be used.

An elevated-temperature bake to relieve hydrogen embrittlement is not required by this document, but can be recommended or required to comply with other specifications. One process that can be used is SAE AMS 2759/9. If used, the maximum bake temperature shall be at least 14 °C below the final heat-treat tempering temperature in lieu of the temperatures stated in SAE AMS 2759/9.

CAUTION — Post surface temper etch inspection bake can also serve as a post-machining stress relief prior to subsequent plating processes (such as cadmium or silver plated). In the event that a rejectable indication is detected and requires rework, the post etch bake should not be performed until an acceptable etch is achieved. Baking could make the remnant of reworked indication(s) harder to detect after the bake and rework. This can conflict with bake delay time requirements.

7 Inspection criteria

7.1 Visual appearance and classification

The appearance of tempering indications is 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 can result. The rehardened area will contain an area of white or light-coloured untempered martensite surrounded by a black tempered area.

False indications which can be caused by smears, stains, rust or other differences in surface conditions shall require cleaning and retesting unless it can be proven that these are false indications. Since repeated etching can result in appreciable metal removal, care shall be exercised to ensure that close tolerance dimensions are maintained. Stains can 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.

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

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

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

It is recommended that users of this document establish their own reference standards. See Clause 11.

7.2 Surface hardness effects

These etching methods can detect surface hardness changes more readily than most hardness testing. Experience can warrant reduction in allowable stress numbers (contact), $\sigma_{H,lim}$, values for carburized surfaces with localized tempering. When possible, inspection of areas with indications can be performed using suitable hardness testing methods to supplement surface temper etch results. Due to differences in hardness test 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, proper use of any hardness tester is essential to ensure accurate hardness readings and that functional surfaces are not damaged. Some areas can be inaccessible to hardness testing equipment.

8 Temper etch discoloration removal

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

9 Rework of surface-tempered parts

If gear metallurgy, stock (case-hardened layer and tooth thickness tolerances) and design permit, finish ground parts found not to be acceptable by surface temper inspection can be reworked. Permission to rework parts can be required by the customer. After rework, parts shall be rechecked. An etching procedure is recommended.

Magnetic particle inspection is recommended before and after rework operations.

Regrinding, refinishing and controlled shot peening, singularly or in combination, can reduce some of the detrimental effects caused by grinding-induced tempering. Use of shot peening on surface-tempered areas shall be agreed upon by the customer and supplier.

10 Operator qualification

Test personnel should be qualified for visual testing (VT) in accordance with ISO 9712 or ASNT-TC-1A.

11 Maintenance and control

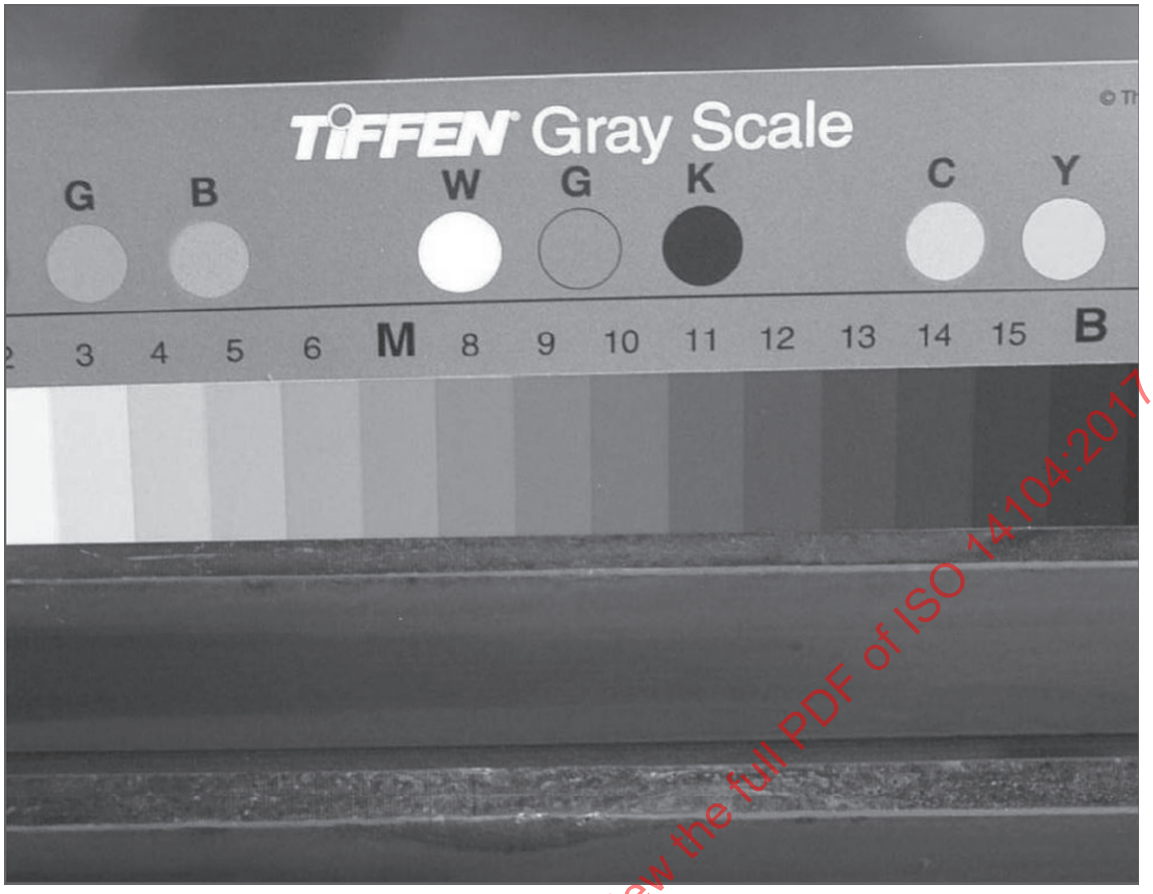
To check solution performance, it is recommended that parts with known temper indications be etched, as required, dependent on solution usage or when new solutions are made. After etching and inspection, the temper etch results shall be removed from the sample as indicated in Clause 8. The sample should then be protected against rust. Such samples should be replaced periodically.

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

It is recommended that users of this document use a sample part with grinding temper and rehardening indications as a calibration and verification aid. The sample part should be cleaned with an abrasive pad with non-metallic fibres.

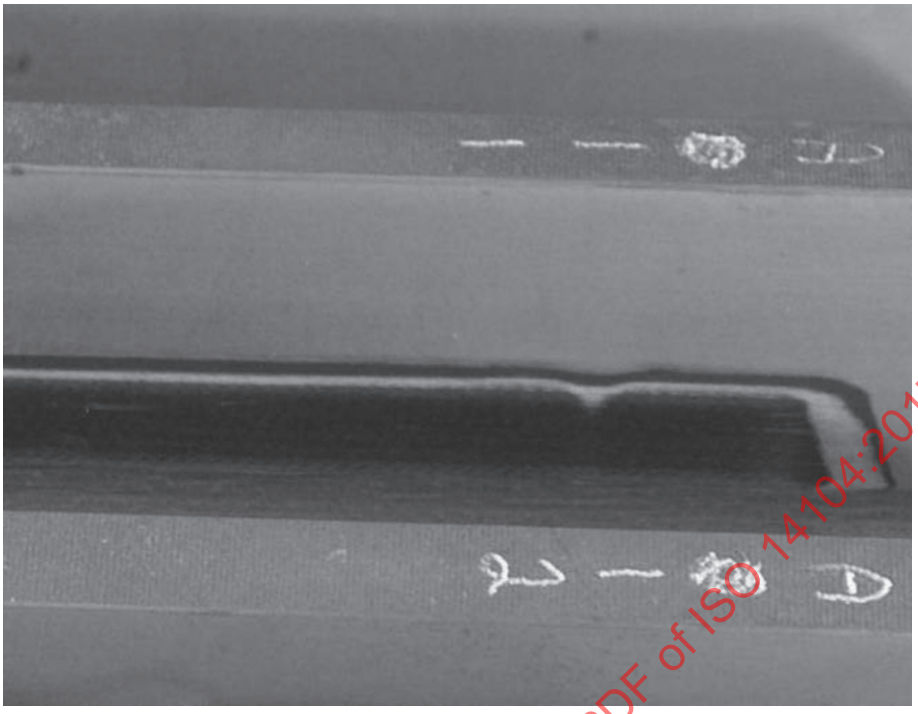
WARNING — Abrasive pads with metallic fibres can cause corrosion.

If the results using the same procedure are the same shade of grey as previously observed for the same part when previously etched, the operator can proceed. A photograph of the same part previously etched is a useful reference document. If there is a difference, the baths should be rechecked and corrected before the operator can proceed. See Figures 3 to 13 for sample photographs.



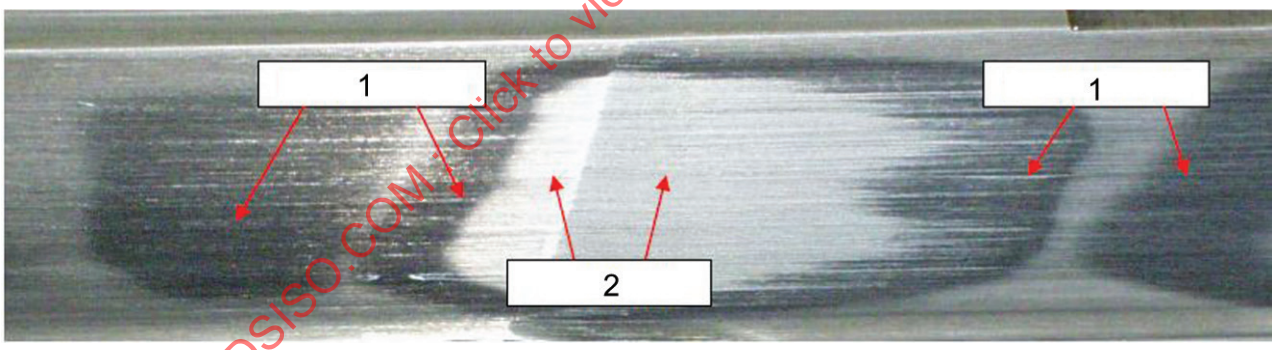
NOTE Grey scale is used for comparison of subtle colour changes across the width of the tooth from the left-hand region that is dark grey (FD) in the root area of the tooth. Surfaces not tempered appear uniform grey in colour.

Figure 3 — Example of using grey scale for progressively heavier tempering, Class FD



NOTE This shows progressive overheating in dedendum area near root area. Rehardened areas (FE) appear as white etching areas with dark grey etching overheated areas (FD) surrounding the white etching areas.

Figure 4 — Progressive tempering and rehardening (severe overheating), Class FD and Class FE



Key

1 heavy tempering

2 rehardening

Figure 5 — Class FE rehardening/severe overheating with adjacent Class FD heavy tempering

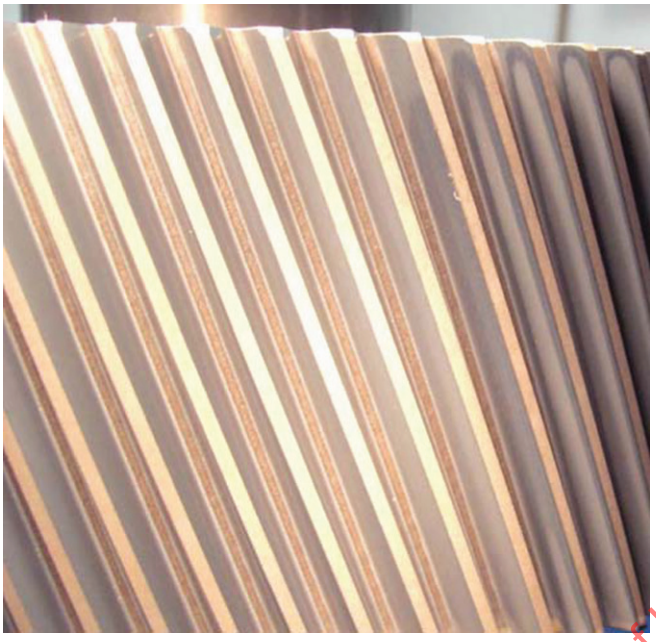
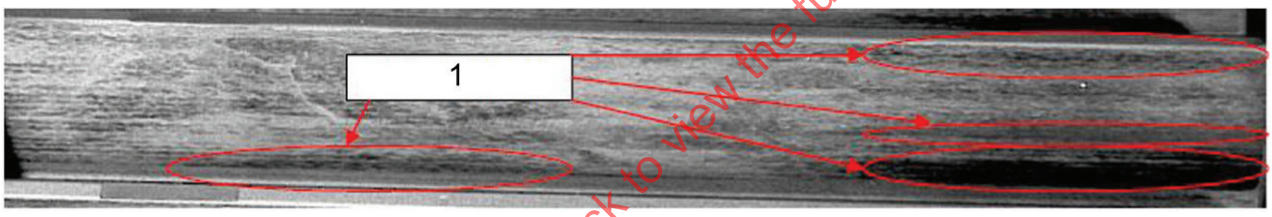


Figure 6 — Progressively worse surface temper of teeth from left with Class FA no tempering to right with Class FE rehardening surrounded by Class FD heavy tempering on four teeth at right



Key

1 heavy tempering

Figure 7 — From Class FB to Class FD heavy tempering

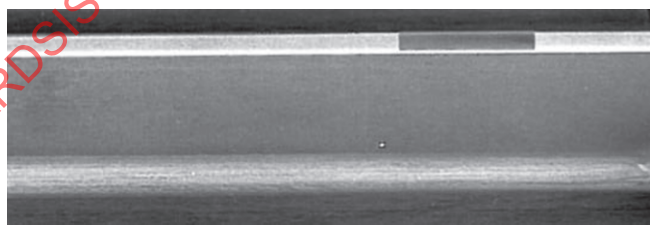
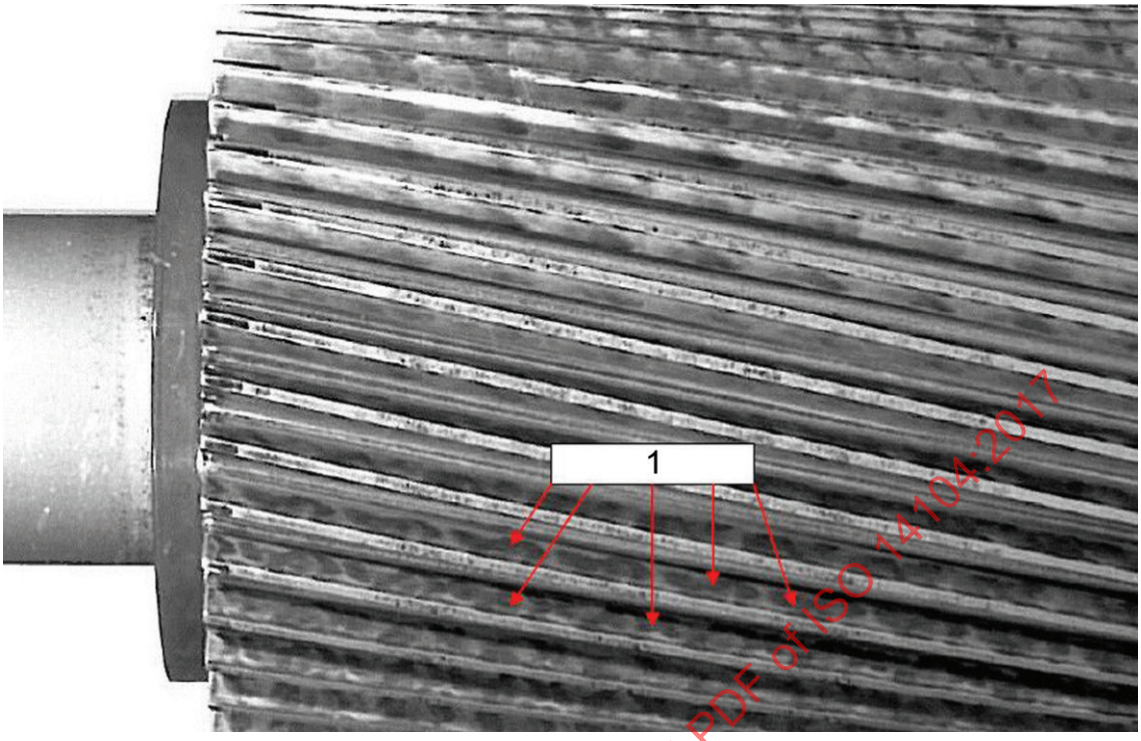


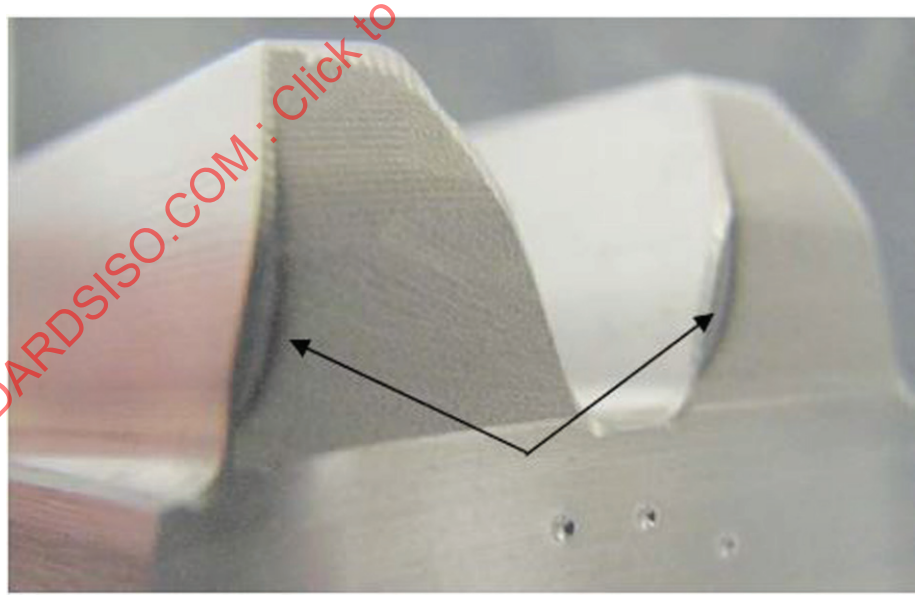
Figure 8 — Class FA no tempering



Key

1 heavy tempering

Figure 9 — Class FD heavy tempering



NOTE Temper bluing on a corner of a gear tooth evident before surface temper etch inspection is a visible clue that grinding is overheating the tooth.

Figure 10 — Tempering at end of tooth