

# The clean status of a refined steel surface and related measuring techniques

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The cleanliness/contamination of a refined steel surface raises a whole series of specific questions and recontamination/cleanliness can frequently extend as far as significant surface damage. Surface damage can certainly be prevented by selecting the appropriate chemical and physical cleaning/purification methods and also by paying attention, in each case, to the final refined steel surface structure of the component surface.

Die Reinheit/Kontamination einer Edelstahloberfläche wirft eine ganze Reihe von fachspezifischen Fragen auf, wobei eine Rekontamination/Reinigung in Grenzbereichen häufig in den Bereich der (massiven) Oberflächenbeschädigung hineinreichen kann. Bei der Wahl der chemisch und physikalisch passenden Reinigungsmethode muss zur Vermeidung der Oberflächenbeschädigung beim Reinigen zudem in jedem Falle auch die endgültige Ausführung der Edelstahloberfläche des jeweiligen Bauteils in Betracht gezogen werden.

**Key words:** Austenitic alloys, Refined steel, Contamination

## Introduction

Austenitic refined steel alloys 316L are put to considerable use in state-of-the-art technology sectors in the pharmaceutical technology/biotechnology field, in semiconductor technology and related areas, where thoroughly clean surfaces are increasingly required. In addition to particle restrictions, the clean status is often also described by the permissible extent of residual contaminants such as approximately  $<1 \text{ mg/dm}^2$  etc. The range of clean conditions is generally stipulated in order to prevent recontamination of new filling media.

On closer scrutiny, the cleanliness/contamination of a refined steel surface raises a whole series of specific questions and recontamination/cleanliness can frequently extend as far as significant surface damage.

In this respect, the following questions must be clarified and synchronised accordingly:

- Type of contamination
- Contamination medium
- Contamination depth
- Extent of contamination
- Cleaning procedures

- Extent of recontamination
- Contamination measuring techniques/procedures.

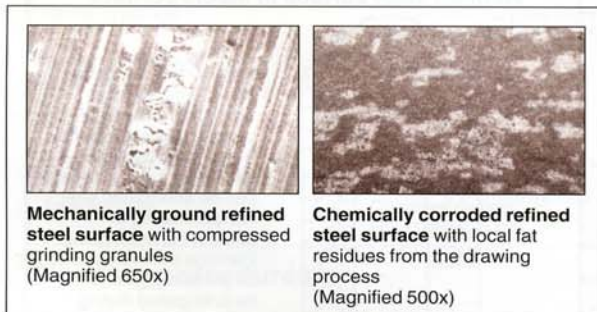
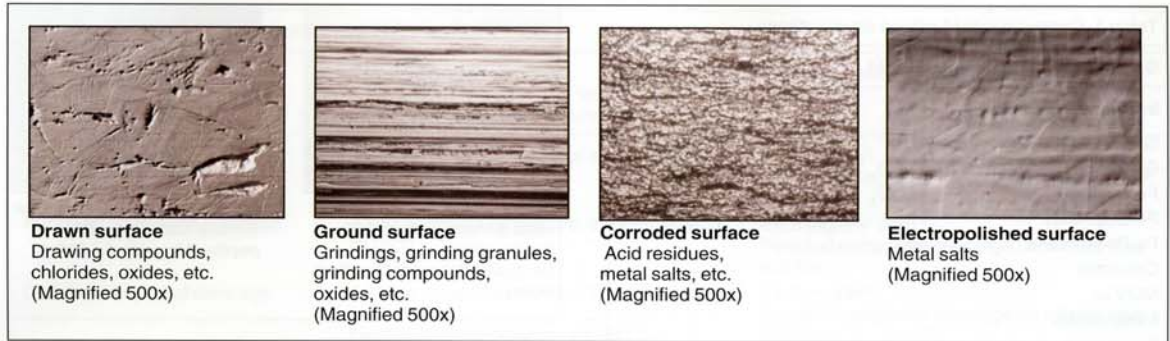
## The impact of final surface processing on surface quality

Every surface-processing technique triggers "characteristic traces" in terms of topography, morphology and energy levels and also generally leaves significant qualitative and quantitative contaminants in its wake (see **Figures**).

Specially formulated mechanical grinding and mechanical polishing techniques are principally (micro-) abrasion methods with the corresponding in-depth effect of the numerous cutting and pressing elements (strip or paste granules) and are capable of definitively extending the problem of surface contamination from the surface itself into the surface interior.

Compressed ground abrasive material, blunt and broken off grinding particles, oils, fats and metal oxides, etc. are forced into the surface and form a typical layer of contaminants on completion of the grinding technique.

The chemical cleanliness of a (pre-ground) refined steel surface facilitates a substantial reduction in contamination status without even coming close to in-depth contamination. Intensified chemical procedures involving temperature and/or concentration or treatment period guard against the risk of substantial damage to the refined steel surface (e.g. corrosion).

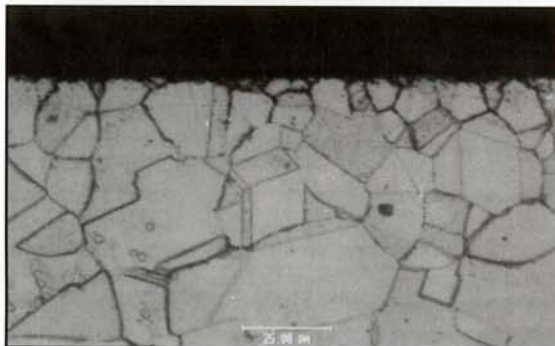


**Figures.** Surface appearance of refined steel surfaces following various grinding and polishing techniques.

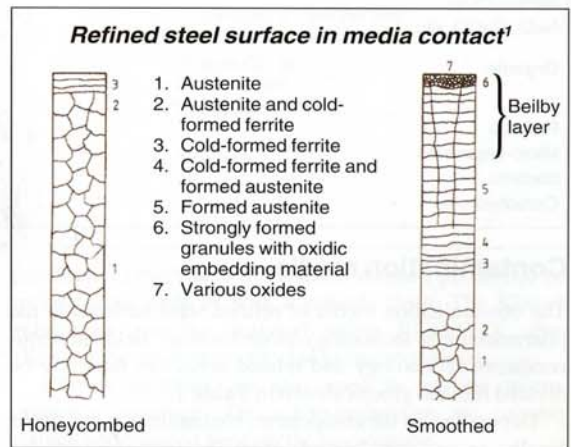
Corresponding cleaning success achieved through electrochemical abrasion of a mechanically ground refined steel surface is at least rewarded with initial success. Topography, morphology and, first and foremost, energy levels are dramatically improved. According to relevant textbooks, the refined steel surface, expertly polished using electrochemical techniques, appears beneath the microscope (>200x) as a typically pure and crystalline structure of the metal.

**The type of contamination**

Contamination with solid substances such as dust or perhaps fatty chinks, dye layers and iron filings from machine processing, etc., is generally given the same



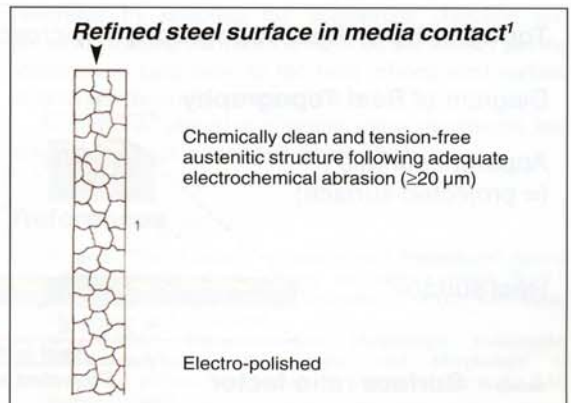
Sustained chemical cleaning (steeped in corrosive material) of a refined steel surface with characteristic evidence of the onset of granule limit corrosion (inter-crystalline corrosion) (Magnified 500x)



recognition as that involving liquid substances such as oils, fats or micro-organisms.

Disruptive surface contamination due to the absorption of gas molecules by the refined steel surface or the uppermost layers of the refined steel surface is also recognised, especially in the sensitive clean room areas. The absorption of oxygen through oxidation within the scope of over-heated, mechanical grinding processes is significant in this instance.

Contamination may also occur either randomly or systematically during component manufacture or the subsequent use of components. This will have an extremely disruptive effect on the respective process being carried out.



**Table 1.** Contaminants of refined steel surfaces.

Solid	Liquid	Gaseous
<b>Inorganic</b>		
Grindings	H <sub>2</sub> O	O <sub>2</sub> , N <sub>2</sub> , CO <sub>2</sub> ,
Grinding particles	H <sub>2</sub> SO <sub>4</sub>	NO <sub>2</sub> , SO <sub>3</sub> etc.
Fe-Oxides	H <sub>3</sub> PO <sub>4</sub>	
Fe-Sulphates	HNO <sub>3</sub>	
Fe-Phosphates	HCl	
Chlorides		
MOV or		
Metal oxides		
Surrounding dust		
Salts such as		
NaCl, CaCO <sub>3</sub> etc.		
<b>Organic</b>		
Fats	Oils	---
Proteins		
Micro-organisms		
Bacteria		
Carbohydrates		

### Contamination media

The contamination media of refined steel surfaces in the pharmaceutical technology/biotechnology field, in semiconductor technology and related areas can basically be divided into the groups shown in **Table 1**.

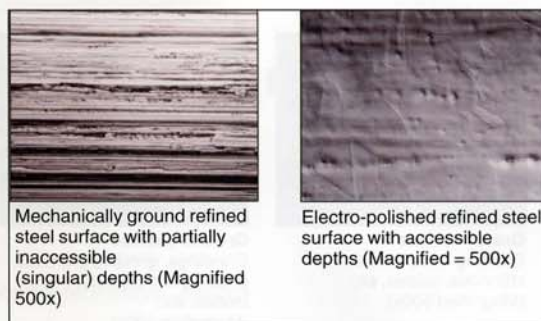
Depending on the component, contamination can occur locally or extensively and affect the mass or particles thereof.

### Contamination depth

Depending on the manufacturing status of the refined steel surface or surface status on contamination of the said refined steel surface, it should be noted that contamination depth plays a key role in recontamination.

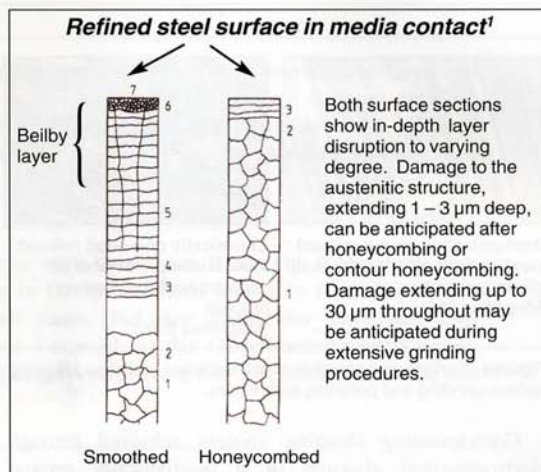
“Depth” can thus be sub-divided into accessible moulds and grooves as well as singular folds, pores and furrows. From both a contamination and corrosion-technical perspective, attention should be paid to grinding particles that are compressed and over-smearred, especially on grinding.

These smearred depths can, in some cases, extend to 3 -



Mechanically ground refined steel surface with partially inaccessible (singular) depths (Magnified 500x)

Electro-polished refined steel surface with accessible depths (Magnified = 500x)



Both surface sections show in-depth layer disruption to varying degree. Damage to the austenitic structure, extending 1 – 3 µm deep, can be anticipated after honeycombing or contour honeycombing. Damage extending up to 30 µm throughout may be anticipated during extensive grinding procedures.

10µm and are thus no longer accessible during straightforward cleaning procedures.

### Extent of contamination

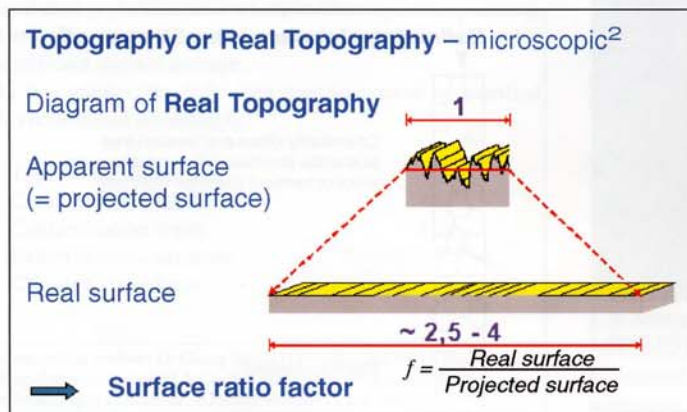
The extent of the contamination mostly relates to the (theoretically) clean status. In this case, the extent of contamination can be expressed as a relative figure, perhaps as a percentage or in ppm, etc.

The total coverage in quantity/surface unit, i.e. perhaps in mg/dm<sup>2</sup>, is another indicator.

In this respect, it is assumed that a test surface must consequently be cleaned under specifically defined cleaning conditions and parameters (temperature, time,

bath movement, etc.) [e.g. completely elute fat coating in acetone, analytical grade, completely dissolve rust coating in HC 1106 (H<sub>2</sub>SO<sub>4</sub>/H<sub>3</sub>PO<sub>4</sub>) etc.] and the respectively dissolved quantity of contaminant analysed (e.g. GC, HPLC, AAS etc.), in order to establish the extent of contamination as a quantity/surface unit.

Particle coatings and corresponding anticipated particle generation by the operating refined steel surface are more difficult to define. In addition, gas coatings must generally be quantified only within the scope of cleaning processes (perhaps the unrestricted rinsing of pipe systems).



**Table 2.** Examples of cleaning procedures

Contamination	Cleaning methods Characterised by medium, temperature, contact time, rinsing pressure, etc.	Extent of residual contamination (Examples)
Polishing compound coverage	Alkaline compound removal through heating HC 500	< 0.5 mg/dm <sup>2</sup> Residual compound coverage on refined steel surface
Punching compound coverage	Alkaline compound removal through heating HC 500	< 0.5 mg/dm <sup>2</sup> Residual compound coverage on refined steel surface
Drawing compound coverage	Alkaline compound removal through heating HC 500 + Electrochemical polishing HE 111 (Electrochemical removal 5 µm)	< 0.5 mg/dm <sup>2</sup> Residual compound coverage on refined steel surface (CFOS application)
Grinding particle	Electrochemical polishing HE 111 (Electrochemical removal 5 µm)	< 10 particles > 1 µm per dm <sup>2</sup> Refined steel surface
Residual chlorides	Chemical rinsing HC 1100	< 10 ppm Cl <sup>-</sup> , or < 0.1 mg Cl <sup>-</sup> /m <sup>2</sup>
Fe-oxides	Chemical rinsing HC 700	< 1 ppm Fe
Fe-oxides as red layer	Chemical rinsing HC 1106	< 1 ppm Fe
Fe-sulphates	Chemical rinsing HC 1106/HC 1100	< 1 ppb S

## Cleaning procedures

The respective cleaning process in terms of type, intensity and outcome is geared, in each case, to the definitively known contamination and the pre-set criteria regarding the extent of residual contamination.

## Contamination measuring methods and measuring techniques

Non-binding terms such as "clean", "oil- and compound-free" and the like are increasingly used in specifications (and, unfortunately, also in DIN standards!). Such statements serve no purpose for technical applications or are generally not required.

In each instance, the aim is to define the cleaning status that can, where possible, be objectively reviewed and technically measured, to agree on the acceptance criteria and thus reach a consensus of opinion in terms of test criteria.

Residual oil and fat contamination, in particular, are defined on the basis of residual coverage quantities (e.g. < 1 mg/dm<sup>2</sup>) and the sample quantity is determined in detail by elution in:

- Acetone (analytical grade)
- Pure ethyl- or isopropyl alcohol (analytical grade)

over time and temperature. The dissolved quantities of fat are determined either by means of GC or HPLC, etc., allowing the surface coverage to be calculated.

Particle contamination of refined steel surfaces is, for example, checked in a controlled argon-pure gas current (< 1 particle per 30 litres basic contaminant) with corresponding stimulation of component oscillation via particle counts in the gas flow.

Residual chlorides on a refined steel surface can also be determined by eluting a test sample (in HNO<sub>3</sub>, analytical grade) and subsequent eluate analysis in AAS. Another

(qualitative) method is the Klatsch method performed on refined steel surfaces with a suitable cloth. The Klatsch substances can be analysed using REM/EDX, thus allowing the amount of chlorine to be determined.

Klatsch methods are also used to analyse micro-organism coverage. The micro-organisms are suitably stained and counted beneath the microscope.

Fe-sulphates and Fe-phosphates on refined steel surfaces are also determined with anticipated minimal coverage < 0.1 µg/dm<sup>2</sup> in addition to the elution test (in HNO<sub>3</sub>, analytical grade + AAS) as well as by REM/EDX analysis (magnified 5000x). These contaminants resulting from the previous electropolishing process mostly appear in clusters and can certainly be eradicated, especially in a controlled acid medium.

One final test to confirm thorough cleaning is carried out by comparing the conductivity of draining water against that of running water.

## Summary

To summarise the subject of contamination, cleaning, methods and limit values, surface damage can certainly be prevented by selecting the appropriate chemical and physical cleaning/purification methods and also by paying attention, in each case, to the final refined steel surface structure of the component surface.

In addition, objective cleaning status parameters and residual coverage parameters are required.

## References

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