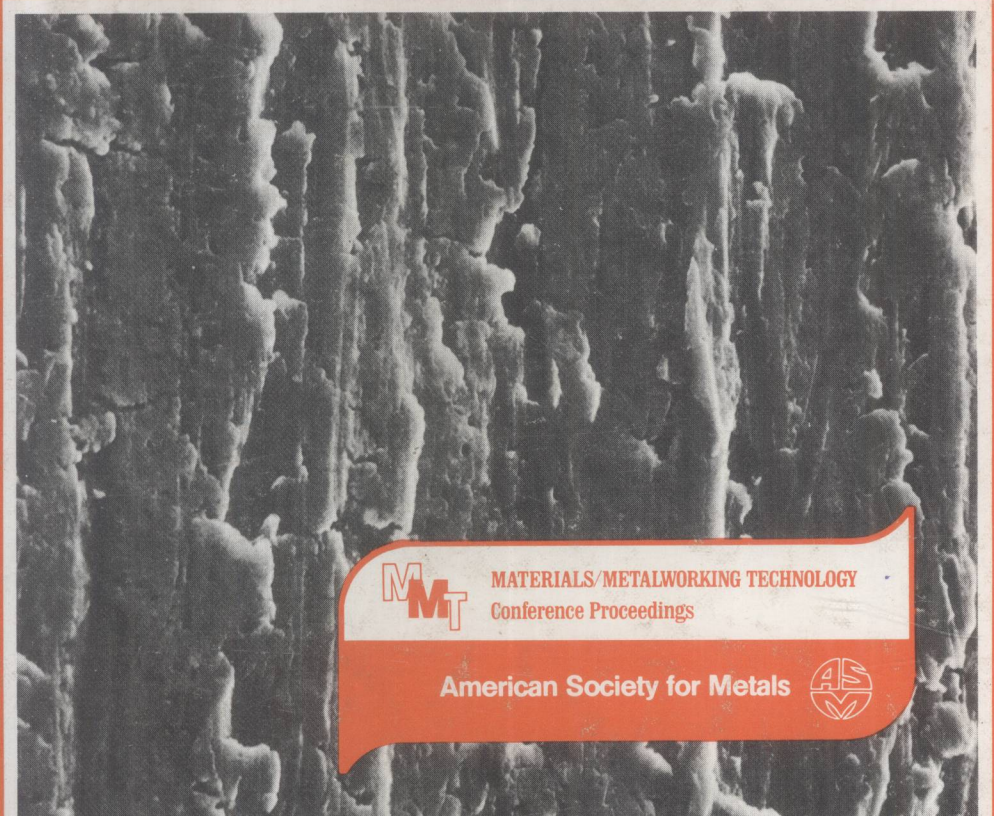
TECHNOLOGICAL IMPACT OF SURFACES

Relationship to forming, welding and painting



MATERIALS/METALWORKING TECHNOLOGY
Conference Proceedings

American Society for Metals

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Relationship to forming, welding and painting

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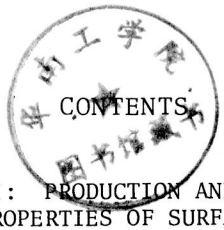

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SESSION I

PRODUCTION AND INTRINSIC PROPERTIES OF SURFACES

THE SURFACE PROPERTIES OF COLD-ROLLED STEEL SHEETS

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INTRODUCTION

Carbodies makers are paying more and more attention on the steel sheet surface characteristics.

Progressively requirements are introduced concerning the surface cleanliness in order to improve the coatings adhesion and consequently the corrosion resistance. Moreover in some cases a better control of the roughness seems to be needed (1-2).

In fact, the cold rolled sheet users wish a drawable and weldable material with a surface :

- reducing the wear of the tooling and more particularly with a low proneness to galling
- having high paintability properties, it means with a surface providing a good adhesion of the coating layers, improving the corrosion resistance (3-10) without any detrimental effect on the aspect and on the gloss of the painted products. Such goals should be achieved at the lowest possible costs.

The two aspects of the metal surface, on the one side the chemistry of the top ultimate layer and on the other side, the texture or the roughness may interfere in a given phenomenon assigned in the agregate to the surface.
Thus, the purpose of the report is twofold :

Firstly, we intend to summarise the principal characteristics with a view to describing the surface of the product. In this context, it will be necessary to discuss in detail the topographical features and the surface chemistry of the steel sheet. In the course of our investigation, we will try to establish the surface characteristics of several important groups of products, with reference to the condition in which

these products are delivered to the user. Our survey will confine itself to a consideration of products manufactured from mild steels.

Secondly, we will attempt, in this investigation, to re-define the most important surface parameters involved in operations such as deep drawing, welding and phosphating. It will be seen that in most cases such processes are associated with several typical surface parameters, rather than with a single one.

2. CHARACTERISATION OF A SURFACE STRUCTURE

A surface is characterized initially by its topographical features and by its surface chemistry.

Both of these are the result of a series of transformation steps to which the product is subjected, beginning with the as-received hot strip and ending with the annealed, skin pass rolled and lubricated state. Thus, the morphology of the surface will be affected to a greater extent by operations such as the cold reduction sequence and by the roughness of the edging rolls and skin pass rolls, whereas the surface chemistry is more dependent on the level of residual elements present during the steelmaking process, on physico-chemical reactions during pickling, and recrystallisation annealing. These reactions give rise to an enrichment in alloying elements (Mn-Cr-P...), in residual elements (Cu-Ni-As-Sb...), or to contamination by extraneous elements (Ca, Mg...)

2.1. The surface texture of cold rolled steel sheet

The surface of a cold rolled sheet as used in the automobile industry is not really flat. Its texture is formed by a series of plateaus and valleys of which the Scanning Electron Microscope (S.E.M.) gives a comprehensive view (Fig.1).
The impression of a chaotic aspect is somewhat corrected by optical micrographs of cross sections (Fig.2).

Nevertheless, size and distribution of peaks and valleys are not regular as illustrated by roughness profiles determined with a stylus instrument (Fig.3). The use of such an instrument is the most widely known method to explore the surface textures. The electrical signal generated by the vertical motion of the stylus is amplified and analysed to provide characteristics parameters of the profile.

Analysis of such profils allows to distinguish a gross waviness upon which a finer roughness structure is superimposed (Fig.3).

Since the stylus tracer is moved at a constant speed, the waviness and roughness components are transformed into a spectrum of wavelengthes. In principle, by filtering the electronic signal it is possible to eliminate the wavelength related to one of the two components : it is the cut off effect. If the fine roughness texture is to be investigated, a high pass filter rejects the undesirable gross waviness whereas a low pass filter allows to remove the lower wavelength due to the roughness component.

As a matter of fact, the filtering equipment of the profilometers does not allow a sharp cut in the accepted and rejected wavelength values. For a preset value of the cut-off a given proportion of the initial amplitude passes through, whose proportion varies with the different wavelengthes.

For usual filters, 75% of the signal amplitude is transmitted for the wavelength selected for the cutt-off; 100% of the original signal passes through for a wavelength 10 times lower while the signal is completely eliminated for a wavelength 10 times higher.

Generally in the case of the cold rolled sheets, the wavelengthes spectra of the waviness and of the fine roughness do not sufficiently differ to avoid interferences between them. On the one hand, when determining the fine roughness profile with a too high cut-off wavelength (high pass filter) the waviness is not completely eliminated and it influences the characteristics of the roughness.
On the other hand, a too low cut off value eliminates completely the waviness but may alterate the fine roughness distribution. It means that the characteristic parameters of a profile vary to a certain extent with the cut-off value. Examples are given in Fig.4.

To represent the surface texture as faithfully as possible, a sufficient number of profiles, are to be determined, each of them on a sufficient base length.

It is recommended to characterise the profiles along the transverse direction in view of a lower scatter of the measurements.

Although many parameters have been proposed, there is no definitive recommendation concerning those to record. This is due to the fact that surface metrology technics concern every type of surface finishes : machined, grinded, shot blasted or rolled ones and that problems to solve differ in each case. According to our experience on cold rolled sheets, specific computations to analyse the shapes distribution, to detect a

periodicity in the patterns by autocorrelation functions do not bring new information in comparison with that given by parameters easy to measure. Table I recalls the most widely used roughness parameters.

For the problems considered in this paper ; i.e. proneness to galling and aspect after painting, the most useful and practical characterization of the roughness texture has been achieved by using the arithmetic means of the roughness (Ra) and the mean peaks (or valleys) length Lmp (or Lmv) at the center line of the profile (Table I)(10-11).

Let us recall that the center line is essentially the line such that the area within the peaks above the line is equal the area within the valleys below the line.

2.2. Surface chemistry

We have attempted, initially, to describe the surface chemistry of an annealed and skin pass rolled sheet by describing and quantifying the contamination which is present on the product.

Within the scope of this description, the surface chemistry may be determined by means of a series of fairly simple tests, which we will discuss briefly here.

"Total contamination" is determined by double weighing and by a test involving cleaning of the surface using adhesive strips; surface stripping should be continued until the opacity of the scotch tape is no longer affected by the operation of sticking and unsticking.

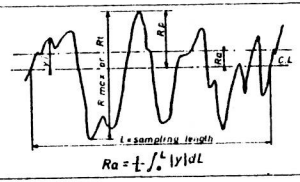
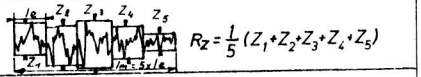
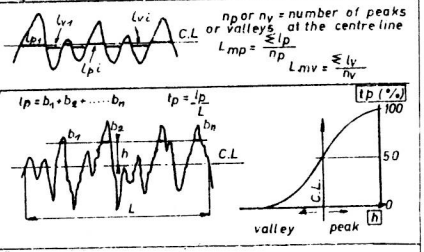
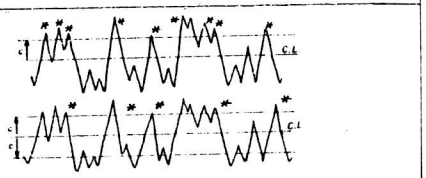
Total contamination C_1^T expressed as mgr/m^2 1 side is given by double weighing. The adhesive strips are then used to determine the quantity of iron present in the total contamination; following combustion in the porcelain crucible, the residue is dissolved in an acid solution 3 HCl + 1 HNO_3 in order to determine, by atomic absorption, the quantity of iron fines $C_1^{T.Fe}$ expressed in mgr/m^2.

For strips which are annealed but not skin pass rolled, we observed that :

$$C_1^{T.Fe} = 0.80\ C_1^T$$

It is evident, however, that a large proportion of these iron fines will be removed during the skin pass rolling operation ; in this context, a distinction must be made between "dry" and "wet" skin pass rolling (i.e. with or without a lubricant).

Table 1. The principal roughness parameters used for cold rolled steel sheet characterization

	Definition	Comments	Graphic illustration
Rp	Maximum height of profils peaks (in µm)	see ISO/DIS/4287/1	
Rma (or R_t)	Maximum height of the profile (in µm)	ISO/DIS/4287/1	
Ra	Arithmetical mean deviation of the profile (or the arithmetical average roughness) (in µm)	ISO/DIS/4287/1	
Rz	The sampling length is divided equally into five parts. On each of them, Rma is determined, Rz is the mean value of the five Rma values (in µm)	following DIN 4768	
Lm	Mean peak (or valley) length : Lmp (or Lmv). In µm : The mean value of the peaks (or the valleys) measured on the centre line of the profile lm = 1/2 (Lmp + Lmv)	not standardized. The peak or valley lengths smaller than 5µ are not taken into account.	
Bearing area	-profile bearing length : l_p -bearing area length ratio tp -bearing area curve : the graphic representation of the relationship between tp and the profile section level h	see ISO/DIS/4287/1	
peak count	The number of peaks per inch (or per µm) -high spot count : Peaks are those cut off by a line at a distance "C" above or below the center line -peak count : Only the peaks cut off both by the upper and the lower levels are counted	-Problem of definition of what constitutes a peak -Problem of the choice of the level within the profile length	

Following "dry" skin pass rolling, we observed that :

$$C_1^{T.Fe} = 0.40\ C_1^T$$

In addition, total contamination is often calculated by means of a test involving tearing off the scotch tape. Evaluation of the opacity ΔU of the scotch tape produces a measurement of the state of total contamination of the surface. This evaluation may be made using a densitometer, with reflections in a scale 0-100 or by comparing reference standards which define the grades G_2, $G_2 \ldots G_5$. Fig.5 gives the correlation observed.

Apart from these simple techniques, there are a number of devices used in testing which are designed to analyse a given element, in particular the carbon present at the surface (3,4,5). We quote :

- the HCl swab-combustion test developed by the Ford Motor Co(12). The object of this test is to eliminate the carbon present at the surface edge by a standard method of etching the surface in a hydrochloric medium using silica fibre paper. After stoving of the silica fibre papers to eliminate traces of hydrochloric acid, the carbon is dosed in a combustion test using an infra-red spectrometer.

In the last few years, the stoving temperature for bearings made from silica fibre has been reduced from 280°C to 180°C, thus giving rise to two types of test depending on the procedure adopted.

- the combustion test, in which the surface carbon is determined by heating the samples up to 550°C at a rate of 20°C/min in the presence of oxygen. The surface carbon is burned to CO_2 and analysed by infra-red spectrometry (13). It has been reported that different carbon peaks may be detected during the annealing cycle which provides some information on the different types of surface carbon contamination.

- the direct oxidation/CO_2 coulometer method developed at INLAND STEEL Co (14,15). Instrumentation for this method consists of a combustion apparatus in pre-purified oxygen flux resulting in the formation of CO_2 and CO; conversion of CO to CO_2 is reached by a barium chromate and resulting CO_2 is quantitatively analysed by a coulometer titration vessel.

- the "cold plasma" technique developed by Cockerill (16) in which the surface of the sheet is bombarded by a plasma of oxygen ions created by a radio-frequency generator.

Under the influence of this ionic bombardment, surface carbon contamination is removed and is then dosed using an infra-red spectrometer and registered continuously in C-t curves. The area under the C-t curve gives the amount of carbon contamination.

The object of these tests is to measure the amount of carbon which affects a conversion treatment such as phosphating. They are generally carried out after an alkaline power wash to remove the protective or deep drawing lubricants. These degreasing conditions are standardised and are intended to provide a treatment similar to that applied at the beginning of phosphate treatment. In contrast to microanalysis techniques, the last tests described give a relative mean value for a surface of several dm^2 and are therefore more applicable to describe a coil.

- microanalysis by secondary ionic emission (SIMS). This method also permits an evaluation of the surface carbon contamination, by recording the intensity of the current emitted by a molecular ion 24_C^- as a function of the pulverisation time (17).

The area below the curve gives the measurement, in coulombs, of the total carbon contamination. Unlike the techniques previously described, SIMS microanalysis can be used to investigate an extremely small surface of the order of 0.05 mm^2. Although this method cannot produce a mean value which could be used to describe a coil, it does make it possible to establish concentration distributions along the length and width of the strip. With this methods, it is also possible to observe particular cases relating to small test pieces.

Within this framework of our research, we have tried to establish a number of correlations between the various techniques described. Fig.6 shows the relationship observed between the Ford test - 280°C and the cold plasma test for sheets removed in the conventional annealed state. For testing purposes, the sheets were not degreased by power washing, although this did not detract from the validity of the correlation observed between the two methods.

Fig.7 gives the correlation observed for a series of non-lubricated sheets removed in the batch-annealed state between ionic microanalysis (SIMS) and the Ford test, as effected in different conditions, i.e.
- without degreasing and stoving of silica fibre papers at 280°C
- with power wash and stoving at 280°C

- with power wash and stoving at 180°C.

These results show that the mechanical action of alkaline power wash degreasing eliminates part of the carbon contamination formed during annealing. In the absence of lubricants from skin pass rolling or from the application of temporary protective coatings, the two types of stoving condition lead to the same correlation.

The correlation between the Ford test and the direct oxidation/CO_2 coulemeter method has been studied by Coduti(14).

For specimens in the as-shipped state which have been cleaned using an alkaline power wash the above correlation shows that surface carbon values are higher by the direct oxidation method than by the HCl swab/combustion method (Fig.6). This discrepancy is due to the swab drying step in the HCl swab/combustion method which induces a substantial loss of carbon, specifically carbon present as organic carbon on lubricated steel sheets, during stoving at 180°C. For carbon contaminations ranging from 1 to 6 mgr/m^2, the average mean difference of the two methods is more or less constant, and is found to be equal to 6 mgr/m^2 by Coduti (14).

It is important to note that although the various techniques used allow measurement of the total surface carbon contamination, they do not distinguish between the different allotropic forms in which carbon may be present at the free surface.

Identifying the chemical form of the surface carbon may be useful in determining the source of carbon contamination.

The different types of carbon included in the "total carbon" present on the steel surface are assumed to be iron-manganese-chromium carbides $(Fe, Mn, Cr)_3C$, organic compounds, amorphous carbon and graphite.

Iron base carbides and graphite are assumed to be associated with the carbon of the bulk material ; as reported by Inokuti (18) graphite may be detected on the free surface of steel sheets which have been predegreased and annealed in a closed-packaged system. We have observed carbide or graphite on a predegreased and vacuum annealed specimen with a free surface (19). It is also well known that graphite formation is also induced during batch annealing of black-plate which is alkaline electro-degreased before annealing (20).

Organic compounds or thermally degraded hydrocarbons are associated more closely with residues of pickler oil or rolling emulsion.