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A profilometric study of levelling in electroplating

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The levelling power of a plating bath is usually assessed from measurements of surface roughness (R_a or r.m.s.) before and after plating. The availability of new parameters for surface characterization now permits a better understanding of the role played by surface microgeometry in levelling. This paper explores the possibility of using these parameters for finding a reliable representation of levelling for quantitative assessment. A new measure based on the profile length parameter is suggested.

1. Introduction

The extent of levelling in electroplating is often assessed by measuring the thickness ratios of deposits on well defined groove shapes by a microsectioning method. In the literature [1–3] these ratios have been defined differently and obtained from grooves of various sizes and shapes for studying the influence of various factors. Besides being tedious and time consuming for routine experiments, these methods preclude the generalization of the results of a single large groove experiment to real surfaces having relatively small roughness depths and wide variations in the surface features [4]. However, this information is readily obtained from profilographic measurement of roughness before and after plating. This paper is restricted to the latter technique.

The per cent reduction in the initial R_a or r.m.s. roughness, called per cent levelling, is commonly regarded as the estimate of levelling. Nobel and Ostrow [5], however, show that levelling can also be indicated by a reduction in the frequency of scratches (number per unit length) on a surface even though there may be no reduction in r.m.s. roughness (R_s). Different behaviours are equally plausible for each of the surface characteristics used to represent a surface. A need, therefore, arises to look for a comprehensive parameter for representing an overall average of the combined factors contributing to levelling. Unfortunately none of the available methods of surface characterization seem to provide a completely satisfactory answer to this problem.

With the advent of digital techniques in the area of surface metrology, many significant developments have taken place in the recent years. A large number of parameters are now available from the new generation of stylus instruments for characterizing surfaces. Small on-line computers may also be used to obtain these parameters from profile signals. An important advantage is that a single profile trace yields a volume of information to permit a more complete understanding of the surface microgeometry. It is now possible to identify the functionally significant parameters by correlating them with performance. This paper reports a fresh appraisal of levelling against this back-drop especially to find a reliable representation of levelling for quantitative assessment. In the following is a brief discussion of the special methods used to achieve this objective.

2. Reliability of stylus instruments

2.1. *Fidelity and surface damage*

For electroplating applications, the suitability of stylus instruments is sometimes questioned [6–8] on grounds of lack of fidelity of their measurement of surface roughness and the damage to the soft metallic surfaces by the diamond stylus. Thomas [9] and Whitehouse [10] have shown that these apprehensions are ill-founded. Experiments indicate that stylus instruments can measure with fidelity relatively compliant surfaces such as those

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of rubber, biological materials [9], paints [11] and micromachined metal mirrors [12]. On very soft surfaces the damage is easily avoided by using low contact force tracing systems having no skid [12] and by lifting the stylus clear off the surface during the return stroke [9]. These facilities are available on modern profilometers.

2.2. *Variability in measured values of parameters*

An important aspect of the roughness measurement by a stylus technique is the inherent variability in the surface and the consequent variation in the measured values of various parameters. The profile traces taken at different locations, even for short distances apart, on the same surface show a sizable variation. A good indication of the scatter, measured by the standard deviation of the observed values, for some of the well-known parameters for a rough ground surface is obtained from the first column of Table 1. Thus, even if an average of many measurements is taken to circumvent the effect of this variation, results may still be quite erroneous especially in a situation like levelling studies wherein the initial and final values of parameters are important.

2.3. *Relocation profilometry*

This technique, described elsewhere [11, 13], allows examination of a particular section through the surface before and after an experiment to see what changes have occurred in the surface microgeometry. Exactly the same section is traversed each time so as not to attribute changes to a lateral displacement of the profile. Even small but significant changes are picked up.

Relocation profilometry is of profound use in levelling studies because it permits repeated observations of a surface section for the purpose of monitoring the changes in microgeometry and its characterizing parameters as the plated layer builds up. Its application in the present study is discussed later.

The variability in the computed values of parameters is reduced to a minimum if a recourse is taken to this technique. A comparison of the scatter of the measurements carried out with and without a relocation table (see Table 1) clearly points out that far greater accuracy is achieved by using this technique.

Because of these advantages, the stylus instruments become the most viable and convenient tools for conducting levelling studies. In the present study these beneficial aspects have been exploited in an attempt to understand the role of initial surface microgeometry in levelling.

Table 1. Scatter of surface parameter values

Parameter	*Standard deviation of parameter for profiles measured at different locations on a surface* (μm)	*Standard deviation of parameter of a single profile re-examined with relocating table* (μm)
R_z	0.3093	0.055
R_{max}	1.1329	0.1119
R_t	1.1026	0.1067
R_a	0.0158	0.00458
R_s	0.0313	0.00489
R_p	0.4265	0.0685
P_t	1.2136	0.088

3. Experimental details

For various machined surfaces, a number of complex surface parameters have been obtained to relate them to the changes occurring in the surface on account of the metal deposition. Rough ground (RG), finish ground (FG) and shaped (S) mild steel surfaces were selected as basis surfaces for depositing nickel in a Watt's bath ($NiSO_4 \cdot 6H_2O$ – 300 g dm^{-3}, $NiCl_2 \cdot 6H_2O$ – 50 g dm^{-3}, H_3BO_3 – 40 g dm^{-3}) containing Superglow-33, a propriatory brightening agent, in recommended proportion. The conditions of electrolysis were selected to be within the range of the normal plating practice. These were kept as given below.

(a) For shaped surface (S) current = 6.2 A dm^{-2}, pH = 3.8, temperature 60° C.

(b) For rough ground surface (RG) current = 4.8 A dm^{-2}, pH = 4.1, temperature 55° C.

(c) For finish ground surface (FG) current = 4.69 A dm^{-2}, pH = 4.1, temperature 55° C.

These were closely maintained throughout every plating run.

A relocation profilometry technique was used throughout the experiments for the present investigation. A Perthometer S5P roughness measuring machine was used to measure roughness before and after plating. The electrical signal corresponding to the vertical displacement of the stylus tip was recorded on a KYOWA 7 channel instrumentation grade FM tape recorder. The signal was then replayed into Digital Corporation's MINC-11 data acquisition and computing system for digitization of the surface profile. Several unconventional parameters were computed from the 1000 digital data samples obtained at a 4 μm sampling interval. Unfiltered profiles were used for computations. The three point method of analysis was adopted for calculating slopes and curvatures at peaks and valleys and for the entire profile after removing the error of tilt [14]. R_a, R_s and other roughness parameters were obtained directly from the instrument.

Measurements were taken after every 10 min of plating to get a total plating time of one hour for each specimen. The plating operation and measurements were performed with the test piece rigidly held in the removable stage of the relocation fixture described elsewhere [13]. Before replating a specimen was given a 30 vol % sulphuric acid dip and thorough rinsing. The deposits obtained were bright and found to adhere well to the substrates.

The variation in the observed values of several surface parameters was studied as a function of the thickness of plating for various substrates nickel plated under the conditions described earlier. The total plating time has been treated to correspond to the thickness of deposit in the discussion of the results in the next section.

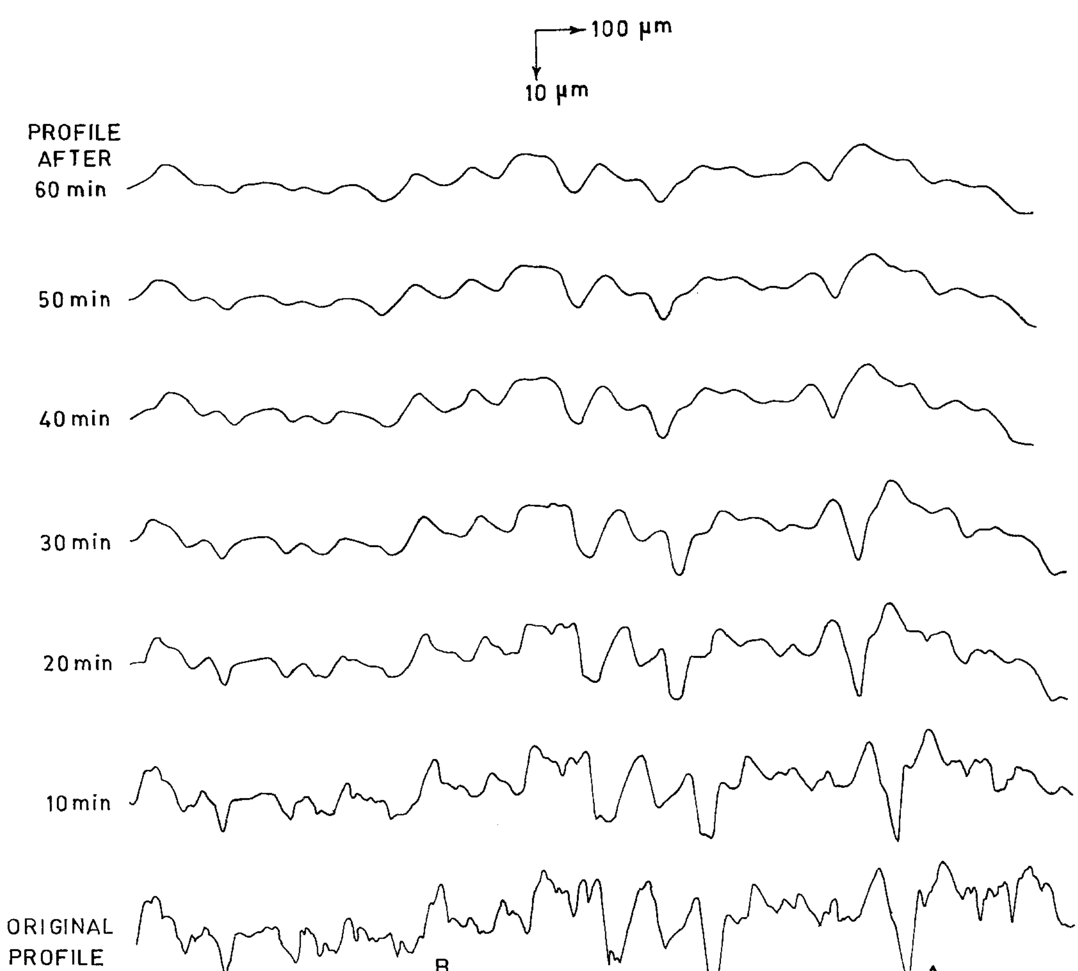

Fig. 1. The variation of a profile with the build up of metal deposit.

4. Results and discussion

The profile traces shown in Fig. 1 were obtained for a rough ground substrate by successively relocating it after depositing metal for 10 min. Only a part of the profile is shown. The progressive changes occurring in the microprofile with the increase in deposit thickness, expressed here in terms of the total time of deposition, are very clearly seen. Levelling is indicated by the filling up of valleys (for example location A) and flattening of peaks (for example location B). Although there is an overall smoothening of the surface it is readily recognised that microroughness disappears more rapidly than macroroughness especially during the early stages of plating. The surface profile rapidly approaches the waviness content of the initial profile. The changes are insignificant after this stage which is reached after about 20 min of plating.

If this behaviour is examined in the context of the terminology of surface finish measurement by stylus technique the inadequacy of R_a or R_s roughness as surface specification criterion becomes immediately apparent. It is known that two surfaces with the same R_a roughness value may widely differ in the average spacing of the scratches or the angle of the scratch flanks. Thus, in plating, the levelling represented by a decrease in R_a roughness may actually be associated with some deterioration of the surface. This may be indicated by an increase in the number of peaks on the surface caused by microscopic crystalline growth. Figs. 2a to d show the variation of peaks and related parameters with the thickness of deposit for shaped, rough ground and finish ground surfaces plated under different conditions. The values shown indicate the per cent variation from the value of a parameter for the original surface. It reveals that a surface gradually improves in finish up to a certain level after which it begins to deteriorate if plating is continued further. This fact is not projected by the monotonously decreasing R_a and R_s values.

To a person not familiar with the working of the stylus instruments the R_a value may appear to give an average of heights of all the irregularities present in the surface profile. In reality, it represents only a part of it [14]. In these instruments, depending on the selected cut off wavelength, the true profile picked up by the stylus is high pass or low pass filtered to correspond, respectively, to the roughness component or the waviness component of the profile. The R_a and R_s roughness, traditionally used for the specification of prepared and plated surfaces, refer to this roughness component which is largely taken care of by the plating process as seen here. The use of R_a and R_s is widespread because these were the only averaging parameters available for measuring surface irregularities until recently. A profile cannot be said to have been levelled unless the waviness error is also ironed out. A specification on waviness, such as waviness depth, thus becomes imperative for surface preparation.

Fig. 3 shows how some of the well known parameters have changed during a plating experiment (same as for Fig. 1). These parameters, except R_a and R_s roughness, were computed from an unfiltered profile for a rough ground surface. The profile slope length ΔL was obtained from the computed value of true profile length by subtracting the assessment length. It appears that ΔL changes most as plating progresses. Evidently, this is the parameter most sensitive to changes in plating. Its computation from different surface profiles has shown that this parameter is also consistent, i.e. the variation in observed values is small.

A close look at the profile slope length parameter ΔL immediately reveals its significance for a reliable representation of the phenomenon of levelling. For the two-dimensional section assessed by a stylus instrument, it represents a combined effect of all the surface elements like peaks and valleys and their spacings. This parameter roughly serves the same purpose as the true surface area for representing levelling. Thus, as levelling takes place, and the surface becomes smooth, ΔL reduces and ultimately becomes zero for a perfectly flat surface. The quantitative estimation of ΔL is, however, much easier.

ΔL is, therefore, suggested here to define per cent levelling as below.

$$\text{Per cent levelling} = \frac{\Delta L_{\text{initial}} - \Delta L_{\text{final}}}{\Delta L_{\text{initial}}}.$$

This expression gives a value of 100 for complete levelling and zero for no levelling. Negative levelling is indicated if ΔL_{final} is more than $\Delta L_{\text{initial}}$. This expression is in keeping with the

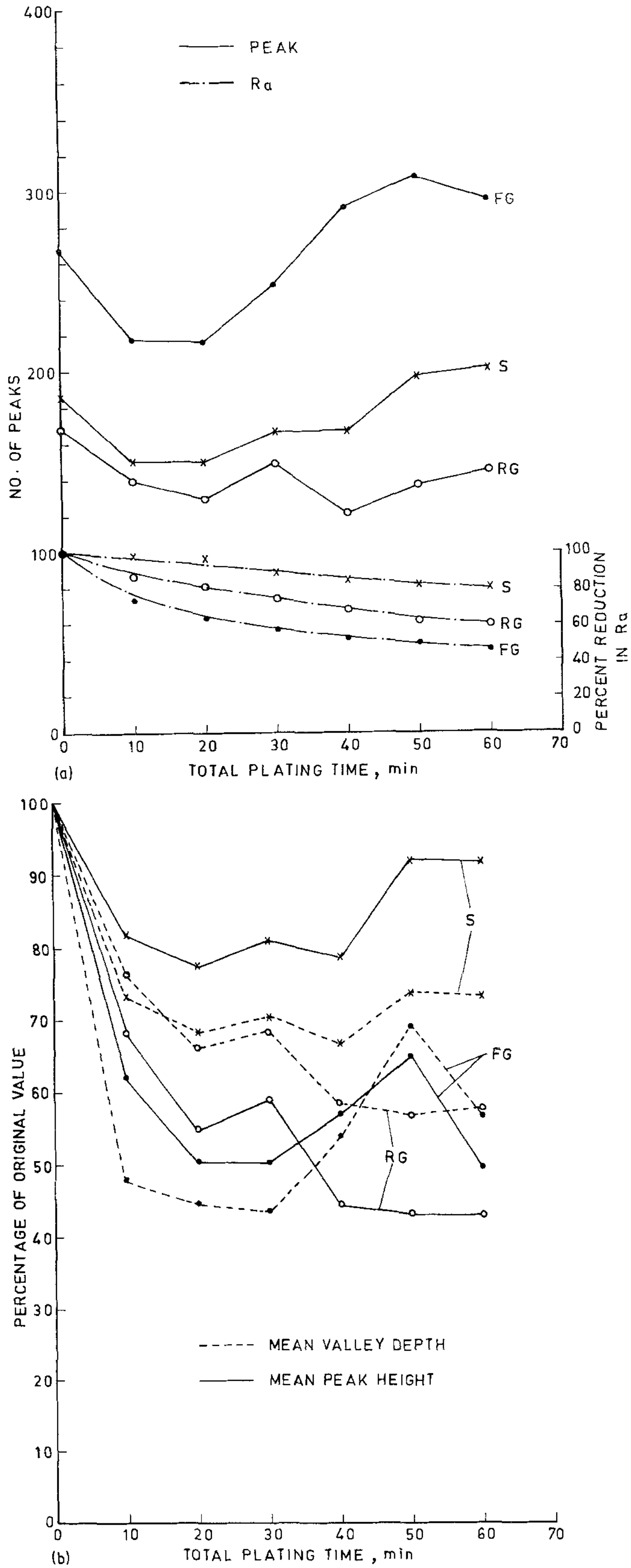

Fig. 2. The variation with the thickness of deposit (a) R_a and number of peaks, (b) peak height and valley depth, (c) mean peak and valley slopes and (d) mean peak and valley curvature [(⋆)S – shaped surface, (○)RG – rough ground surface, (•)FG – finish ground surface, —— peak, – – – valley].

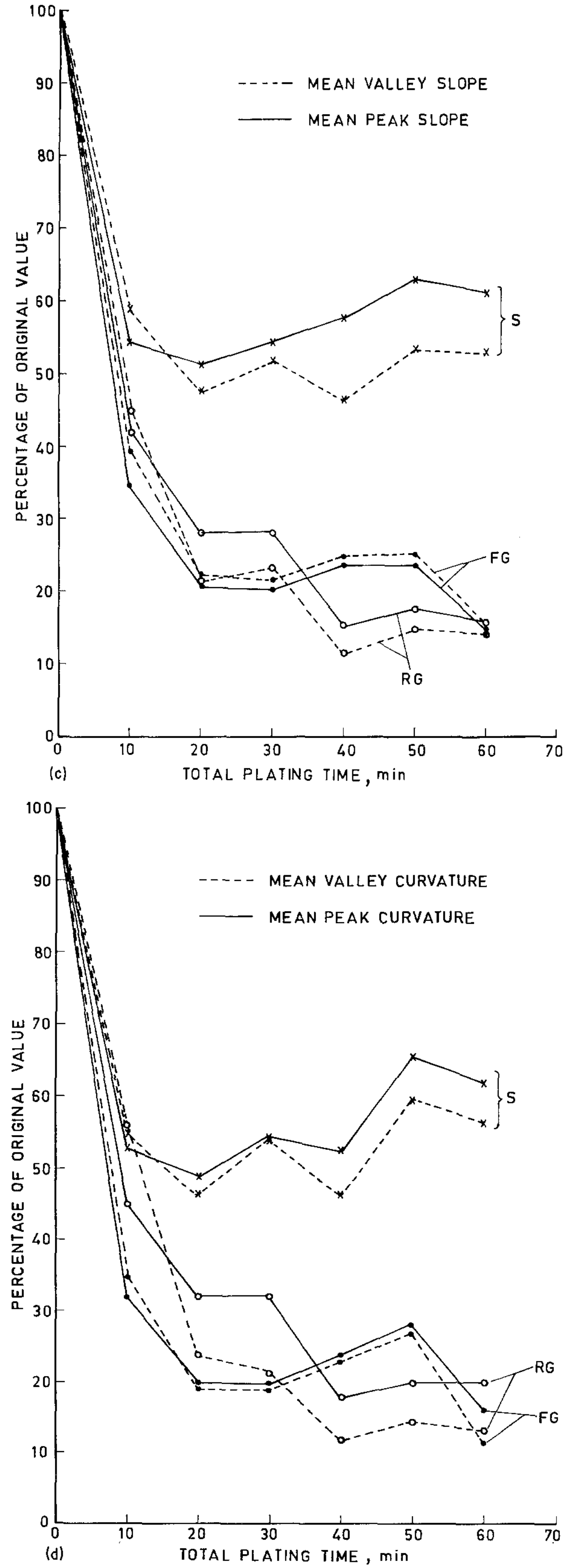

Fig. 2. (*Continued*)

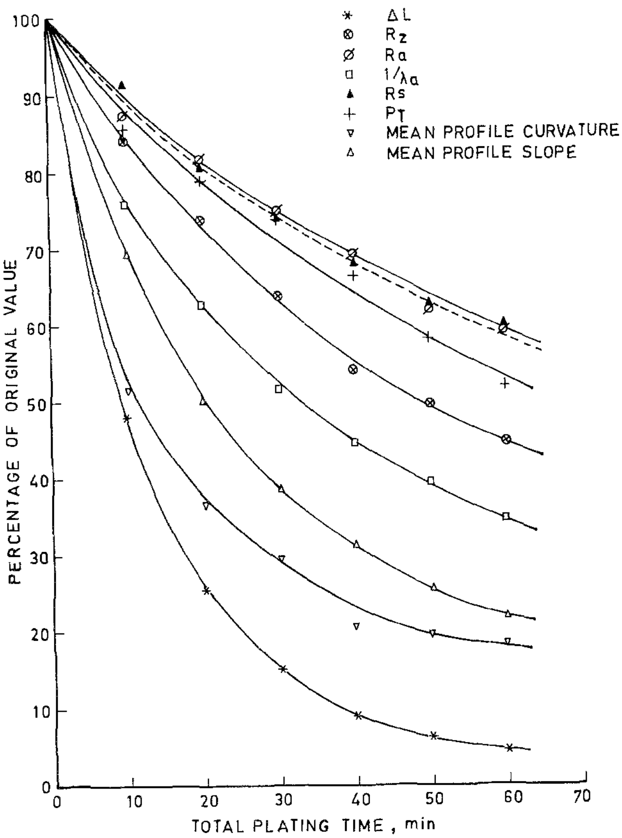

Fig. 3. The variation of surface parameters with the thickness of deposit.

practice of evaluating per cent levelling in terms of R_a or R_s roughness. The values of per cent levelling computed from R_a and ΔL, for different stages of deposition on a rough ground surface (Fig. 1), are presented in Fig. 4 for comparison. It is easily noticed that the rapid initial levelling, indicated in Fig. 1, is effectively reflected in curve II in contrast to curve I. A multiple linear

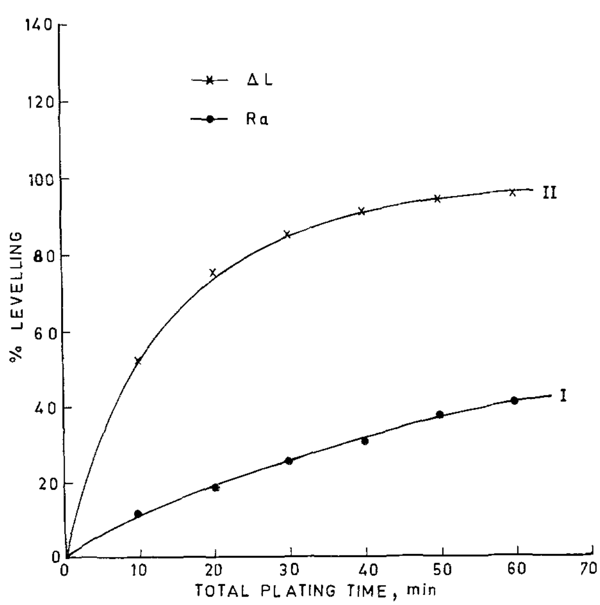

Fig. 4. Variation of per cent levelling with the thickness of deposit.

regression analysis was performed by taking the difference between the initial and final profile length as the response and the various surface parameters of the initial profile as the independent variables. This analysis revealed that amongst the microgeometrical parameters the initial profile length has the greatest influence in determining the final profile length. The suitability of ΔL, which is directly obtained from profile length, for evaluating the levelling ability is thus affirmed.

5. Conclusions

For studying the phenomenon of levelling in electrodeposition the profile slope length parameter ΔL is a more reliable parameter than the traditionally favoured R_a and r.m.s. roughness. Routine assessment of levelling, for quantitative evaluation of plating conditions and additives, can be reliably obtained from the expression of per cent levelling based on this parameter.

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