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**Paper:**

RESEARCH ARTICLE

# Identifying interlayer surface adhesion failure mechanisms in tinplate packaging steels

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Tinplate surface morphology and chemistry is adjusted during the manufacturing process in order to meet the demands of its subsequent product use, the commonest being visual appearance and food packaging stability. A comprehensive experimental study on an industrial tinning line varied the surface roughness and the tin coating weight with the characterization through X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), white light interferometer (WLI), optical imaging, and lacquer adhesion measurement. Increasing tin weight lowers the adhesion through the production of a thicker disorganized tin oxide layer which has a greater tendency to fracture under shearing forces. There is no evidence that the substrate roughness improves the adhesion of the lacquer. Analysis of the failure location identifies fracture in the tin oxide layer below the passivation layer. The findings have impacts on the next generation of passivation materials for tinplate as it has been clearly demonstrated that growth in tin oxide thickness, particularly when unstructured, has a detrimental impact on lacquer adhesion.

KEYWORDS

adhesion, chrome oxide, steel packaging, surface roughness, tinplate

## 1 | INTRODUCTION

Tinplate remains an important material for the metal packaging industry for the preservation of foodstuff providing long-term safe food storage capabilities.[1,2] The tinplate substrate is a multilayer system consisting of electrolytically deposited tin to a base steel substrate which can be subsequently reflown and passivated using a dichromate solution, either catholically or via dipping (Figure 1).

The passivation fulfils four primary roles. It removes oxides present after flow melting of tin and subsequent water quench. It forms a stable and protective tin/chromium oxide film. It forms an oxide film amendable to lacquering, decorative printing, and welding, and it

forms an oxide film that is stable towards sulphur staining by protein containing foodstuffs.[3] This manufacturing process produces a layered metallic system which can then be coated with a lacquer or left without a coating depending on the final product requirement. Following cutting, these coated parts are then formed into a variety of common container shapes.

The surface finish of the base steel metal is often manipulated for final product aesthetic reasons through the use of a roughening roll prior to plating. The quantity of tin applied to the surface (tin coating weight) also provides a means of aesthetic control with higher tin coating weights providing a more mirrored gloss finish at the expense of higher cost (associated with the tin) and lower scratch resistance
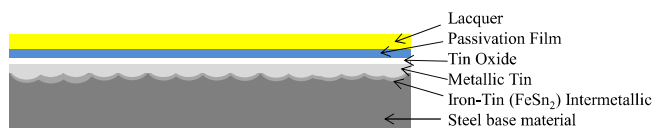
**FIGURE 1**  Tinplate substrate structure

capability. The thickness of the tin layer interacts with the steel surface roughness as rough surfaces over a large area can lead to variations in local tin coating thickness, particularly after reflow, with possible exposure of the steel substrate surface.[4,5] Being a relatively mature material, process, and product, the fundamental research that has been carried out on the impact of surface on adhesion during the last 20 years is limited.

Legislative pressures, which see the ban of the use of epoxy phenolic lacquers[6] and chrome VI[6] passivation in Europe, are restarting the interest in the area as the combined performance of these materials have been shown to perform less robustly in terms of lacquer/substrate adhesion than the incumbent technology.[7,8] These changes potentially have significant implications for the European steel packaging industry. Alternatives to chrome (VI) passivation systems have been studied including cerium,[9] $TiO_2$,[10,11] sol-gel derived silanes,[12] polymers, and fluorozirconic acid.[13] While these represent advances in the pursuit of Cr (VI) free passivation, Cr (VI) has remained the dominant industry method.

Where surface characteristics and its impact on adhesion have been examined, this has focused on electrochemical characterization of adhesion[14,15] and the interaction between the packaged foodstuff and substrate corrosion.[12,16-18] Where there has been an examination of the surface properties and the adhesion force, a direct correlation was derived where the adhesion was related to surface passivation constituents through[19] $Adhesion = Cr_m + 10\frac{Cr_{ox}}{Cr_T}$ where the $Cr_m$ is the chrome metal content, $Cr_{ox}$ is the chrome oxide content, and $Cr_T$ is the total chrome. Failure was attributed to the fracture of brittle $CrO_x$, and the inference of this is that surface roughness and other surface species play no role in adhesion. Further work highlighted other possible surface species, but their role in determining adhesion was not examined.[19,20] Although this equation provided a reasonable fit, given the sample variation and measurement technology of the time, it ignores the role of the Sn and SnOx and other surface species on adhesive failure. Other literature has identified two adhesion failure mechanisms in the lacquer/tinplate system[21,22]: failure induced by contamination between tinplate passivation and lacquer and detachment of the tin oxide film.

Anecdotal evidence suggests that the roughness plays a role in tinplate/lacquer adhesion with roughness improving adhesion through a mechanism which has been reported in other substrate/coating applications as it offers greater surface area for mechanical adhesion[23,24]; there is no such published evidence for tinplate/lacquer adhesion. However, increasing the roughness can come at the expense of important visual characteristics such as gloss and haze[25] and lead to the exposure of base steel iron, providing potential corrosion initiation sites,[26-28] particularly at low tin coating weights.

Increasing tin coating weight is costly but can lead to a more scratch prone softer surface. A balance must therefore be met which addresses the primary issues of visual appearance, cost, and processing robustness.

In order to investigate the impact of the surface topology and tin coating weight on the adhesion of the lacquer, a comprehensive experimental study was carried out where tinplate was produced with range of base steel surface roughness and a range of tin film weight using current standard Cr (VI) passivation technologies. These substrates were characterized and then subsequently lacquered with epoxy phenolic lacquer and their adhesion measured such that any relationships between surface and adhesion characteristics could be established. The objective of the study was to ascertain the impact of the surface topology and tin coating weight on the physical and chemical characteristics of the surface and to relate these to lacquer/substrate adhesion performance.

## 2 | MATERIALS AND METHODS

### 2.1 | Sample manufacture

A full factorial methodology was carried out where tin coating weights of 2.8, 5.6, 8.4, and 11.2 $g/m^2$ were applied to each substrate which had been surface textured by either in line ground roller (I, II, and III) or in line shot blasting (IV, V, and VI). With each roller finishing technology, the roller roughness was varied through abrasive grit particle size. Conformation of the tin coating weight was obtained through measurement using the referee method in accordance with ASTM Specification A630[29] as this provides the most reliable and direct method of absolute tin coating weight. The substrates were produced on a commercial production plant in IJmuiden, The Netherlands, and samples were taken from the middle of each operational roller and tin coating weight. In each instance, five samples were characterized for each condition.

A comparison of the tinplate produced by each finishing method illustrates the morphological differences between the surfaces (Figure 2). The surfaces (I, II, and III) produced by a ground roll are characterized by continuous grooves in the surface in the direction to rolling direction whose amplitude and frequency vary with the grinding roller. The surfaces produced by the shot blasted roller consist of a random surface which is the inverse of the craters produced by the shot blasting of the texturing roll. The underlying pattern created by the texturing roll becomes less apparent with increasing tin coating weight as the tin reflowing process planarizes the substrate. There is evidence of scratching (distinct vertical lines in rolling direction) as result of offline processing used on the sample on the higher tin coating weight samples (8.4 and 11.2 $g/m^2$) which highlights the role of tin coating weight on scratch resistance. Such scratches are not representative of correctly processed commercial material but are a result of the sample manual handling post tinning and the removal of the lubricating and protective di-octyl sebacate (DOS) for surface analysis.
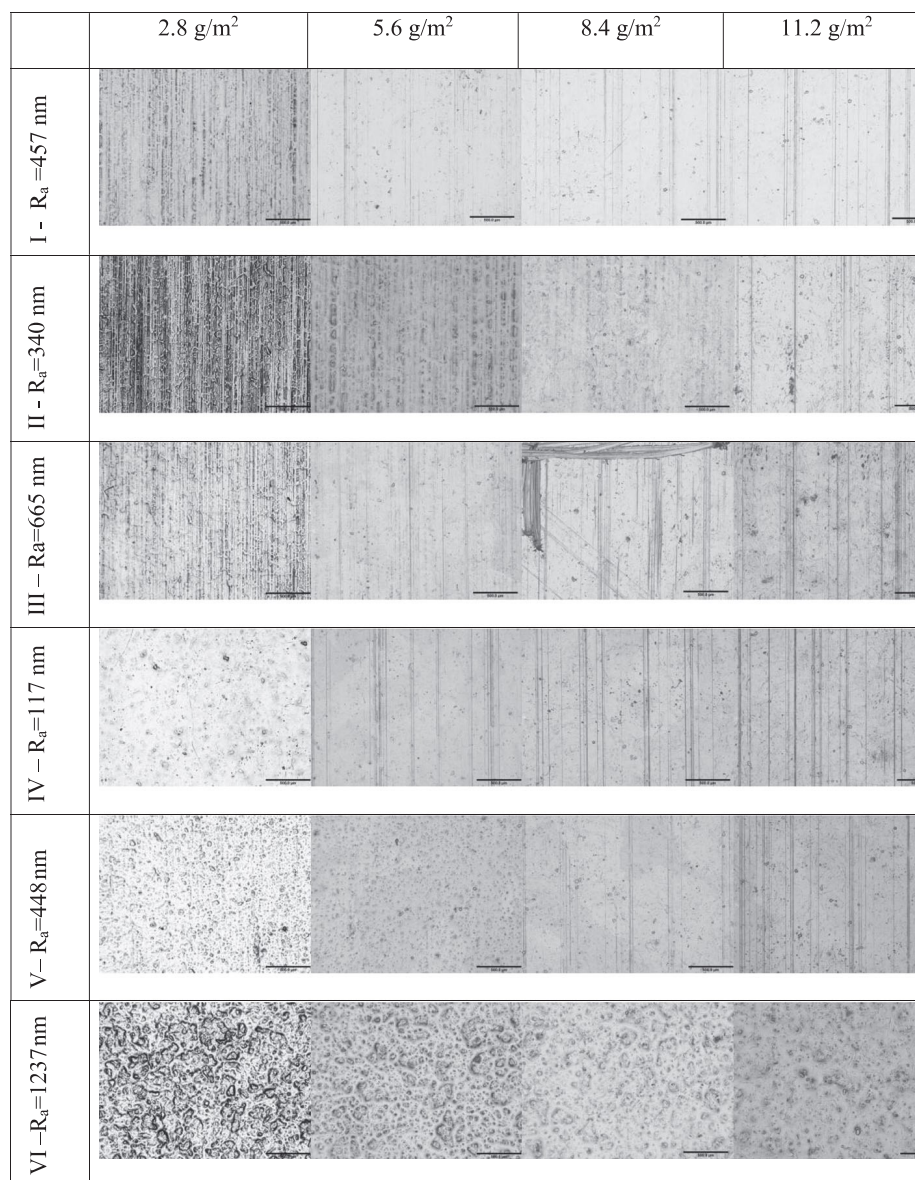
**FIGURE 2** Surface comparison the substrates I, II, and III are produced by a ground finishing roll, while IV, V, and VI are produced from a shot blast finishing roller. Ra values for each black-plate base steel finish are noted perpendicular to the rolling direction

An Akzo Nobel Vitalure 345 lacquer was deposited using an RK automatic Meyer bar coating system which resulted in a coating thickness of approximately 10 μm. Substrate samples were put through a 3-m belt dryer at an air temperature of 200°C with a dryer residence time of 15 minutes.[30]

## 2.2 | Surface analysis

Surface and near surface chemical analysis was carried out using a Kratos Axis Ultra X-ray photoelectron spectroscopy (XPS) using a monochromatic Al Kα X-ray with a source energy of 1486 eV at TATA Steel Ijmuiden. The spectra obtained were analysed using CasaXPS. X-ray diffraction (XRD) characterization was carried out on a Bruker D8 using with measurements taken in 2θ mode between 20 and 110°. Surface roughness was measured using a Veeco NT200 white light

interferometer (WLI) over a 305 μm × 232 μm area with each pixel representing 0.415 μm.

## 2.3 | Adhesion measurement

Although the tape test is a common industrial method for adhesion measurement, the results are open to subjective assessment[31] and insensitive to subtle changes in performance. It can therefore be difficult to measure repeatedly in a scientifically quantifiable manner. Scratch testing provides a thin film quantitative analysis of the force required to induce shear adhesion failure at the organic/metal oxide/metallic interface. Thus, adhesion characterization was carried out using a Sheen Instruments BS3359 scratch tester operated in accordance with ISO 1518.[32] A steel stylus was used where the point of adhesive failure being defined by the mass required to produce
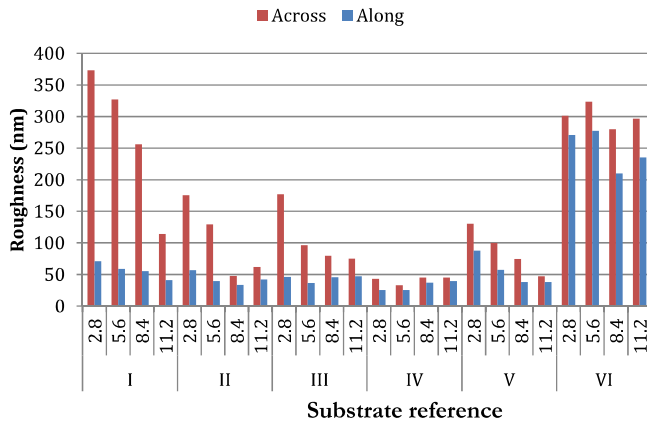
**FIGURE 3** Mean surface roughness of each material in the rolling and across direction for each surface finish and tin weight. Denomination

electrical continuity between the stylus and substrate. This adhesion measurement method provides a physical stress on the surface which is akin to that produced during can forming which can be a primary source of can defects[33] and provides a single nonsubjective numerical value for adhesion. It has been validated in other studies[7,8] and was repeated for five samples, and repeated measurements were shown to be +/− 0.25 g in terms of adhesion force.[8]

## 3 | RESULTS

### 3.1 | Physical and structural attributes

Increasing the tin coating weight reduces the surface roughness (defined by $R_a$) for all the surfaces (Figure 3). This impact is most
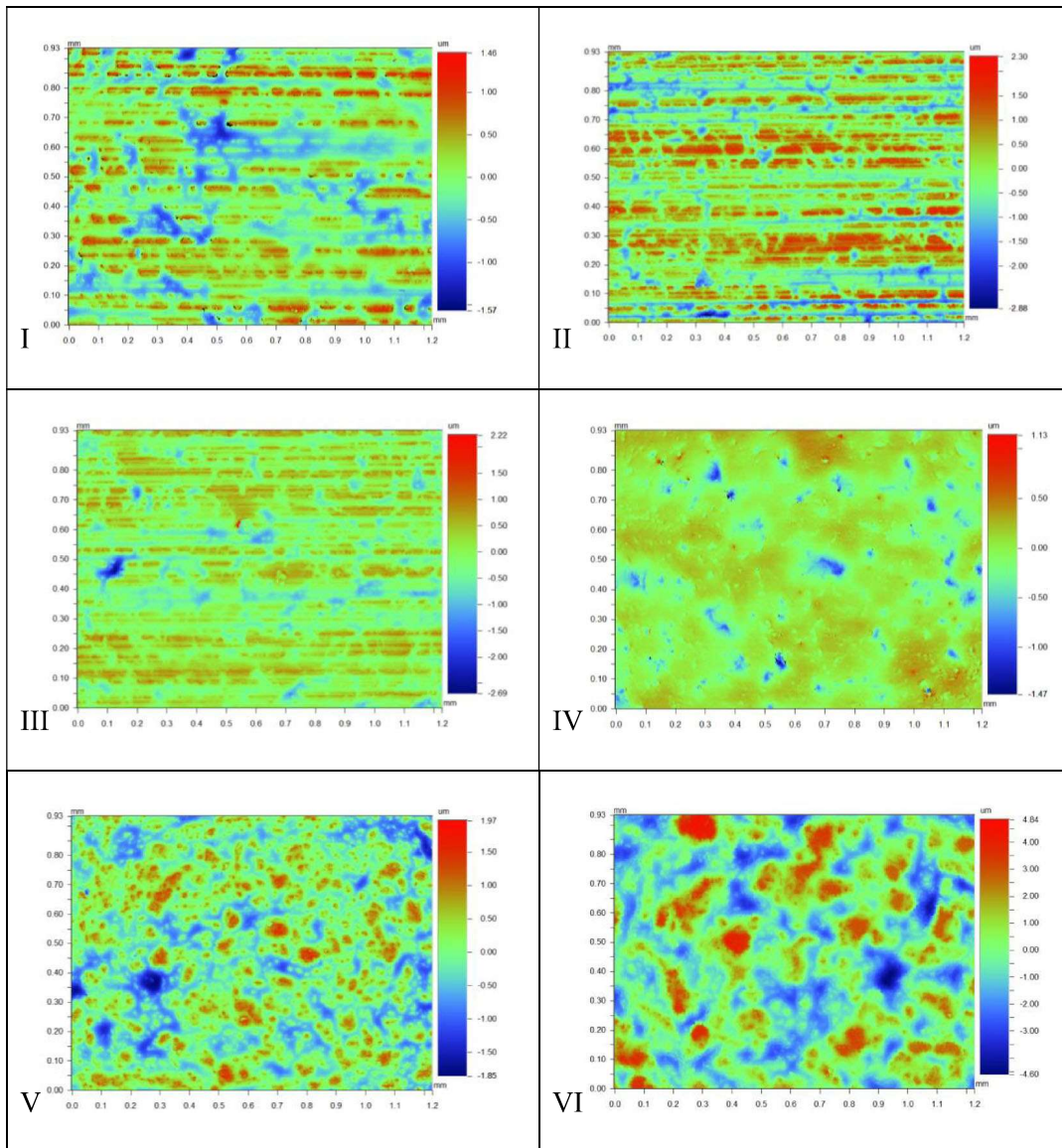


**FIGURE 4** WLI surface topologies of the substrates at 2.8 g/m$^2$ (lowest tin weight). In each instance, the sample area is 1.2 mm (x) by 0.93 mm (y)

evident on the roughest surfaces and reflects the means by which the tin is electrolytically coated to the surface and then softens during the reflow process which results in a planarizing action as the tin fills in the valleys inherent in the surface profile of the substrate, or the irregular cavities observed for the IV-VI substrates. Increasing tin coating weight has a lower impact on the roughness along the rolling direction for substrates I, II, and III as the additional tin and the subsequent reflow process predominately fills the valley between the peaks which run in the rolling direction.

Those substrates (IV, V, and VI) which have been produced using shot blasting exhibit less surface roughness anisotropy, and this is best illustrated by examining the surface topology measured by WLI of the lowest tin coating weight (2.8 g/m$^2$) (Figure 4). In the topographic maps at the lowest tin coating weight, the anisotropy produced by the ground roller surface (I, II, and III) remain while IV, V, and VI are more isotropic in their nature. As the tin coating weight increases, the difference between the roller finish induced topologies reduces (Figures 2 and 3).

For brevity, the presentation and discussion of the XRD results for the surface structure focus on those which identify key findings from the dataset. The surface of the 2.8 g/m$^2$ tinplate for each roller finish (Figure 5A) shows a peak at 30° corresponding to tin with a {200} orientation. There is also evidence of the FeSn$_2$ intermetallic which can be observed with the reduction of the FeSn$_2$ peak at 38°. These findings are in line with literature[34] who postulated structural changes at the iron/tin interface. As the tin coating weight increases, the materials tend towards {−101} orientation; however, it retains a strong

{200} orientation. This is shown best for the flattest base material, substrate IV (Figure 5B). This substrate shows a reduction in the {200} peak at 30°, with a corresponding increase of the {−101} orientation of tin at 33°and {220} and {211} orientations at 44° and 45°, respectively. There is also a slight increase in the crystal orientation at (301) and (112) as tin coating weight increases.[35] This modification of the surface tin is believed to be due to the reflowing of the tin after deposition, whereby the tin forms lower entropy structures due to the reduced interactions with FeSn$_2$.

## 3.2 | Chemical attributes

XPS chemical analysis of the surface and near surface of the surfaces produced by ground rollers shows that the surface is dominated by the materials laid down during the substrate passivation (Figure 6). For clarity, compounds with proportions less than 2% are omitted, while the labels "OH" and "oxide" represent the hydroxyl and oxidic species which are associated with the chrome ($Cr_2O_3$ and Cr $(OH)_3$), respectively. On the very uppermost surface, the primary surface species are chrome hydroxide, chrome oxide, tin oxide, and a small quantity of metallic tin and chrome. The dominance of the chrome oxides/hydroxides at the surface and the relatively thin tin oxide layer (<4 nm) demonstrates its excellent passivation capability given that un-oxidized metallic tin is the most abundant material 3 nm from the surface. With the lowest tin coating weights, there is a presence of iron from about 4 nm below the surface, while increasing the tin coating weight beyond 2.8 g/m$^2$ eliminates the iron from the near surface region (Figure 6A-C). The smoother surface reduces the quantity of near surface iron, and this can be attributed to flow of the tin during reflow which leaves a thinner tin layer on the peaks. The impact of the presence of the subsurface iron is likely to be more prominent in the longer term tinplate corrosion.[28] The presence of near surface Fe reduces as the tin coating weight increases (Figure 6D-L), and there is also evidence of an increasingly thick tin oxide layer. The level of the chromium metallic, hydroxide, and oxide species is largely independent of the surface roughness and tin coating weight. The general findings in terms of dominant species and their distribution are in line with other studies,[19,36] but the depth resolution and comprehensive nature of these findings are significantly higher than the literature.[20,34]

These measurements therefore provide a useful addition to the knowledge base on these substrates.

Similar characteristics are observed with the tinplate surfaces produced with the shot blasted rollers (Figure 7), with primary species of chrome hydroxide, chrome oxide, tin oxide, and a small quantity of tin. Where the surface is rougher and the tin coating weight low, then iron is observed closer to the surface (Figure 7A-C). The presence of iron is absent for any roughness above a 5.6 g/m$^2$ tin coating weight level (Figure 6D-L).

The nature of the surface roughness, and how it is produced, does not therefore play a primary role in determining the near surface species concentration, although the rougher surfaces show the presence of iron closer to the surface.
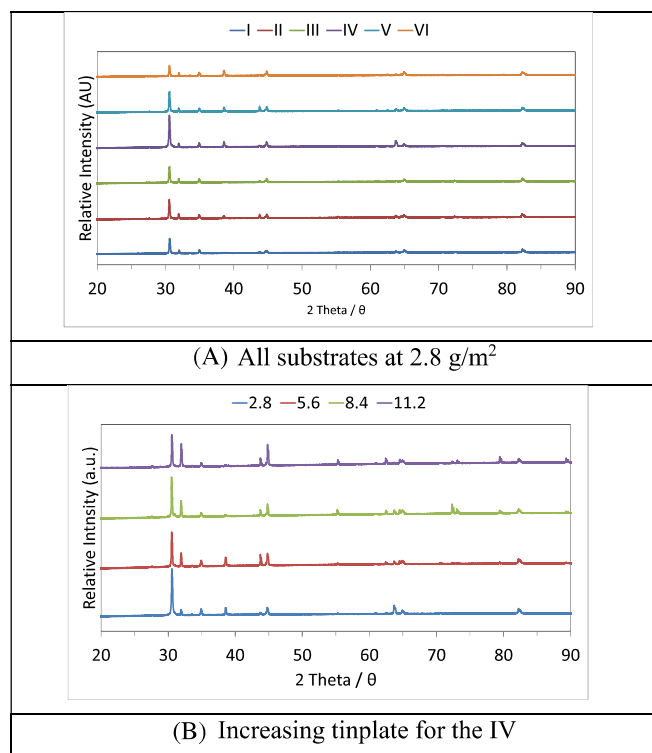
(A) All substrates at 2.8 g/m$^2$

(B) Increasing tinplate for the IV

**FIGURE 5** A, Comparison of spectra for XRD at 2.8 g/m$^2$ and B, influence of increased tin weight on orientation for sample IV
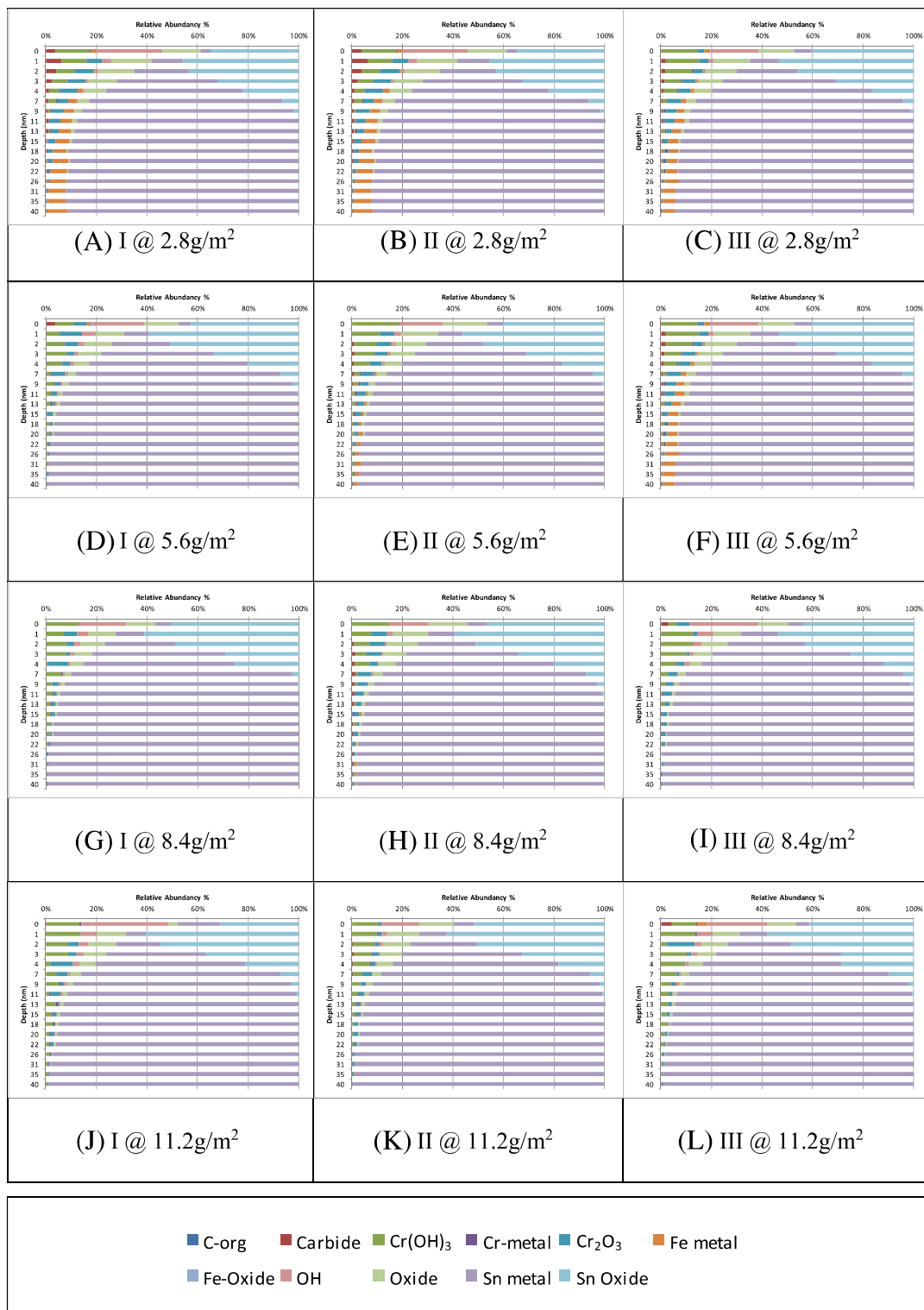
**FIGURE 6** XPS species distribution through the tinplate near surface region for ground roller tinplated substrates

In order to examine the near surface chemistry which is pertinent to the interface formed with the lacquer, the species abundance in the first 4 nm of the surface was analysed (Figure 8) for each texture/tin coating weight combination. The analysis focused on metallic chrome, chrome oxide, and tin oxide as these were identified from literature as possible materials which could dictate loss of adhesion. Exploring the relationship between the surface chemistry abundance surface textures indicates that there is minimal interaction between the surface roughness and the surface species (Figure 9A). The surface species abundance is more highly dependent on the applied tin coating weight (Figure 9B), with increasing $SnO_x$ growth as the tin coating weight increases; a straight line fit with a gradient
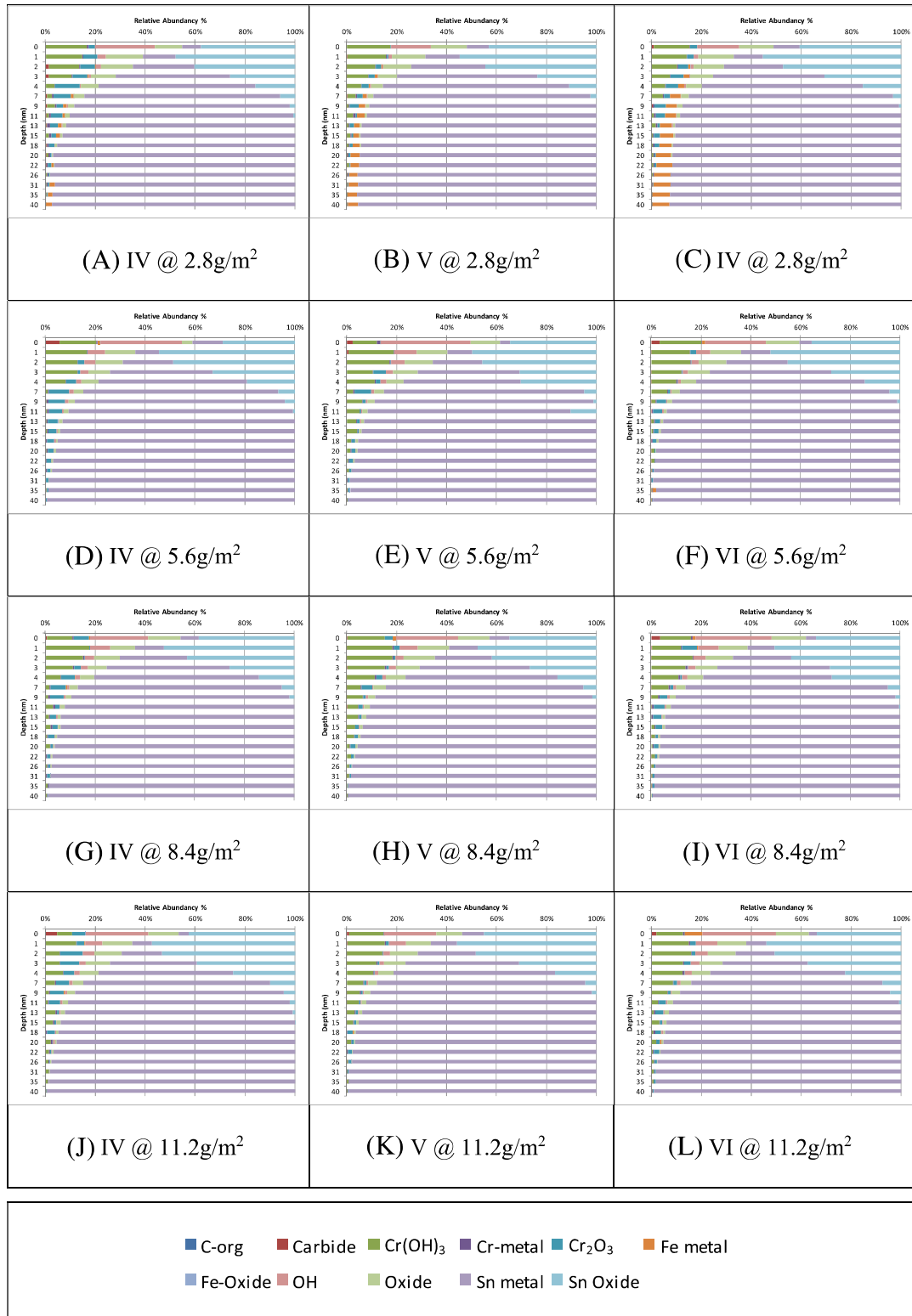
**FIGURE 7** XPS species distribution through the tinplate near surface region for shot blasted roller substrates

of 0.145 mg/m² of SnOx per g/m² of Sn. There is no clear indication of when or where in the manufacturing process or in the post manufacturing storage the increased SnOx is produced, but there is clear evidence from Figure 9B and the XRD (Figure 5) that the quantity of tin present impacts the growth and structure of the SnOx layer.

No clear relationship exists for the relationship between the chromic species and the tin coating weight. This is to be expected as the chrome passivation is applied after the tin, and thus the tin thickness (above a minimum which is less than 2.8 g/m²) does not dictate the formation of the chromic species which are above the tin.
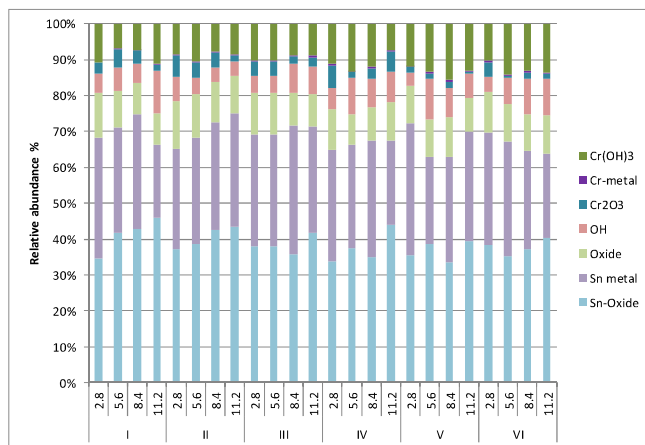
**FIGURE 8** XPS measurements of species abundance by mass averaged for the first 4 nm of each tinplate sample. Abscissa roman numerals denote sample topology; numbers designate tin weight in g/m$^2$

## 3.3 | Adhesive attributes

The range of adhesion measured is 2.2 N (6.5 to 8.7 N), which is an order of magnitude compared with a calculated measurement accuracy of 0.2 N. Changes in the surface structure and chemistry species therefore contribute to changes in the lacquer/substrate adhesion. What is evident from the surface/near surface chemical analysis is that there remains a considerable quantity of tin oxide and chrome hydroxide species at the upper bonding surface. This is contrast to literature[19] where these species are ignored in the prediction of adhesion. This current work shows that there is no correlation between the predicted adhesion and the measured surface adhesion (Figure 10). The performance of the predictive model is therefore extremely poor. The poor performance may be related to the quality of the XPS surface species determination at the time, the larger range of substrates used in this data set, and nature of definition of failure, defined in the literature electrochemically while measured physically in this work. Thus, while the $CrO_x$ may provide the bonding sites between the lacquer and the substrate, this organic/inorganic surface bond is not the cause of lacquer/tinplate failure.
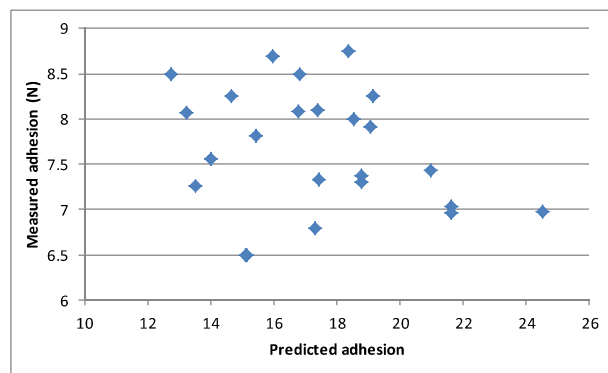


**FIGURE 10** Measured and predicted adhesion according to Mora et al

With the tinplate finished with ground rolls (I, II, and III), there is a reduction in the lacquer adhesion as the tin coating weight and tin oxide level increase (Figure 11A,B). The reduction in adhesion with increasing tin coating weight (Figure 11A) causes an increase in the tin oxide level (Figure 9B), which subsequently reduces the force required to produce lacquer/tinplate failure (Figure 11B). This finding is in line with more recent literature[30] which attributed surface failure to the brittle tin oxide layer. As the thickness of the $SnO_x$ layer increases, so there is a greater opportunity for failure in this layer.

In order to confirm that the tin oxide layer was indeed the plane of failure within the structure, XPS measurements were repeated in the line of failure produced by the scratch testing for the smoothest and roughest ground roller finished substrates (Figure 12). The ground roller finished surfaces were chosen due to their nonisotropic surface structure. This meant that scratches made in the rolling direction would be less susceptible to variations in surface species due to local topology. There is a noticeable difference in the composition of the upper surface and the composition in the valley created by the scratch. Within the scratch, there is no evidence of any chrome species (metallic, oxide, or hydroxide) indicating that these have been removed by the stylus. There is no exposed iron at the surface indicating that the failure has occurred above the iron and the iron/tin intermetallic layer. The primary difference in the surface chemistry is an increase in the oxides associated with the tin oxide formation. As
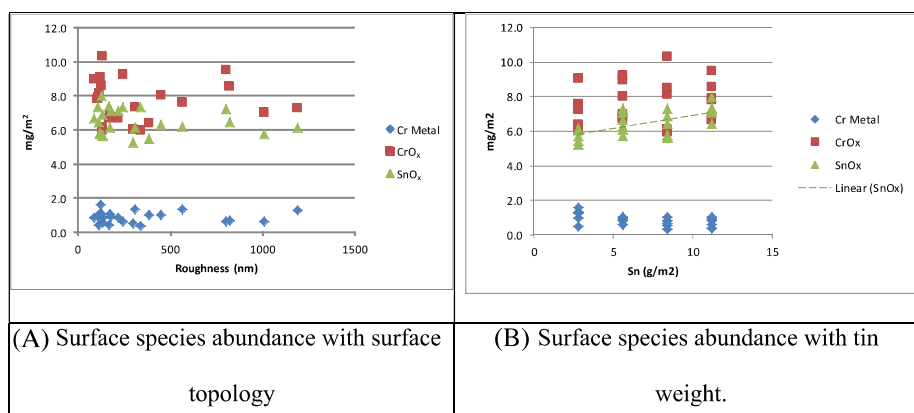


| (A) Surface species abundance with surface topology | (B) Surface species abundance with tin weight. |
|---|---|

**FIGURE 9** The impact of surface A, physical topology and B, tin weight on the chrome metal, chrome oxide, and surface tin oxide (averaged for the first 4 nm of the XPS measurements). Dashed line indicates linear fit
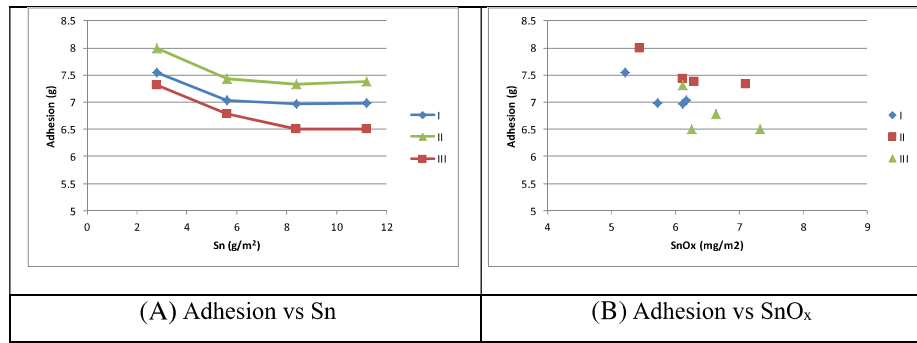
| (A) Adhesion vs Sn | (B) Adhesion vs SnO$_x$ |

**FIGURE 11** The influence of (A) tin oxide and (B) tin on surface adhesion as measured using the load on a mechanical scratch tester
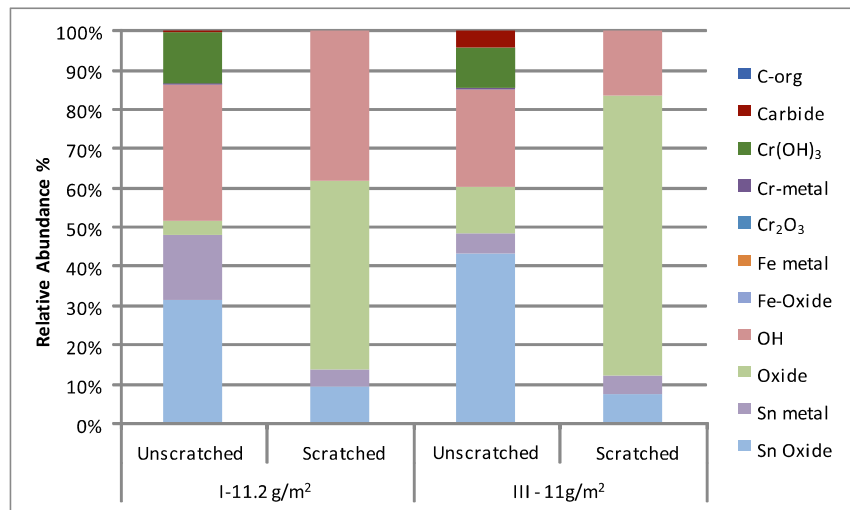


**FIGURE 12** XPS surface composition of the unscratched and scratched surface for the I—11.2 g/m$^2$ of Sn and III—11.2 g/m$^2$ of Sn substrates

these species dominate the scratched surface, it implies that the failure has occurred within this layer. The increase in the ratio of the oxide to tin in the oxide layer also suggests that some further oxidation, from SnO to SnO$_2$, has occurred in air between scratch and measurement as the protective chrome passivation layer has been removed. This chemical study provides the first public domain analytical evidence known to the authors that the SnOx layer is the cause of failure.

The identification of the SnO$_x$ as the point of adhesion failure has implications for next current and future developments and usage using tinplate. Any passivation technology which replaces Cr (VI) will need to prevent the growth of oxide with performance at least comparable with the incumbent Cr (VI). Thermal history[7] and prolonged storage have been shown to be contributory factors to tin oxide growth, and this imposes additional requirements on passivation. The dataset provided by this paper provides a measure against which the future passivation technologies can be compared in terms of the adhesion. An optimum production value for the tin oxide is difficult as it must consider the impact of the passivation layer and likely growth and the requirement for some amount of tin oxide in order to improve scratch performance.

## 4 | CONCLUSIONS

An experimental study of the effect of surface roughness and tin coating weight on adhesion has been carried out. There is a link between an increase in the tin coating weight and a change in the orientation of the tin on the surface indicating that higher tin coating weights have a more disorganized structure. This in turn means that the higher tin coating weights lead to thicker oxide films and these thicker tin oxide films lead to a measurable reduction in the adhesion performance. The study concluded that there is no correlation between the surface roughness and the measured adhesion and that the underlying macrostructure of the texturing roll surface (ground or shot blasted) does not play a significant role in the adhesion. The primary means of adhesion failure has proven to be the brittle tin oxide layer between the tin surface and the chrome passivation layer.

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