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Final Report

**IMPROVED SURFACE QUALITY OF
EXPOSED AUTOMOTIVE STEELS**

By

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EXECUTIVE SUMMARY

Surface quality of sheet steels used for exposed applications represent an enormous economic, technical, and operating issue for steel producers and their customers, particularly automotive manufacturers. (Exposed applications are the visible components of a final assembly, such as the outer body of an automobile.) Surface evaluation is usually based on subjective criteria, and this project was initiated to develop quantitative methods to assess surfaces and to facilitate improvements by defining more quantitatively the dimensional characteristics of steel surface imperfections which do not lead to visible features after painting. Other hurdles identified for the project included acquiring the capability to sensitively measure and analyze surface imperfection topographies before and after painting, applying paint films that adequately simulate automotive production, and assessing defect appearance in a manner that is acceptable to the automotive painting community. The objectives for the proposed work involved 1) characterizing the topography of various coated sheet surface imperfections that are encountered by automotive producers, and assessing the severity of these imperfections after painting, and 2) characterize the changes in topography which result from stamping during the manufacturing process, and their potential to mask surface imperfections. It was also considered that recent changes in automotive paint systems (such as the use of increased film thickness for “anti-chipping” performance) could provide reduced sensitivity to some surface imperfections, which could have important industrial implications. The associated benefits include lower costs, higher quality, reduced rejections, production efficiencies, etc.

A detailed workplan was proposed for this project, and the various steps in the plan were completed successfully. A three-dimensional optical profilometer was obtained to conduct the project, and was demonstrated to be a powerful tool for assessing imperfection topographies and their evolution during forming and/or painting. Visual inspection was conducted on 4" by 12" or larger sample areas. The methodology developed for this study yielded considerable quantitative information, and was able to quantify the surface geometries at much higher resolutions than the capability of the human eye. That is, all imperfections detectable through visual inspection were easily characterized with the instrumental techniques employed in this research, and many imperfections that were completely invisible through human inspection after painting were still measurable by 3D topography. While visual inspection clearly involves some level of subjectivity, experience, and operator sensitivity, it is clear that imperfections, which were not detectable by profilometry, are (unambiguously) not visible. Even by this most conservative standard, the results of this project showed that some imperfections of genuine concern to steel producers can be rendered completely invisible by painting, and many more imperfections are effectively invisible after painting when assessed by more reasonable standards of evaluation using human vision. Imperfections evaluated during the project involved a variety of laboratory simulated imperfections, as well as real surface imperfections on commercial coated sheet products provided by participating companies, carefully selected to be "questionable" in terms of their expected visibility after painting. (Successful methodologies were developed to simulate geometric features of real imperfections, thereby creating reproducible surface imperfections for systematic investigation.)

The most important topographic characteristic of imperfections that controls visibility after painting was suggested to be the depth or height. Key paint system variations in this study included a liquid primer surfacer over the conventional electrolytic primer, a developmental 2-step electrolytic primer, and a conventional high-build powder primer surfacer, all in combination with a black basecoat plus clearcoat. Substantial differences in the ability of the different paint systems to cover or attenuate imperfections were noted, with the high-build powder system showing substantially better attenuation of imperfections. A simple model was hypothesized to explain surface topography evolution during various painting steps, with the aim of better understanding the observed inability of organic paint films to successfully cover or attenuate imperfections of much lesser height or depth than the thickness of the paint film. This model assumed that a smooth liquid surface formed during application (or initial curing) of the topcoat, and was based on shrinkage of the polymer film during curing, associated with solvent removal and/or crosslinking. Using reasonable values for the film shrinkage ratio, it was predicted that geometrical imperfections would be “projected” through the organic film with residual surface displacements quite similar to the actual displacements measured after painting using 3-D optical profilometry. The model would suggest that the ability of paint films to successfully cover imperfections would increase with reduced film shrinkage ratios, and increased numbers of liquid layers, while it was clear that the film thickness (perhaps surprisingly, or counterintuitively) would not have a first-order effect. The results of this study provide specific data to support quantitative inspection criteria for quality-assurance of sheet surfaces. The structure and details of such criteria are best developed collaboratively,

and thus a specific recommendation is not stated in the report, but will be addressed directly through technology transfer activities with the participants.

A second component of the project examined the influence of forming strains on simulated and real surface imperfections on coated sheet steels. The behaviors of different surface imperfections during macroscopic straining of the sheet material were found to differ dramatically, depending on the characteristics of the initial imperfection. The depth of dent-type imperfections, for example, was found to diminish during straining, while dross-type imperfections on coated sheet increased in height during straining, even in the absence of direct contact between the dross particles and the forming tools. The background roughness increased during straining for all of the coatings examined, including electrogalvanized, hot-dip galvanized, and galvanized, although the extent of roughening was much greater for galvanized, due to the extensive network of microcracks that formed through the coating thickness. Some shallow imperfections (of depth/height similar to the amplitude of the unstrained surface roughness characteristics) were found to disappear during straining. The microcrack “imperfections” created during forming of galvanized remained visible after the e-coat primer application, but were completely invisible after final topcoating.

The results of this work should have direct implications in terms of 1) adoption of the quantitative methodology for assessing topography evolution of imperfections during painting, 2) adoption of quantitative criteria for defining the acceptability or unacceptability of imperfections on sheet surfaces, 3) understanding of the behavior of specific imperfections in response to forming and/or painting processes, and 4)

understanding the role of key painting process characteristics in improving the tolerance to topographic imperfections on sheet surfaces.

1.0 INTRODUCTION

Surface imperfections are an important cause of material rejections on exposed-quality panels, such as automotive outer body applications, and surface imperfections remain an important economic and technical issue in both the steel producing and steel consuming industries. Inspection related to sheet surface quality has traditionally been carried out through visual examination by skilled individuals using the naked eye. Decisions for acceptance or rejection of surface imperfections are made by experience, without the benefit of extensive quantitative or objective methods. The lack of objective means for measuring and specifying imperfections contributes to the cost of steel and manufacturing, and much effort is expended in deciding whether a particular imperfection is likely to exceed the threshold of acceptability.

While there has been a significant amount of research on the surface characteristics, friction, corrosion behavior and other properties of coated sheet steels, there are no definitive studies regarding visibility of surface imperfections after painting. Therefore, research on the forming and painting response of various types of surface imperfections was initiated through this project. Imperfection geometry is assessed utilizing surface topography data generated through optical profilometry and comparison with visual inspection, with the expectation that quantitative assessment will lead to improvements in acceptance criteria and ultimately improve efficiency in steel production and manufacturing. The research is focussed on painting studies, and forming studies, where evolution of the surface topography of imperfections is examined in response to painting, and forming, respectively.

The painting component of this research consists of creating and sampling imperfections on galvaneal coated surfaces, and carefully characterizing the topography. Changes in the surface are then measured after painting simulations, and visibility after painting is assessed. The results are analyzed to understand the changes that occur during painting, and to determine whether it may be appropriate to consider quantitative inspection criteria. The results of this research should provide new data quantifying the relationship between the geometry of imperfections before painting, and the resulting geometry after painting, and a methodology to measure and analyze such surface imperfections. The results are expected to contribute significantly to the ability of automotive sheet steel producers to understand and deal with one of their most troubling issues, i.e. surface quality. A clear definition of surface topography characteristics that lead to satisfactory performance after painting would be of value, potentially reducing material production costs and improving perceived product quality. Furthermore, the improved understanding resulting from this work should assist efforts to make additional improvements in exposed surface quality, by providing the tools to evaluate and prioritize imperfection severity in a more quantitative manner.

The forming component of this work assesses the severity of imperfections before and after macroscopic deformations simulating sheet stamping. Dent-type and raised imperfection morphologies are created in the laboratory, and compared with real imperfections on commercial samples originating during the metallic coating process.

In the following chapter, the technical background for this program is reviewed, including classification of surface imperfections and techniques to quantify surface morphology

as it applies to painting, along with a basic review of automotive painting and optical considerations related to appearance of imperfections. Results, discussion, and final thoughts on factors controlling imperfection appearance after forming and painting conclude this study.

2.0 BACKGROUND

2.1 Coated Sheet Steel for Exposed Quality Automotive Panels

2.1.1 Hot-Dip Coating Process

Zinc coating processes involves a series of sequential stages, depicted in Figure 2.1 (1). Imperfections can be created at any point in the line and may also be inherited from earlier processing steps. Chemically induced imperfections originate in the galvanizing bath that is detailed further in Figure 2.2 (1). The galvanizing bath is the location of interest for the following sections that address formation mechanisms of various “chemically induced” imperfections.

Figure 2.1: Schematic of a typical hot-dip galvanizing line (1).

Figure 2.2: Schematic of the roll arrangement in the galvanizing bath from the hot-dip galvanizing line depicted above (1).

2.1.2 Sheet Steel Products

To better understand the effect of imperfections on painting, it is worthwhile to review the galvanized/galvannealed coating processes and the characteristics of each coating.

2.1.2.1 Galvanized Coating of Steel Sheet

Continuously passing a steel strip through a molten bath of pure zinc (at least 99%) and aluminum (up to about 0.2%) results in galvanized steel. When the steel strip is dipped into the molten bath, an immediate reaction occurs between the zinc and iron, which come into contact. The resultant coating is a combination of pure zinc on the surface and an intermetallic layer (e.g. Fe_2Al_5) at the steel/coating interface. Formation of Fe_2Al_5 creates a barrier, which slows the formation of Fe-Zn intermetallic phases (2). As the steel strip leaves the molten zinc bath, the thickness of the coating is adjusted by wiping with a gas knife while it is still liquid, followed by controlled solidification.

For hot dip galvanized steel used for exposed panels, an additional step may be added prior to painting to improve the surface finish and paintability. Temper rolling (on line) gives the surface a microroughness well suited to automotive needs such as forming and painting (3-5).

Automotive quality galvanized coatings have a very thin intermetallic layer, no visible Fe-Zn intermetallic crystals, and minimal dross (Zn oxide particles or intermetallic) likely to damage surface appearance after forming (2). Obviously, scratches, dents, etc. are also not desired.

2.1.2.2 Galvannealed Coating of Steel Sheet

A galvannealed coating is produced by rapid heat treatment of the galvanized layer immediate after coating, in the temperature range of about 450-470°C. During the heating (or annealing) process, the pure zinc layer of the galvanized coating is transformed to a series of Fe-Zn alloy layers by diffusion. Compared to galvanized coatings, galvannealed coatings on steel are known to offer good spot weldability, paint adhesion, and perhaps corrosion performance (resistance to perforation) after painting due to the Fe-Zn alloy coating layers (6,7).

The Fe-Zn alloy layers have different iron concentrations and crystal structures. When present, gamma ($\text{Fe}^8\text{Zn}^{10}$, fcc) and gamma-1 ($\text{Fe}^3\text{Zn}^{10}$, bcc) phases, which are hard and brittle, form nearest the coating/steel substrate. The delta phase (FeZn^7 , hexagonal) is found adjacent to the gamma layer. The delta phase has a rectangular grain shape with the long axis perpendicular to coating/steel interface. Last is the zeta phase, which contains long columnar crystals (FeZn^{13} , monoclinic). Zeta phase has the lowest Fe-content and is soft in comparison to the delta and gamma phases. Generally, the surface of a galvannealed coating has a combination of zeta and delta phases, and is somewhat rough and irregular when compared to a pure-Zn coating.

A major concern for automotive producers is the susceptibility of the galvannealed coating to mechanical failure of the coating or interface (powdering or flaking) during press forming. It is generally accepted that adhesion of the coating is influenced by factors such as galvannealing conditions and substrate chemistry, although recent publications have also highlighted some effects of steel substrate surface topography (7,8). Generally, increasing the iron content (higher degree of annealing) decreases the roughness, but increases the severity of powdering and flaking.

Temper rolling is usually regarded as the primary method of controlling surface topography (9). The work roll roughness is selected to impart the desired coating surface topography, based on the initial coating roughness and the texture transfer characteristics between the work roll and the coated substrate. Work roll texturing methods are evolving from traditional shot blast textures to electro-discharge (EDT), Lasertex (Laser Texture) and most recently the EBT (Electron Beam Texture). These methods allow entirely new surface characteristics to be imparted. The use of EBT steel has been reported to be particularly beneficial to surface appearance after painting, and also to forming of galvannealed steel (9,10).

2.2 Classification of Imperfections

A surface imperfection is a flaw that ranges in size, morphology, and origin. Many authorities on the subject of surface imperfections attempted to classify imperfections according to appearance, and subsequent surface degradation. For instance, the American Iron and Steel Institute published an article entitled “Classification of Imperfections” (11) in 1985 that consisted of photographs, systematically documenting imperfection appearance. For the purpose of this study, a classification scheme was formulated to help categorize surface imperfections, highlighting formation mechanisms and associated size ranges. The classification scheme is presented in this section, followed by a review of imperfections pertinent to this research program.

The classification scheme used in the present research program primarily grouped imperfections according to occurrence in the life of the steel sheet, shown in Table 2.1(11). Surface imperfections can either occur during steel production and be present in the steel coil prior to shipment, or they can arise during the processes involved in automotive manufacturing. Each of these two localities was further divided into four equivalent subsets describing formation mechanisms for surface imperfections. Imperfections can be created by means of external contact, mechanical response, non-uniform coating deposition, or by a chemical reaction. Some examples are provided in Table 2.1.

Surface imperfections formed by external contact are a consequence of local contact with a hard object, while mechanical response include imperfections that arise as a result of macroscopic straining. Surface imperfections can also form due to non-uniform coating deposition. For example, in steel production, dross lines and sink roll marks form when zinc coatings are applied to steel substrates for improved corrosion resistance and enhanced paintability. Similarly, in automotive manufacturing, organic coatings are applied as liquids (paints) and are cured to become solid coatings. Surface imperfections can also result from chemical reactions that occur during non-uniform coating deposition and/or mechanical processes in steel production and automotive manufacturing.

In Table 2.1, distribution refers to how the imperfection appears to the naked eye. An imperfection was perceived in two dimensions (2-D) either as a point (0D), line (1D), or area (2D). Accordingly, specified size ranges only apply to how an imperfection with 3-D geometry was viewed in 2-D by the naked eye. Each imperfection considered in the table was described according to a single occurrence. Therefore, it is important to note that every imperfection could also become a linear or areal imperfection given a linear distribution or a broader presence across the surface area of the steel sheet. Some imperfections only occur in a linear or area distribution and were classified accordingly. For example, dross particles typically form imperfections in a linear fashion, and rarely occur singularly. Meanwhile, sink roll marks on sheets examined in this investigation

Table 2.1: Classification of surface imperfections for steel production and automotive manufacturing (11).

Locality	Formation Mechanism	Examples	Size Range			Distribution	Description
			< 0.5 mm	0.5 mm - 2 mm	> 2 mm		
Steel Production	External Contact	Scratch			X	Linear - 1D	IN
		Gouge (Dent)		X	X	Point - 0D	IN
		Roll Texture	X			Area - 2D	IN/OUT
	Mechanical Response	Non-uniform Rolling	X			Area - 2D	IN/OUT
		Entrapped Inclusions	X			Point - 0D	OUT
		Strain Lines	X			Linear - 1D	OUT
	Non-Uniform Deposition	Dross Particles	X			Linear - 1D	OUT
		Sink Roll Marks	X			Area - 2D	OUT
	Chemical Reaction	Outbursts	X			Point - 0D	OUT
		Stains and Spots		X	X	Point - 0D	OUT
Automotive Manufacturing	External Contact	Outdings		X		Point - 0D	OUT
		Scratch			X	Linear - 1D	IN
		Gouge (Dent)		X	X	Point - 0D	IN
		Roll Texture	X			Area - 2D	IN/OUT
	Mechanical Response***	Orange Peel	X			Area - 2D	IN/OUT
		Ridging		X	X	Area - 2D	IN/OUT
		Slip Lines	X			Linear - 1D	IN/OUT
		Twinning	X			Linear - 1D	IN/OUT
		Luders Bands		X	X	Linear - 1D	OUT
		Cracking	X			Area - 2D	IN
	Non-Uniform Deposition	Craters	X	X		Point - 0D	IN
		Dirt	X			Point - 0D	OUT
		Pinhole Gassing	X			Point - 0D	IN
		Solvent Pop	X			Point - 0D	IN/OUT
	Chemical Reaction	Lubrication			X	Area - 2D	OUT
		Oxidation			X	Area - 2D	OUT

occur throughout the surface area of steel sheet and are noted as an “area” imperfection. Imperfections in the table were also characterized by positive (out) or negative (in) deviation from a surface free of imperfections, where “out” and “in” describe the imperfection geometry. Imperfections can thus be hills or valleys, or a combination of both, protruding out from the surface or intruding below the nominal surface.

Only sampling of the overall specimen of imperfections from Table 2.1 was considered in this program. This sampling included: dents, dross particles, sink roll marks, scratches, spots, and outdings. The following discussion will concentrate on imperfections of mechanical and chemical origin.

2.2.1 Mechanically Induced Imperfections

Dents and scratches that occur by external contact can be induced mechanically in the laboratory. Controlled dent-type imperfections can be created by hardness indentations that result in localized plastic flow of material away from the indenter. It is useful to understand plastic flow of material under a hardness indenter and recognize the strain field shape to completely identify the imperfection morphology and its evolution with forming.

As a flat indenter presses normal to the sheet surface, maximum shear stress occurs on planes orientated 45° to the compression axis shown in Figure 2.3. The plastic zone beneath a hardness indentation is surrounded by elastic material and impedes plastic flow in a similar fashion as die constraint forces in a closed-die operation (12). This deformation zone geometry beneath the indenter is defined by the locus of planes of maximum shear stress, and is referred to as the slip line field (13).

Figure 2.3: The plastic flow of material under a hardness indenter in two-dimensions (13). F is the force applied to the indenter, and u represents the distance the indenter moves past the material surface.

Slip line fields are dependent on intrinsic properties of the material such as: crystallographic texture, grain size, and yield strength. The imperfection which is produced from a hardness indenter in this case, is defined by the local area of plastic deformation, which is constrained by a volume of elastic material.

2.2.2 Chemically Induced Imperfections

Chemically induced imperfections originate from non-uniform coating deposition and chemical reactions during coating processing. Specifically, non-uniform coating deposition encompasses processes like alloying and solidification, while chemical reactions during coating processing include preferential diffusion paths. Some examples are dross particles and sink roll marks. Dross particles are associated with alloying and solidification (non-uniform coating deposition), while sink roll marks are a consequence of entrapped particles that form due to alloying, solidification, and mechanical processes involved in galvannealing (non-uniform coating deposition coupled with mechanical response). These imperfections are exclusive to zinc based coatings and are discussed in further detail in the following sections.

2.2.2.1 Dross Particles

Dross particles are hard intermetallic compounds that result from steel dissolution and reaction with chemical components in the galvanizing pot during production of galvanized and galvannealed sheet steel (14). Dross particles are often categorized as top or bottom dross, referring to their location in the galvanizing pot. Top dross particles are Fe-Al rich intermetallic compounds (Fe_2Al_5 , γ particles) that float to the surface of the galvanizing pot. Bottom dross particles are Fe-Zn rich intermetallic compounds with a greater iron content compared to that of top dross particles, and consequently sink to the bottom of the galvanizing pot.

Top dross particles are frequently skimmed off the top of the bath, leaving bottom dross particles behind. Accordingly, bottom dross particles are usually large particles that formed by coalescence through time. Sizes of bottom dross particles can range from 50-250 μm , compared to top dross particles of 5-15 μm (14). This size difference between top and bottom dross particles can be viewed with electron microscopy, as seen in Figure 2.4 which shows a backscattered electron image of a top dross particle attached to a bottom dross particle at the interface.

Figure 2.4: A top dross particle attached to a large suspended bottom dross particle in the back of the galvanizing pot. Backscattered SEM micrograph taken at 200X (14).

In Figure 2.4, the darkest area is the Fe-Al rich top dross particle, the larger region, slightly lighter in shade, is the Fe-Zn rich bottom dross particle, which is surrounded by a matrix consisting of the Zn-Al alloy in the galvanizing pot. These large bottom dross particles remain suspended in the galvanizing pot as a consequence of agitation generated by the pot inductors and the moving steel strip. As a result, dross particles are deposited back onto the steel strip resulting in dross imperfections within the galvanized coating.

The severity of dross imperfections is dependent upon the processing conditions during galvannealing (14). Dross formation is temperature dependent as iron rapidly dissolves from the steel strip upon exposure to molten zinc, and is incorporated into the liquid zinc coating producing intermetallic phases at the substrate/coating interface. Iron dissolution occurs in three steps in the presence of liquid zinc: (1) Fe atoms separate from the strip surface, (2) then Zn atoms are separated by the intruding Fe atoms, and (3) Fe atoms mix with the Zn atoms (15). The associated enthalpy change is the sum of the enthalpies for the three sequential steps. The number of Fe atoms that are capable of dissolving in Zn is primarily dependent on temperature and composition, with the associated probability of a Fe atom escaping from the steel strip varying exponentially with temperature (15). These factors dictate whether dross particles form, and the resulting imperfection size within the zinc coating thickness following solidification.

2.2.2.2 Sink Roll Marks

(a) On Galvanized Steel Sheet

Sink roll marks examined in this study are a result of non-uniform coating deposition and mechanical response during steel production. As the steel strip tightly passes around the sink roll in Figure 2.2, free floating dross particles in the galvanizing bath are trapped between the roll and the strip, forming an imperfection (16). A typical sink roll mark of this type is included in Figure 2.5. Spots covering the surface of the sheet are bumps that formed from entrapped dross particles during steel production, and are distinguished with an arrow. This photograph shows a hot dipped galvanized surface with sink roll marks covering the surface of the steel sheet

Figure 2.5: A photograph of a sink roll mark on a hot dipped galvanized sheet surface, taken at 2X.

(b) On Galvannealed Steel Sheet

Sink roll marks (or striations) are often seen on the surface of hot-dip galvannealed steel sheet in the direction of processing through the hot-dip coating line. These darker bands run along the strip direction and correspond with grooves that are present in the submerged roll around which the steel sheet passes in the galvanizing bath. The surface appearance of the galvannealed product is compromised by this imperfection and it can render the finished galvannealed steel sheet product unsuitable for critical exposed automotive applications.

The variation in appearance of the bands is a direct result of the galvannealed strip having a non-uniform iron-aluminum interfacial layer (5). The greater the thickness of the iron-aluminum layer, the more difficult it is to nucleate and grow iron-zinc compounds. Since a three component system is involved (Zn, Al, Fe), there is no a priori necessity for stable planar interfaces between the growing phases (iron-zinc) and the zinc melt (17). A greater amount of initial iron-aluminum alloy formation results in more interface destabilization when zinc-iron compounds are formed during galvannealing. This results in rougher galvannealed coatings because of more non-uniform growth. The dark bands in the galvannealed steel sheet appear so because the iron-zinc coating in those areas has grown in a highly non-uniform manner, resulting in holes, or pits on the surface. This is a direct result of the thicker iron-aluminum layer that was formed in those regions. The lighter regions are seen to have a lesser number of pits or unevenness in the coating. Rougher areas of the coating appear darker in reflected light, and hence the final galvannealed steel sheet shows dark striations (5).

2.3 Automotive Paint Systems

2.3.1 Automotive Painting Process

As shown in Figures 2.6 and 2.7, automotive body panels are coated with a series of layers to add visual appeal and provide corrosion protection (18). On the exterior side of the panel, a phosphate conversion primer is applied for protection and to enhance paint adhesion. A series of paint layers including an electro-deposition (ED) primer, base coat and topcoat, comprise the complete exterior paint finish (additional layers might include a primer surfacer, or a chip resistant primer) (19). Below is an explanation of the role of each coating layer.

1. Metallic coating: metal surface preparation that enhances corrosion protection
2. ED-primer: primary corrosion protection & bonding surface for subsequent paint layers, electrolytically applied
3. Primer surfacer: serves as a leveler, allowing for a smooth paint finish and bonding surface for the base coat
4. Base coat: the color coat (primary visual effect)
5. Clear coat: provides environmental protection and enhances appearance

Figure 2.6: Schematic diagram of the automotive painting process (18)

Figure 2.7: Typical automotive coating system layers (19).

2.3.2 Coating Layer Properties

2.3.2.1 Phosphating

Phosphate conversion coatings are applied to coated sheet steel to provide an adherent base for subsequent paint finishing. In the case of automotive sheet steel, such a base is essential because the adhesion obtained without such a coating would not meet durability requirements (20).

A phosphating bath consists of an aqueous acidic solution containing phosphate ions and one or more bivalent metal cations. Zinc phosphate solutions are commonly used. The reaction occurs in two stages. Acid attack of the metal surface produces substrate metal ions to form a conversion coating on the substrate. The reaction is often accelerated by one or more oxidant ions, such as nitrates or chlorates (20).

There are two common phosphate structures that form on steels: phosphophyllite and hopeite. Phosphophyllite is a mixed hydrated phosphate of zinc and ferrous iron, crystallizing in the monoclinic system (20,21):

On bare steel, the zinc and ferrous ions come from the phosphate bath and from the acid attack of the steel, respectively. The name is applied to mixed phosphates of iron and zinc. This type of phosphate exhibits excellent adhesion to the substrate. Hopeite is a tertiary zinc phosphate crystallizing in the orthorhombic system (21):

The product forms in reaction with zinc surfaces, such as galvanized steel. The zinc ions originate partially from acid attack. Commercial phosphating solutions also contain additions of nickel, iron and /or manganese ions that regulate crystal nucleation and growth (22). Mixed metal phosphates result that enhance corrosion protection and paint adhesion (23).

The type of phosphate coating formed is dependent on whether one is processing bare steel or zinc coated steel. Only hopeite is developed on zinc-coated steel, while both compounds form on bare steel. With zinc-coated steel, no iron ions are available to form phosphophyllite. Interestingly, only hopeite is produced on zinc-alloy coated steel, such as zinc-iron (galvannealed steel sheet). Phosphate coating layers are very thin (approx. 250-300mg/ft²), and hence are not shown in the schematic Figure 2.7.

2.3.2.2 Electrodeposition Primer

Before applying the finish paint layers, a primer coat is applied by electrodeposition (ED-primer or E-coat). E-coat paints contain three major constituents-pigments, binders and solvent, the latter being principally water. The binder has the properties of a colloidal dispersion and the paint can be regarded as a low viscosity electrolyte. Analogous to a metal electro-plating cell,

the substrate is immersed in the water-borne paint and a current is applied to charge the paint particles electrically (24,25). They migrate to the cathodically charged substrate and coat the surface. After removal from the process tank, the primer coat is cured, at a temperature typically in the range of 160°C, to meld the particles into a continuous layer (24).

Crater imperfections are a concern in the primer process. Zinc-coated steels are more susceptible to this phenomenon than are uncoated materials. Cratering is characterized by visible crater-like imperfections (300 to 500µm in diameter) in the surface of the primer coat. Their formation is reportedly due to a localized dielectric breakdown of the E-coat film as it is deposited (25). The discharge energy displaces the organic film and the local heat prematurely cures the adjacent paint film, setting the resultant crater-like shape in place. Subsequent baking does not reflow the paint and heal the imperfection. Cratering can be avoided by designing the E-coat process to coat the steel properly below the threshold potential. For zinc-coated substrates, the threshold voltage at which the discharge can occur is about 250V, vs. 400 to 450V for cold-rolled steel sheet. However, galvanized steel shows cratering at a potential 100V lower than for pure zinc coatings, limiting their use for visible parts if the E-coating line has not been specially adapted (by adding an intermediate voltage plateau lower than the critical value, increasing the application temperature, adding solvent, etc. (25)). Cratering can also be minimized by the use of high-build primers to build thickness quickly, by control of voltage, AC ripple and E-coat bath temperature (25). Generally E-coat primer thickness is in the range of 15 to 30µm. This is sufficient to cover most features of the steel substrate, which has a roughness on the order of 1µm.

2.3.2.3 Primer Surfacer

The requirements placed on a surfacer have increased over the last few decades. In addition to the original surfacing function for the substrate, i.e. to mask surface irregularities, coupled with a requirement for good sandability, the emphasis today is increasingly on good stone-chip resistance. This compromise between hardness and flexibility is satisfied particularly well by polyurethane-modified systems, which accounts for their high current market share (26).

2.3.2.4 Base Coat and Clear Coat

Modern color coat technology consists of applying a color base coat followed by a clear topcoat. The total film thickness is in the range 50 to 75µm. Baking, or curing, finishes the multiplayer coating. The base coat is also formulated to fill minor surface imperfections. It is sometimes designed to provide additional corrosion resistance. While remote from the zinc layer; the appearance of the finish coats can still be affected noticeably by imperfections in the underlying steel. If craters are large enough or the paint is thin enough, leveling of these contours can be incomplete. Furthermore, if sufficient volatile contaminants remain in trapping sites on the substrate, rupture of the paint (i.e. “micro-pops”) can occur in the underlying primer. Despite their small size, on the order of hundreds of microns, the visual impact of such imperfections on a high gloss finish is very dramatic. (27).

2.3.3 Paint Properties

Adhesion quality and visual appearance of coatings are determined by film formation. Several factors, including application parameters, rheological behavior and interfacial properties interact in a complicated manner (28).

A coating must flow over the surface of the object to form a smooth surface, possibly a surface more smooth than surface of the object prior to the application of the topcoat. The coating is required to adhere to a vertical surface (vertical relative to gravity), or even "upside down". Thus, the viscosity of the paint must be low enough to permit flow, but high enough so that the paint doesn't "sag". A dramatic example of sag is a droplet that moves, or "runs" due to gravitational force. Issues such as leveling and sag, and others involving paint flow fall under the category of rheology (28).

2.3.3.1 Rheology

Rheology is the science of flow. A coating must flow over the surface and form a uniform topcoat with a smooth surface. Viscosity is expressed (29);

$$\text{Viscosity} = \frac{\text{Shear Stress (force per unit area)}}{\text{Shear Rate (velocity per unit thickness)}} \quad [2.3]$$

Surface tension minimization can be the driving force for wetting and viscosity is a resistance to leveling. Generally, the viscosity of a polymer decreases with increasing solvent concentration.

(a) Surface Tension

The liquid paint molecules usually experience the highest level of attraction for other molecules of the same liquid so packing takes place in a way that minimizes the contact with other surfaces. For the purposes here, these surfaces include both the solid surface a drop of the liquid is on, and the interface with the atmosphere. Molecules surrounded by like molecules tend to be more stable; the degree to which controls the magnitude of the surface tension (29).

(b) Wetting

Wetting involves the spread of a liquid over a surface. A low energy droplet spreads over a higher energy surface to minimize the surface free energy (for the high energy surface, coverage by a liquid allows more "energy release" than contact with gas molecules). The liquid continues to spread as long as the additional benefit from the high energy surface more than offsets the increase in energy to the liquid (spreading exposes more liquid to the atmosphere interface and this is the restricting factor to wetting).

Generally, aliphatic groups form materials with the lowest surface energy. Methyl groups give lower surface tensions than methylene groups. Water is a media with relatively high surface energy. The surface tension of a resin in solution increases as the solution concentration increases (30,31).

2.3.3.2 Curing and Polymerization

Curing of the coating is important. How fast the paint media (solvent, water, etc.) dry, whether there is a crosslinking reaction that forms the paint film, and the required time frame for the crosslinking reaction are all important factors. There are different classifications of dryness (31):

- dry to the point where the film has the appearance it will maintain for the life of the paint
- dry to the point where one can touch the film without leaving a fingerprint
- dry to point where mechanical tests of the paint would indicate complete curing. Naturally, the paint should not cure in the storage container, or during application.

2.3.4 Powder Coatings

Powder coatings, whether dry or slurry, are being used with excellent results for the painting of car bodies, either as primers, single-layer coatings or as a clear topcoat. In the United States, powder coatings are in full commercial use as primer surfacers at 13 General Motors and DaimlerChrysler plants (32). In Germany, BMW has also proven powder clearcoats to be a commercial success. In fact, there are millions of vehicles worldwide with powder coating as a layer of their body paint (32).

Ideally, appearance and other properties of a powder coating should be equivalent to those of a liquid coating of the same thickness. However, the unique requirements of a thermosetting powder coating impose restrictions on the binder system (the resins and curatives), making this objective difficult to achieve (33). Most importantly, powder coatings are attractive from an environmental stand point (reduced solvent emissions). Of course, they must be economically justifiable and practical in their utilization.

While liquid paint and powder coatings are similar in many respects, the technology used to prepare them is markedly different (34):

- Liquid paints are prepared by dispersion of the solids in a resin solution in a mill. Powder coatings are prepared by dispersion of the solid ingredients in a molten resin in an extruder.
- Paint dispersions must be stabilized to prevent pigment agglomeration and settling. Non uniform distribution of the components, such as floating and pigment agglomeration are not problems in powder coatings.
- Powders have only a short time after melting to coalesce and flow before cross-linking starts. Therefore, the melt viscosity, functionality and reaction rate of powder coatings must be carefully controlled.

2.3.4.1 Rheological behavior of powder coating

The rheological behavior of powder coating melts, as well as the influence of different composition and application parameters (e.g. particle-size distribution, film thickness, etc.) on film formation are briefly reviewed here.

Wetting of a substrate and leveling of a fluid film strongly depend on the surface tension of the coating. However, these two processes have opposing requirements in terms of surface tension. If the surface tension is too high, poor wetting occurs, which leads to imperfections such as craters. On the other hand, if the surface tension is too low, the leveling is adversely influenced (too much flow of paint), which leads to wavy surfaces known as orange peel (35). Moreover, surface flow can cause surface imperfections due to local surface-tension differences, known as the Marangoni Effect (36). These differences can arise from temperature gradients and local inhomogenities (e.g. contaminants). Therefore, the surface tension of a coating must be controlled carefully (35).

2.3.4.2 Glass Transition Temperature

One of the most important characteristics of a powder coating is its glass transition temperature (T_g). In simple terms, this is the temperature at which solid polymeric materials start to soften and flow. Powders with a low T_g can cause problems with "impact fusion," the buildup of solid deposits in powder delivery lines and venturi components of the spray equipment (34).

The T_g of a powder coating is affected by all of the resinous and polymeric ingredients present. It is generally agreed that the T_g of the binder resins used in thermosetting powders should be at least 50°C, preferably higher. The T_g must be high enough to prevent sintering and agglomeration during storage and shipping of the powder. To promote maximum flow and leveling, the T_g and molecular weight of the binder system should be low. In solvent born coatings, on the other hand, the binder system often has a T_g below room temperature and is a liquid in some cases (37).

The formulator must be aware of the T_g of additives and curatives and their effect on the T_g of the total system. The necessity of maintaining an adequate T_g for the formulated powder places constraints on both the resin manufacturer and the formulator. The T_g of a powder coating is usually measured by differential scanning calorimeter (DSC) techniques (34).

2.3.4.3 Powder Application of Automotive Coatings

Powder primers are already being applied successfully using conventional application technology (spray gun and paint booth) in automotive industry. The future direction is toward thinner films for powder primers and similarly with powder clearcoats. This will be accomplished by a combination of improvements in resin system rheology and through particle-size engineering. Improving the melt-flow rheology of the powder coating is one approach to providing smoother films at lower film thickness. The powder chemist is always being challenged to do this, but very often this cannot be achieved without some compromise in the powder coatings' physical stability. The consequence of lowering powder stability is the potential for application problems such as poor powder fluidization, inconsistent feed to the

spray guns and the potential for impact fusion--all of which make film-thickness control by the OEM more difficult (38).

The future use of powder clearcoats is linked to the further development of engineered, fine particle-sized powders. Some powder manufacturers are already applying finer particles in the production of both powder primers and powder clearcoat materials. In fact, these fine particles provide film smoothness and image clarity similar to liquid clearcoats. While this would appear to be a very simple solution to appearance issues with OEM powder systems, fine particle-sized powders do have some associated problems, especially in the area of application. It is typically found that as powder particle size is reduced, powder fluidization and transfer efficiency decrease (38).

(a) Powder for Basecoat/Clearcoat

The basecoat provides not only the color effect, but also contributes to the total system's appearance and performance. At present, most basecoats under consideration for automotive applications are waterborne paints that are flash dried before application of the powder clearcoat. Because of space limitations within an automotive plant, the OEM has to complete this flashing process in a very short period of time. However favorable these conditions are, there is the potential for the basecoat to retain some water and/or solvent, which the powder clear coat must accommodate in its film-forming process. If this does not happen, a defect known as popping will occur. Popping is not a new problem and occurs within liquid systems as well. The extent of this problem relates directly to the OEM's line conditions (total basecoat process time, application equipment, air velocities, humidity, oven type and temperature of bake/flash). It is also a function of basecoat chemistry, atomization characteristics, film thickness and the ability of the powder clearcoat to accommodate any evolving materials during its film-forming process (38).

A powder clearcoat finish must be equivalent or superior to liquid paint systems used on car bodies. The vertical and horizontal surfaces on the whole vehicle are important. It is relatively easy to produce a very smooth powder clearcoat film in the laboratory when the powder coating is baked in the horizontal position over a basecoat and primer that have also been baked in the same mode. However, the surface smoothness can be very different when all of the system components are baked in the vertical position. The variations of final appearance due to film-thickness inconsistencies and orientation during the bake cycle are as numerous as those with liquid clearcoat systems. Other factors, such as powder electrostatics and differences due to part grounding and basecoat color, add to the complexity of this issue. The point is that a powder clearcoat cannot be designed in isolation from other factors, since its ultimate performance will depend very much on the underlying layers.

Another known problem is yellowing of light colors. Yellowing of powder clearcoats over water-reducible basecoats has been reported widely. Most powder clearcoats are based on GMA (glycidyl methacrylate acrylic) resin chemistry that has a strong tendency to yellow in the presence of nitrogen-containing compounds. The presence of these materials in any of the underlying paint layers, or in the powder composition itself, can cause the clearcoat to yellow. This effect is more obvious in white and very light colors (34). The yellowing effect depends on film thickness and is also very much a function of bake temperature, so the baking temperatures cannot be adjusted simply to minimize yellowing. However, other film properties are also bake-

dependent. Therefore, for a product to be feasible for use in an OEM's plant, it must meet all requirements, including color over the range of bake temperatures likely to be experienced in a commercial application.

Again, a thorough understanding of the chemistries of the underlying layers is important. Powder manufacturers have extensively studied these interactions and worked with their raw-material suppliers in the advancement of new materials for powder clearcoats. At this point, high-reflectance white paints can be color-matched in a complete powder system, but these systems are less robust than liquids in terms of color variation due to bake temperature variations. Continuous improvement in this area of the technology is expected (26, 38).

2.4 Surface Profiling

It is important to be able to accurately characterize surface topography to relate surface geometry to appearance or performance. Surface texture is defined as the local deviations of a surface from the smooth ideal intended geometry of the part (39). Surface texture affects the functionality and reliability of certain components, and can be utilized as a diagnostic tool in monitoring processes. For instance, the effectiveness of grinding can be gauged by the surface texture of the ground part. The surface texture is essentially a “fingerprint” of a manufacturing process. It is very sensitive to changes in production, material composition, hardness, tool wear, strains in the material, and environmental factors, which all play a significant role in surface texture changes (39). Thus, surface profiling is essential in assessing imperfection evolution after forming and painting in this project, and is reviewed in this section.

“Surface texture includes closely spaced random roughness irregularities and more widely spaced repetitive waviness irregularities. American National Standard B46.1-1985 defines it as the repetitive or random deviation from the nominal surface that forms the three-dimensional shape of the surface. As such, it includes roughness, waviness, lay, and flaws...” (40) which are depicted in Figure 2.8.

Figure 2.8: Illustration of surface characteristics and terminology (39).

Roughness measures the closely-spaced irregularities left on a surface from a production process, such as machining. Waviness relates to the more widely-spaced irregularities and results from vibration, chatter, heat treatment, or warping strains. The lay of the surface refers to the direction of any predominant pattern in the surface texture as seen in Figure 2.8 (39).

A surface profile can be analyzed using descriptive parameters calculated from the profile data. There are three basic categories for these surface parameters: amplitude, spacing, and hybrid parameters. Peak heights and valley depths determine amplitude parameters (e.g. average roughness, R_a). Profile spacing deviations along the surface determine spacing parameters (e.g. peak count), and the combination of amplitude and spacing determine hybrid parameters (e.g. average wavelength) (39).

2.4.1 Basic Topography Components

Surface topography is a three-dimensional representation of geometric surface irregularities. A surface can be curvy, wavy, rough, or smooth depending on the magnitude and spacing of the peaks and valleys. Experimental data generated from a steel sheet sample is shown in Figure 2.9 illustrating these components. Here the original surface profile is given, along with the decomposition of the profile into roughness, waviness and form. The roughness, waviness, and form profiles sum together to create the original profile. Each profile component was obtained by filtering the original profile with appropriate wavelength cutoffs for roughness ($\lambda < 0.8$ mm), waviness ($0.8 < \lambda < 8$ mm), and form ($\lambda > 8$ mm) (41).

Figure 2.9: Decomposition of a profile into roughness, waviness, and form (41)

Roughness, waviness, and form are usually specified by typical wavelengths, which are used for filtering. Digital filters precisely separate desired ranges of wavelengths from the remainder of the profile. A roughness filter, or “high-pass” filter retains the short wavelength features in a profile ($\lambda < 0.8$ mm), and eliminates the waviness and form components. A waviness filter is referred to as a “band-pass” filter, and forces wavelengths within an interval ($0.8 < \lambda < 8$ mm) to pass through. Likewise, “low-pass” filters known as form filters allow only

long wavelength components to pass through ($\lambda > 8 \text{ mm}$), eliminating roughness and waviness from the profile (41). Specified wavelength cutoffs noted in parenthesis are common in North American steel production and manufacturing, where analog filters are also used.

2.4.2 Surface Measurement Techniques

To appreciate the influence of surface topography on manufacturing, it is essential to understand how surfaces are generated and the limitations of the instruments used to measure surface topography (42). This section provides some comments on techniques used to measure surfaces and the corresponding limitations of these instruments. The nature of surface topography and its role in manufacturing is discussed later in the section, “Elements of Surface Metrology”.

When measuring surfaces of engineered materials, there are several methods and issues to consider. Some common methods are summarized in Table 2.2, comparing spatial resolution, range and resolution in the z direction, and measurement frequency.

Table 2.2: Common methods used for surface characterization (42).

Method	Spatial resolution	z resolution	Range z	Frequency	Comments
Stylus	$0.1 \mu\text{m}$ to 1 mm	0.3 nm	$50 \mu\text{m}$	20 Hz	Contacts workpiece; easy to use; traceable
Optical probe	$0.5 \mu\text{m}$	$0.1 \mu\text{m}$	$20 \mu\text{m}$	$10 \text{ Hz} - 30 \text{ kHz}$	Non-contacting less traceable; with servo drive; range extended to $50 \mu\text{m}$
Heterodyne TIS	$2.5 - 200 \mu\text{m}$	$0.2 \mu\text{m}$	$0.5 \mu\text{m}$	10 Hz	Requires computer unravelling
Scatterometer diffraction	$0.6 - 12 \mu\text{m}$ $\sim 10 \mu\text{m}$	1 nm	$0.1 \mu\text{m}$ $\lambda/8$ 100 nm	seconds seconds	Resolution depends on aperture; insensitive to movement
TEM	2 nm to $1 \mu\text{m}$	$2 \mu\text{m}$	100 nm	minutes	Replication needed can destroy surface
SEM	10 nm	2 nm	$2 \mu\text{m}$	minutes	Vacuum needed
STM	2.5 nm	0.2 nm	100 nm	minutes	Vibration-free mounting
Normarsky	$> 0.5 \mu\text{m}$			minutes	Needs interpretation and certain minimum reflectivity
Capacitance	$2 \mu\text{m}$	1 nm	50 nm	2 kHz	Needs conductors
Interferometry	$2 \mu\text{m}$	1 nm	$1 \mu\text{m}$	minutes	

Note that ultrasonic pneumatics are suitable for rougher surfaces only.

The resolution and range in the z direction is of particular importance in acquiring surface topography data, and is primarily dependent on the operating transducer system. For example, a stylus instrument uses an inductive transducer that works by variable reluctance and coupling of two coils. Advantages include: a small size; high resolution; and insensitivity to changes in environment. Disadvantages include: inaccuracies increase proportionally with range due to complex coil construction; and electrical frequency response is limited to a quarter of the modulation frequency. Comparatively, an optical interferometric system operates by periodic light intensity variations with several advantages: digital output; high accuracy over range; the electrical zero can be adjusted; and the resolution is independent of the range. Disadvantages include: thermal instabilities due to variety of optical components, possible count loss if a

maximum velocity is exceeded, and high instrument cost due to the coherent light source and precision optics often used. Capacitance uses a variable area conversion that can be used at high temperatures, but is not capable of measuring large depths or heights. Accuracy of capacitance methods is affected by changes in geometry and condition of electrodes, and the electrical frequency response is limited to a quarter of the modulation frequency (42). These are just a few examples of the instrumentation found in surface metrology today.

Generally, stylus methods are more common in steel production and manufacturing facilities, due to versatility, and low cost. A schematic of a typical stylus instrument is shown in Figure 2.10. In this system the solenoid is coupled with a linear variable differential transducer (LVDT) which follows the vertical motion of the stylus, while the step motor moves the stage laterally between measurements. Stylus instruments have measurement capability to cover a large range of roughness ranges and processes. However, there are drawbacks to these conventional instruments which are used to measure roughness, waviness, and form. Some drawbacks are (42): slow measuring technique; the stylus force can potentially damage the surface of interest; and a limitation on measuring areas. Advantages include (42): versatility to accommodate a wide diversity of shapes; high range to resolution in the vertical direction; and a high spatial bandwidth.

Figure 2.10: Schematic of a typical stylus system (42).

Most optical methods use variations of a Michelson or Mirau interference microscope. The microscope consists of an objective interferometer and a beam splitter which is mounted on a piezoelectric stage and driven by a computer (42). A basic Michelson interference microscope is illustrated in Figure 2.11 (43).

The diagram illustrates a three-plate contact system. It consists of three main components: a left plate labeled 'R', a central plate labeled 'H', and a right plate labeled 'L'. A vertical line labeled 'G' is positioned to the left of plate 'R'. A horizontal line labeled 'O' is positioned below plate 'H'. A vertical line labeled 'P' is positioned to the right of plate 'L'. The plates are shown in cross-section, with hatching indicating different materials or layers. The central plate 'H' has a diagonal line across its face, and the right plate 'L' has a hatched section on its right side.

2.4.3 Surface Measurement Utilizing Optical Interferometry

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Figure 2.12: An interference microscope.

The Wyko NT2000 optical interferometer uses two techniques to measure surface heights. Phase-shifting interferometry (PSI) allows the user to measure very smooth surfaces, while vertical scanning interferometry (VSI) is used for rough surfaces and steps. While operating in the PSI mode, a white light beam is filtered and passed through the interferometer objective. The interferometer beamsplitter reflects half of the incident beam to a reference surface within the interferometer. The other half travels to the specimen surface. The beams reflected from the specimen test surface and the reference surface later recombine to form interference fringes, with spacing that is a function of wavelength of the incident light and slope of the test surface. These interference fringes are alternating light and dark bands that appear when the surface is in focus (39).

During measurement, a piezoelectric transducer linearly moves the reference surface a small, known vertical displacement to cause a phase shift between the objective and reference beams. The system records the intensity of the resulting interference pattern at many different relative phase shifts, and then converts the intensity to phase data by integrating the intensity data. The data are processed, ambiguities between adjacent pixels are eliminated, and the relative surface height, h is calculated from the phase data in this manner:

$$h(x, y) = \frac{\lambda}{4\pi} \phi(x, y) \quad [2.4]$$

where λ is the source beam wavelength, and $\phi(x,y)$ represents the phase data (39).

The VSI mode operates in a similar manner, with the exception that the white light source is not filtered, and the system measures degree of fringe modulation (coherence), instead of phases of interference fringes. While operating in the VSI mode, the reference arm containing the interference objective moves vertically, scanning various heights. White light has a short coherence length, so interference fringes are present over shallow depths and approach a peak value as the sample is translated through focus.

In an interference microscope, the bi-directional reflectance distribution function (BRDF) describes the amount of power scattered at various angles when light impinges on a surface. The scatter angles are relative to the direction of specular reflection. The BRDF is dependent on the angle of incidence and the incident wavelength, as well as reflectance and roughness of the material. A ray diagram shows the angles used in BRDF, in Figure 2.13. Both the angle of incidence, θ_i , and the scatter angle, θ_s , are measured relative to the Z-axis. Out of plane scatter, ϕ_s , refers to the angle at which scattered light is detected and is measured relative to the plane of incidence (Z-axis). The angle of incidence and the spatial sampling of the objective determine the range of scatter angles over which BRDF is “collected”. BRDF is directly correlated with surface reflectivity functions, and is characterized by the following equation (39):

Figure 2.13: Angles used in BRDF (39).

$$BRDF(f_x, f_y) = \frac{16\pi^2}{\lambda^4} \sqrt{R(\theta_i)R(\theta_s)} \cos\theta_i \cos\theta_s PSD(f_x, f_y) \quad [2.5]$$

where $R(\theta_i)$ and $R(\theta_s)$ are surface reflectivities as a function of θ_i and θ_s respectively, and the wavelength, λ . Position f_x , and f_y are determined by :

$$f_x = \frac{1}{\lambda} (\sin\theta_s \cos\theta_s - \sin\theta_i) \quad [2.6]$$

$$f_y = \frac{1}{\lambda} \sin\theta_s \sin\phi_s \quad [2.7]$$

The out-of-plane scatter angle can range from -45° to $+45^\circ$, where 0° is in the plane of incidence, and the incidence angle can range from 0° to 85° , where 0° corresponds to the light source pointing directly onto the surface.

System performance of the Wyko NT2000 depends on the measurement technique. The ranges and corresponding vertical resolution for the two operating modes are included below in Table 2.3, as reported by the manufacturer.

Table 2.3: Range and vertical resolution for PSI and VSI modes (39).

Mode	Range	Vertical Resolution
PSI	160 nm	3 ?
VSI	500 μm	<1 nm

Range refers to the greatest vertical distance the profilometer can accurately measure in a single image. So in the VSI mode, the maximum height difference resolvable between adjacent pixels is 500 μm . Resolution is defined as the smallest step height that the profilometer can accurately measure. This research program dealt with surface heights and lateral measurements of surface features, so both vertical and lateral resolution were important. Lateral resolution is a function of the magnification objective and the detector array size chosen by the user, and the number of pixels recorded for a given surface area.

2.4.3.1 Graphical Display and Analysis Functions

The software (supplied by the WYKO manufacturer) provides a large number of graphical output display files that allow the user to produce meaningful data from test results. The default file is called Contour Plot, a color-coded topographical map of the surface which also includes the basic statistics from the measurement (see graphical Figure 2.14). 2D Analysis displays profiles taken from the surface height data. The user can adjust the locations and size of the region from which the profiles are taken. 3D Plot creates a 3-dimensional rendering of the

surface data. The user can manipulate the apparent view angle, lighting, rotation, etc. of the surface.

2.4.3.2 Stitching

If the measurement area is larger than a single field of view (2.47mm X 1.88mm, 5x lens), the WYKO NT 2000 provides a Stitching option to accommodate this. Stitching allows data from several measurement areas to be "stitched" together to form one dataset. This can be done automatically or manually.

Figure 2.14: Graphical display results from WYKO NT 2000.

2.4.4 Elements of Surface Metrology

Surface metrology relates the measurement of surfaces to manufacture of these surfaces and their influence on performance (42). Surface metrology is defined by three elements: generation, characterization, and function (44). These three elements are interdependent and important in many industries for surface engineering, process control, and quality control. Figure 2.15 shows this triangle and how each factor is related to one another.

Figure 2.15: The surface metrology triangle: generation, characterization, and function (44).

Generation describes methods by which surface topography is generated. For instance, grinding a surface, applying a coating such as zinc onto a steel substrate for corrosion protection, or depositing a TiN coating onto a steel substrate for wear resistant applications. Every surface is generated to serve a function in manufacturing. Wear resistance, corrosion resistance, and paintability are just a few examples. Surface characterization can either be utilized to monitor a process, or it can be used to assess the functionality of a surface.

2.5 Visual Inspection of Automotive Painted Surface

2.5.1 Optical Properties of Imperfections

Overall appearance of an automotive coating is a combination of the effects of spectral and geometrical factors, seen by the eye and interpreted by the brain. Spectral characteristics depend on the wavelengths of light incident on and reflected from objects. A common set of terms to describe the way these characteristics are perceived by the human eye is hue (perception of color), lightness, and saturation. In contrast, geometrical characteristics that contribute to appearance do not depend on these spectral characteristics. This group includes gloss, and metallic effects. These phenomena are determined by such geometrical factors as the location of the light sources illuminating the objects, the surface characteristics of the objects, and the location of the eye of the observer (45).

2.5.1.1 Visual Acuity of the Human Eye

The eye has a visual acuity threshold below which an object will go undetected. This threshold varies from person to person but as an example, a person with normal 20/20 vision can be considered. Figure 2.16 shows a picture and cross section of the human eyeball. As light enters the eye through the pupil, it passes through the lens and is projected on the retina at the back of the eye. Muscles called extra ocular muscles move the eyeball in the orbits and allow the image to be focused on the central retinal region or fovea shown in Figure 2.17 and 2.18.

The retina is a mosaic of two basic types of photoreceptors, rods and cones (Figure 2.19). Rods are sensitive to blue-green light with peak sensitivity at a wavelength of 498 nm (Figure 2.19), and are used for vision under dark or dim conditions. There are three types of cones that give us our basic color vision; L-cones (red) with a peak sensitivity of 564 nm, M-cones (green) with a peak sensitivity of 533 nm, and S-cones (blue) with a peak sensitivity of 437 nm. Cones are highly concentrated in a region near the center of the retina called the fovea region. The maximum concentration of cones is roughly 180,000 per square mm in the fovea region and this density decreases rapidly outside of the fovea to a value of less than 5,000 per square mm. There is a blind spot associated with the location of the optic nerve which is void of any photoreceptors.

Figure 2.16: Picture and cross section of human eyeball (46).

Figure 2.17: Cross section of human head from top view (46).

Figure 2.18: Picture of human retina (46).

Figure 2.19: Schematic and color sensitivity of retina (46).

The standard definition of normal visual acuity (20/20 vision) is the ability to resolve a spatial pattern separated by a visual angle of one minute of arc. Since one degree contains sixty minutes, a visual angle of one minute of arc is $1/60$ of a degree. The spatial resolution limit is derived from the fact that one degree of a scene is projected across 288 micrometers of the retina by the eye's lens (46).

In this 288 micrometers dimension, there are 120 color sensing cone cells. Thus, if more than 120 alternating white and black lines are crowded side-by-side in a single degree of viewing space, they will appear as a single gray mass to the human eye. With trigonometry it is possible to calculate the resolution of the eye at a specific distance away from the lens of the eye (Figure 2.20).

For the case of normal visual acuity the resolved viewing angle θ is $1/60$ of a degree in Figure 2.20. By bisecting this angle we have a right triangle with angle $\theta/2$ that is $1/120$ of a degree. Using this right triangle it is easy to calculate the distance $X/2$ where X is the resolution for a given viewing distance d (46).

$$X/2 = d (\tan \theta/2) \quad [2.8]$$

When visually inspecting an object for a defect such as a crack the distance d might be around 12 inches. This would be a comfortable viewing distance. At 12 inches, the normal visual acuity of the human eye is 0.00349 inches. What this means is that if alternating black and white lines were each 0.00349 inches ($88.6\mu\text{m}$) wide (or less), they would appear to most people as a mass of solid gray (46).

Figure 2.20: Schematic showing resolution of the eye at a specific distance away from the lens of the eye (46).

2.5.1.2 The Human Eye's Response to Light

The three curves in the Figure 2.21 show the normalized response of an average human eye to various amounts of ambient light at different wavelengths. The shift in color sensitivity occurs because two types of photoreceptors, cones and rods, are responsible for the eye's response to light. The cones respond to light differently under different lighting conditions. As mentioned previously, cones are composed of three different photo pigments that enable color perception. The curve on the right shows the eye's response under normal lighting conditions and this is called the photopic response. This curve peaks at 555 nanometers, which means that under normal lighting conditions, the eye is most sensitive to a greenish yellow color. When the

light levels drop to near total darkness, the response of the eye changes significantly as shown by the scotopic response curve on the left. At this very low light level, sensitivity to blue, violet, and ultraviolet is increased but sensitivity to yellow and red is reduced. The rods are most sensitive at this level of light. Rods are highly sensitive to light but are comprised of a single photo pigment, which accounts for the loss in ability to discriminate color. The heavier curve in the middle represents the eye's response at the ambient light level found in a typical inspection booth. This curve peaks at 550 nanometers, which means the eye is most sensitive to yellowish green color at this light level. Fluorescent penetrant inspection materials are designed to fluoresce at around 550 nanometers to produce optimal sensitivity under dim lighting conditions (47).

Figure 2.21: The normalized response of an average human eye to various amounts of ambient light (47).

2.5.1.3 Contrast Sensitivity

When conducting a visible inspection, the contrast sensitivity of the eye is important. Contrast sensitivity is a measure of how faded or “washed out” an image can be before it become indistinguishable from a uniform field. It has been experimentally determined that the minimum discernible difference in gray scale level that the eye can detect is about 2% of full brightness (47).

Contrast sensitivity is a function of the size or spatial frequency of the features in the image. However, this is not a direct relationship as larger objects are not always easier to see than smaller objects when contrast is reduced as demonstrated by the image in Figure 2.22. In the figure, the luminance of pixels is varied sinusoidally in the horizontal direction. The spatial frequency increases exponentially from left to right. The contrast also varies logarithmically from 100% at the bottom to about 0.5% at the top. The luminance of peaks and troughs remains constant along a given horizontal path through the image. If the detection of contrast was dictated solely by image contrast, the alternating bright and dark bars should appear to have equal height everywhere in the image. However, the bars seem to be taller in the middle of the image (48).

Figure 2.22: Image for contrast sensitivity test (48).

2.5.2 Surface Characteristics of Imperfections

Real surfaces are not perfectly smooth and have various imperfections. An imperfection generally is small in comparison with the resolution of an observer's eye, and it scatters light into a large solid angle, in transmission or reflection (Figure 2.23) (49).

Observer in reflection

Figure 2.23: Observation of an imperfection (49).

Figure 2.23 was the schematic explaining optical condition for imperfection on flat surface. The expression for light-flux received by the observer ($\Delta\Phi$) is :

$$\Delta\Phi = \int_{\Delta\Omega' \text{ observer}} d\Omega' \cdot \int_{\Delta\Omega \text{ light source}} dL(\Omega) \cdot RS(\Omega) \cdot D(\Omega, \Omega') \cdot d\Omega \quad [2.9]$$

where $\Delta\Omega$ and $\Delta\Omega'$ are the solid angles representing sample illumination and observation, respectively. $L(\Omega)$ is the distribution of intensity of the illumination. RS represents either the reflection (R) or transmission (T) coefficient of the sample material, and D is the scattering function of the imperfection. This expression becomes simpler if:

$L = L_0$ (sample is illuminated by an integrating sphere), and if $\Delta\Omega'$ is small in comparison with the angle of illumination, then

$$\Delta\Phi \approx L_0 \Delta\Omega' \int_{\Delta\Omega} D(\Omega) RS(\Omega) d\Omega \quad [2.10](49)$$

defect+material = D^*

light source ← $\Delta\Omega$ observer = 0

where $\Delta\phi$ = light flux received by the observer

L_o = light source

O = Observer

D^* = defect + materials

which can be further simplified as (70)

$$\Delta\phi = L_o O D^* \quad [2.11]$$

This equation shows that the reflected intensity is a strong function of the amount of illumination and the viewing angle.

Several phenomena play a role in the distribution of light reflected or scattered from a surface (49-51). The most important effects are the variations of surface orientation and shadowing (Figure 24-25) (50). The variation of surface orientation (represented by the surface normal in Figure 2.24) generally occurs on two scales. A fine scale variation is present that represents the basic surface roughness. In the case of surface imperfections, there also exists a more gradual variation corresponding to the surface distortion associated with the imperfection. The combined variation is illustrated in Figure 23 (50).

Figure 2.24: The reflected geometry (50).

Figure 2.25: The variation in surface height for a rough surface in the vicinity of a surface imperfection (50).

In the case of shadowing, portions of the surface are occluded by variations in the surface due to the imperfection. This condition is shown in Figure 2.26 for a pit-type imperfection.

Figure 2.26: The shadowing of the surface due to an imperfection (50).

2.5.3 Measurement and Evaluation of Imperfections

2.5.3.1 Visual Inspection

Effective inspection of automotive paints and finishes requires a lighting environment that produces strong luminance edges which can be seen in reflection from the surface of an automobile (52-54). Paint imperfections such as embedded dirt, runs, sags, and dents reflect light from many different angles. For an imperfection to be visible to an inspector, the imperfection and its immediate background must have different luminances (55). To achieve this, surfaces in inspection rooms must have differences in luminance that can be seen in reflection. Each automaker tries to enhance imperfection inspection by changing the lighting design and lighting surfaces of the inspection environment. Conditions for visual inspection from one automotive maker are listed below (55):

- Inspectors find more imperfections when light reflects onto the surface of a vehicle from a patterned wall rather than from a plain wall. A patterned wall provides many luminance "edges" that produce specular highlights on the imperfection.
- Inspectors find more imperfections with darker paints than in lighter paints. The pigments of the lighter paints reflect more ambient illuminance, reducing the contrast of the specular highlights on the imperfection.
- luminance In an optimal inspection environment, the walls, ceiling, and floor are covered with a strong pattern, ensuring that imperfections on the vehicle surface will always be close enough to a luminance edge to be detected.
- The larger the reflected highlight, the more likely inspectors are to find imperfections. Highlight size depends on the angular distance from the lamp image.

2.5.3.2 Measurement of Gloss

Other measurement techniques include gloss and distinctness of image (DOI) measurements. These two methods measure reflective properties of surfaces.

The modern study of gloss measurement began in the mid-1930's, when Hunter began to design and build a gloss meter. His instruments were the fore-runner of those in use today, and very few fundamental changes have taken place since then. Table 2.4 (45) lists various types of gloss and DOI techniques.

Table 2.4: Types of gloss and DOI measurement (45).

Type of Gloss	Type of Judgment	Angular Range
Specular	Intrasample comparison of brightness of specular reflection	Specular angle for both samples
Distinctness of Image	Distinctness of a reflected image	Specular angle plus or minus 0.5°
Contrast	Intrasample comparison of brightness on and off the specular angle. Usually no image formed	From specular to an angle far away from specular
Haze	Reflected light spreading out from specular reflection	From specular to 5 ° to 15 ° away from specular
Sheen	Distinctness of image or contrast evaluated at a near-grazing angle	Specular, but evaluated at 85 ° from the normal
Macroscopic	Directionality, texture, and other easily visible defects	Any

2.6 Forming Induced Imperfections in Brittle Coatings

During forming processes, the steel substrate in coated sheet steels deforms by plastic flow without necking or fracture at moderate strains. Correspondingly, the zinc coating accommodates substrate strain by plastic flow or by the development of an array of through-thickness cracks. Particularly during biaxial stretching and tensile testing, the coating cracks to accommodate the strain mismatch. Cracks can nucleate at the substrate/coating interface and propagate towards the coating free surface. A strain gradient is produced from the center of the cracked region, to the uncracked region, and a local shear force develops at the interface (56).

This mechanical failure observed in brittle coatings is a consequence of the coating microstructure and texture. A cross-section of a galvanized sheet steel reveals the layered array of phases typically seen in a brittle galvanized coating.

The delta phase in a galvanized coating has a hexagonal closed packed (HCP) structure and is intrinsically brittle due to a limited number of slip systems available at room temperature. This region usually comprises the largest fraction of the coating microstructure and is the most vulnerable to coating failure. However, cracking also has been observed at the gamma/substrate interface and the delta/gamma interface. This phenomenon is a result of the iron gradient originating at the steel substrate, propagating through the coating microstructure. The degree of cracking and coating failure depends on the presence of a gamma layer, the zeta to delta ratio, and the iron gradient within the layers of the microstructure (56). Cracking within brittle coatings is an example of imperfections which form as a consequence of mechanical response during automotive manufacturing, classified in Table 2.1.

2.7 Surface Roughness Changes During Forming

Surfaces are also influenced or altered as a consequence of forming. A surface roughens due to the different tendencies of grains to rotate, thicken or thin during plastic deformation, which is a result of constrained plastic flow during stretch-forming operations (e.g., drawing, stretching). Consequently, there are multiple interpretations concerning texture effects during forming. This section will review briefly how an increase in roughness affects appearance on a macro-scale for different materials in addition to mechanisms for roughness increases in zinc coated sheet steels.

On a macroscopic scale, there are forming-induced imperfections that are visible with the naked eye. Some examples are: orange peel, ridging, and stretcher strains. Orange peel is visible on the order of the grain size, and is a result of different orientations within neighboring grains which are pronounced after stretching in a coarse grained material. An example of orange peel is pictured in Figure 2.27.

Figure 2.27: Example of orange peel. American Iron and Steel Institute. Dimensions are in inches (57).

Orange peel is common to any material with an initial coarse-grained structure and is observed in materials where the free surface does not come in contact with tooling during stretch-forming operations.

Ridging is a similar surface phenomenon and is common in ferritic stainless steels and some aluminum alloys. During sheet processing, grains tend to preferentially orient themselves and elongate in the rolling direction. During deformation, grains of a particular orientation are more favorable to thinning in the through-thickness of the sheet compared to those of other orientations. This process induces compatibility issues at the grain boundary interfaces and results in an incremental increase in surface roughness as large ridges develop parallel to the rolling direction (57). Imperfection visibility is similar to that of orange peel.

Stretcher strains refer to incomplete Luders bands that form on a sheet surface during forming and are a consequence of non-uniform deformation. Stretcher strains are obvious at low strains, as pictured in Figure 2.28. They develop in materials that have more prominent yield points, which may develop as a consequence of strain aging (57).

Figure 2.28: Stretcher strains on a 1008 steel sheet stretched past the yield point (7/8 size) (57).

The phenomena are relevant because a portion of the work that follows addresses effects of forming strains on surface imperfections, and subsequent imperfection visibility before and after painting. Automotive sheet steel samples containing various imperfections were stretch-formed in a series of experiments and systematically analyzed with a combination of optical interferometry, scanning electron microscopy, light optical microscopy, and x-ray diffraction techniques.

3.0 OBJECTIVES

The primary objectives of this research were as follows:

1. Implement measurement capabilities to objectively assess imperfection characteristics.
2. Understand the relationship between surface imperfection characteristics before painting, and the detectability or visibility of these imperfections after painting.
3. Understand the influence of forming strains on imperfection appearance, and the sensitivity of imperfection appearance to different paint system characteristics.

4.0 EXPERIMENTAL PROCEDURES

The parameters for the painting study were selected based on current industrial practices and experimental needs. Electrogalvanized, hot dip galvanized, and galvanized zinc coated sheet steels were selected due to their extensive use among automotive manufacturers around the world. Laboratory imperfections of dent-type, scratch-type and raised morphologies were created in an attempt to replicate common imperfections seen in industry. Industrial samples of hot dip galvanized and galvanized material containing “real” imperfections are also systematically painted for comparison to the laboratory results. Testing and analysis of laboratory-induced imperfections coupled with industrially relevant imperfections should then facilitate a comparison between imperfection origin.

A series of experiments for each imperfection was conducted in the laboratory as follows:

- a) Examine each class of surface imperfections using the optical microscope and SEM/EDAX. Identify the characteristics and origin of the industrial imperfections.
- b) Measure the surface topography of each imperfection using a 3-D profilometer, and record its location in manner suitable to identify its exact location.
- c) Paint the representative imperfections with a suitable automotive system. A paint system especially sensitive to surface blemishes is included for “baseline understanding”, in addition to the modern system incorporating heavier film builds which might be expected to mask imperfections more effectively. A horizontal panel orientation is used for paint application, as these are the high reflectivity surfaces of greatest interest (hood, deck lids, etc).
- d) Inspect the painted surfaces visually (and quantitatively if possible) to assess visibility, or visual severity, of the imperfections after painting.
- e) Quantitatively characterize the topography of the imperfection after painting using 3-D profilometry.
- f) The data are then analyzed to relate the initial imperfection characteristics to their visibility and dimensions after painting. The results are interpreted to identify potential for developing surface imperfection acceptance criteria for exposed quality sheet products and to understand the evolution of topography during painting.

The experimental procedures are detailed separately for the laboratory-induced imperfections, and for the commercially-produced imperfections provided by participating companies.

Parameters for the evaluation of the forming response of imperfections were selected based on current industrial practices and experimental needs. Electrogalvanized, hot dipped galvanized, and galvanized zinc coated sheet steels were selected due to their extensive use among automotive manufacturers around the world. Laboratory imperfections of dent-type and raised morphologies were created in an attempt to replicate common imperfections seen in

industry. Accordingly, modified hardness indenters were fabricated with 2° and 4° face angles in effort to produce imperfections with shallow geometries. Likewise, hard particles were chosen to create raised imperfections during stretch-forming experiments (the particles were located at the sheet/tooling interface during forming). A die insert was designed to reproduce curvature and forming conditions typical of automotive body panels. Finally, a strain matrix was developed to examine changes in imperfection morphology as a function of strain.

Industrial samples of hot dipped galvanized and galvanized material containing “real” imperfections were systematically formed and painted for comparison to laboratory imperfections. A series of three independent experiments for each imperfection was conducted in the laboratory as follows:

1. Form a panel with strain typical of automotive body panels, and characterize the forming effects on an imperfection by collecting surface data before and after forming.
2. Form a panel with strain equivalent to experiment 1, characterize the combined effects of forming and painting on an imperfection by collecting surface data before forming, after forming, and after painting.
3. Incrementally strain a sample and monitor the topographical change in an imperfection at forming strains until fracture.

Testing and analysis of laboratory-induced imperfections coupled with industrially relevant imperfections facilitated a comparison between imperfection origin and mechanisms of surface changes during forming.

4.1 Laboratory Induced Imperfection Samples for Painting Studies

The following sections will systematically explain the laboratory sample panel and imperfection design for painting studies. The laboratory panel has various shapes of simulated imperfections to assist quantitative analysis.

4.1.1 Materials

Four substrate materials were obtained, including hot-dip galvanized (GA), hot dip galvanized (HDG), electrogalvanized (EG) and cold rolled (CR). While the primary focus of this project is on galvanized, it was considered useful to examine differences between the other substrate materials. Substrate chemistries, mechanical and coating surface properties of materials are provided in Table 4.1 and 4.2, based on results supplied by the manufacturers. There were no data included for the CR substrate material, provided by ACT Laboratories.

Table 4.1: Substrate chemical compositions in weight percent.

Coating	C	Mn	P	S	Si	Cu	Ni	Cr
GA	0.003	0.130	0.009	0.009	0.010	0.024	0.010	0.029
HDG	0.0056	0.089	0.009	0.008	0.008	0.014	0.015	0.021
EG	0.040	0.180	0.013	0.009	0.008	0.015	0.015	0.031

Coating	Mo	Sn	Al	N	Cb	V	Ti	B
GA	0.005	0.004	0.047	*	0.012	*	0.031	*
HDG	0.003	0.005	0.059	0.0032	<0.005	0.005	0.085	<0.0002
EG	0.004	<0.002	0.053	0.007	<0.005	<0.002	0.003	<0.0002

Table 4.2: Material properties for GA, HDG, and EG sheet steels.

Coating	Thickness	Coating Weight	Coating	YS	Total El	n	Ra	PPI
	(mm)	(g/m ²)	Thickness (μm)	(MPa)	(%)		(μm)	(50μin)
GA	0.8890	60.6/58.7	6.3	155.14	43.1	0.230	0.94	231
HDG	0.70104	60.4/53.9	7.0	164.10	44.3	0.228	0.76	113
EG	0.70104	68.9/67.1	8.4	194.44	42.8	0.210	0.86	172

* Not stated from the respective steel producer.

4.1.2 Panel Design

The panel design for painting studies is presented schematically in Figure 4.1. Each panel is 4"x12", and incorporates 30 dents ("A"), 30 scratches ("B"), and 6 "outding" raised imperfections ("C"). Using specially designed indenters, along with a scratch tester (ST-2200, Teer Coating Ltd.) and hardness tester, features with a variety of depths, widths, and sidewall slopes were successfully created. The cross-section of each series of imperfection is illustrated schematically next to or above each series. The first set of imperfections ("A") is a series of dents that have different depths, shapes and slopes. The second set of imperfections ("B") is a series of linear scratches with different sidewall slopes, depths and widths. The third set of imperfections ("C") is a series of raised imperfections or "outdings" that have different height. Figure 4.2 shows an example 3D plot from each series of imperfections.

Figure 4.3 shows schematic drawings of the cross-section of three conical indenters. The tips have different face angles of 100°, 70° and 40°. Changing the tip geometry of the indenter and the load on the indenter allowed different depths and shapes of surface imperfections to be created. Dent-type imperfections with different depths were created by indenting normal to the sheet surface with different indenter loads in the hardness testing machine. Translation of the specimens (using a scratch tester) while the indenter was under load allowed different scratch

imperfections to be created. Finally, the raised imperfections or “outdings” were created using hardness indentions from the reverse side of the sheet, supporting the sheet over a backing plate containing a circular hole centered on the indentation.

Figure 4.1: Schematic illustration of laboratory sample panels.

Figure 4.2: 3D plot of example imperfection types.

Figure 4.3: Schematic cross-sections of conical indenters with machined face angles (θ) of 110° , 70° and 40° .

4.1.3 Painting

For the painting experiments used with the laboratory panels, seven “imperfection” panels in each of the four substrate sets were sent to PPG R&D and painted with a PPG designed coating system. The seven panels included the following conditions:

1. Pretreatment
 2. Pretreatment + ED5100 (e-coat primer)
 3. Pretreatment + ED5100 + Primer Surfacer
 4. Pretreatment + ED5100 + Primer Surfacer +BC/CC
 5. Pretreatment + ED5100 + Primer Surfacer +BC/CC (Replicate)
 6. Pretreatment + Developmental Primer
 7. Pretreatment + Developmental Primer + BC/CC
- Steps 1-5 represent a general coating system for automotive painting.
 - Step 6 and 7 represent a special coating system with the 2-step developmental primer surfacer applied electrolytically rather than by spraying.

Table 4.3 shows additional details of the individual paint layers applied by PPG.

Table 4.3: Details of the individual paint layers.

Coatings	Type	Thickness or weight
Pretreatment	Standard Tri-cation type, immersion, no Cr rinse	250-300mg/ft ²
ED5100	Standard Daimler-Chrysler E-coat primer	23-26µm
Primer Surfacer	Standard GM type, GPX (spray)	45-48µm
Developmental Primer	2-coat electrolytic primer system by PPG	49-52µm on coated substrates, 55µm on CR
BC (Base Coat)	Black, waterborne basecoat	Total 71-75µm
CC (Clear Coat)	PPG Diamond coat (Used at Daimler-Chrysler)	

4.2 Commercially Produced Imperfection Samples

4.2.1 Imperfection Samples

Replicate specimens of 14 different imperfections on coated sheet steel were provided by participating companies. The characteristics of these imperfections are summarized in Table 4.4. The “imperfection type” is identified using descriptions provided by the producing company. All of the imperfections provided for this portion of the study were on hot-dip galvanized or galvanized coatings. It should be noted that substrates H-4 to H-9 are thicker than the others and are not likely to be used for exposed automotive applications. These imperfections were classified into 4 types according to size, morphology, origin and appearance. The types include dross, irregular surface, a combination of dross + irregular surface, and mechanically produced imperfections.

- Dross type imperfections: light sink roll dross mark, light sink roll line, light drag dross line, splash mark, light dross, mid/heavy dross, rub/gouge dross, white spot
- Irregular surface: rough surface, chatter mark
- Dross + irregular surface imperfections: rub/gouge dross
- Mechanically induced imperfections: dent, cold rolled spot, small white spot

Figure 4.4 shows 3D profiles of selected imperfections; the detailed characteristics of each imperfection will be presented in the results section.

Table 4.4: Characteristics of commercially-produced imperfections provided by participating companies.

	Imperfection type	Coating type	Characteristics
H-1	Light sink roll dross mark	HDG*	Randomly distributed dross particles
H-2	Light sink roll line	HDG	Aligned dross particles
H-3	Light drag dross line	HDG	Aligned dross particles
H-4	Splash mark	HDG	Congregated dross particles
H-5	Light dross	HDG	Tiny dross particles
H-6	Mid/heavy dross	HDG	Larger dross particles
H-7	Rough surface	HDG	Irregular surface
H-8	Rub/Gouge dross	HDG	Irregular surface with dross particles
H-9	Chatter mark	HDG	Wavy surface
G-1	White spot	GA**	Dross-like Particles
G-2	Dent	GA	Dent
G-3	Cold rolled spot	GA	Narrow and shallow scratch
G-4	Small white spot	GA	Zinc pick-up mark

* HDG: Hot dip galvanized coating

** GA : Galvannealed coating

Figure 4.4: Surface topography of selected commercially produced imperfection-types.

4.2.2 Painting

For painting, 34 test panels sheared to 4" x 12" were selected, with varying imperfection types and sizes. Table 4.5 listed summary of painting conditions. The test panel were painted by ACT Laboratories Inc., Hillsdale, MI. Details of each painting layer are listed in Table 4.6. After ED-coating, all the samples were returned and surface analyses were conducted on the imperfections. One ED-coating sample of each imperfection was retained for later examination. In addition, two types of primer surfacer (liquid and powder) were applied to compare painting response. The powder and liquid primer surfacers represent high and low-build systems, respectively. These two systems are used by Diamler-Chrysler.

Table 4.5: Summary of painting conditions for imperfection samples.

	Samples	ED	Liquid primer	Powder primer	BC/CC
H-1	Light sink roll mark	O	◇	O	O
H-2	Light sink roll line	O	◇	O	O
H-3	Light drag dross line	O	◇	O	O
H-4	Splash mark	O	N/A	O	O
H-5	Light dross	O	N/A	O	O
H-6	Mid/heavy dross	O	N/A	O	O
H-7	Rough surface	O	N/A	O	O
H-8	Rub/Gouge dross	O	O	O	O
H-9	Chatter mark	O	O	O	O
G-1	White spot	O	O	O	O
G-2	Dent	O	O	O	O
G-3	Cold rolled spot	O	O	O	O
G-4	Small white spot	O	O	O	O

Table 4.6: Details of the individual paint layers used for commercial imperfection samples. (Provided by ACT laboratories)

Coatings	Type	Range of thickness or weight
Pretreatment	ParkerAmchem Bonderite 958 /Parcolene 60 Rinse pretreatment	-
Phosphate	DI water immersion zinc phosphate, C700 C59	250 –525 mg/ft², Crystal size < 10mm
Electrodeposition Primer	ED5100	1.10 –1.25 mils (27.9 – 31.7 mm)
Primer surfacer	PCV70100M (Powder type)	2.5-3.5 mils (63.5 - 88.9 mm)
	DPX1809K (Liquid type)	1.6-1.8 mils (40.6 - 45.7 mm)
Base coat	HWB9517 Black	0.8-1.0 mils (20.3 - 25.4 mm)
Clear coat	DCT5002H	1.8-2.2 mils (40.6 - 55.8 mm)

-Total coating thickness with liquid primer surfacer: 109 - 158 mm

-Total coating thickness with powder primer surfacer: 129 – 201 mm

4.3 Characterization Techniques

4.3.1 Visual Inspection

Four observers were requested to rank imperfections visually for severity. Evaluation was performed in an area with good illumination, using natural and/or artificial light. This simulates the visual assessment of vehicles in inspection areas or outside in daylight. Personnel were instructed to move the panels to any preferred position or place themselves into any preferred position to most carefully evaluate the imperfections.

4.3.2 Topographic Measurement

The WYKO NT2000, non-contacting optical profiler, used in this project is shown schematically in Figure 2.12. Profilometry was utilized to capture and monitor the geometrical characteristics of imperfections before and after painting. A Michelson interferometer is used for the 5x magnification objective applicable to this work.

The phase-shifting interferometry (PSI) mode allows measurement of smooth surfaces, while the vertical-scanning interferometry (VSI) mode is used for rough surfaces and steps. VSI and PSI modes each have their own measurement ranges. Table 2.3 shows the dynamic range for each mode, and the vertical resolution capabilities.

In this project, surface profiles were obtained using VSI mode for unpainted, pretreated (phosphated) and ED-coated samples, while PSI mode was used for primer surfacer and top coated samples. Surface profiles were analyzed using the Vision 32TM software package provided with the instrument. The data are represented as contour plots that show surface heights as a function of x and y-position, two-dimensional plots that presented traces in the x and y-directions, and three-dimensional plot. Other parametric descriptions of the surfaces are also used, where appropriate.

4.3.3 Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray Spectroscopy (EDS)

Surface morphologies of the as-received and phosphated zinc coated sheet steels were evaluated in the JEOL scanning electron microscope (SEM), and the surfaces were photographed at 100X and 1,000X. Also, EDS analyses of imperfections were performed to help establish their origin. Excellent resolution and depth of field are key advantages of the SEM. Electron/sample interactions also produce x-rays that have energies specific to the chemical elements present in the sample. These characteristic x-rays can be collected and the chemical elements in the sample identified. The intensity of x-rays detected for each element can also be used to quantify the amount of each element present.

4.3.4 Photography

Photographs were taken of the commercially produced samples containing surface imperfections in the as-received condition. Painted samples following ED-coating and after top coating were also photographed to record the changes in appearance, and visibility after painting. As-received samples containing dross lines, sink roll marks, white spots, and small white spots were captured at 2X with a 35 mm camera equipped with a macro lens for improved depth of field. Photographs of samples containing white spots and small white spots were also taken at 21X to gain a better perspective of the imperfection morphology. Dross lines and sink roll marks were captured at 1X and 3X and compared to a surface free of imperfections.

4.4 Forming Studies

Sheet samples were stretch-formed on the Colorado School of Mines (CSM) limiting dome height (LDH) test equipment with a modified die designed to impose strains similar to those observed in stretch forming of automotive body panels. The following sections will systematically explain the die design which was used to replicate forming of automotive body panels, the mechanical testing system, and the strain calibration required for calculating strain induced by biaxial stretching, and for developing a strain matrix.

4.4.1 Die Design

A pre-existing Marciniak die was fitted with a modified curved punch designed to approximately reproduce curvature and forming conditions in automotive door panels and hoods. The curvature was chosen to impose 0.5% bending strain. Die curvature was designed from interpretation of the “segment of a circle” theory, and is based on circular geometry. A geometric section showing a “segment of a circle” with a chord length, l , radius, R , and segment length, c is represented in Figure 4.5.

Figure 4.5: Segment of a circle with chord length, l , segment length, c , and radius, I (4.25)

A critical radius for the circular arc, characteristic of a segment length of three inches was calculated by subtracting two functions, $X(r)$ and $Y(r)$ given below (where r is equal to I):

$$X(r): h = r - 0.5 \sqrt{4r^2 - c^2} \quad [4.1]$$

$$Y(r): h = r (1 - \cos((\theta/2))) \quad [4.2]$$

$$\text{where } \theta = (57.296 * l) / r \quad [4.3]$$

Assuming 0.5% bending strain at the surface, a die radius of 11.7 inches was calculated, corresponding to a height, h , of 0.1722 inches. Figure 4.6 shows an engineering drawing of the modified, curved punch, which was machined from A-2 tool steel, hardened to 61 Rockwell C, and lap finished.

Figure 4.6: Engineering drawing of the modified curved punch, designed from 0.5% bending strain, originating from a circular arc with a radius of 11.7 inches. All dimensions are in inches.

4.4.2 Stretch-Forming Set-up

A stretch-forming press originally designed and built by Burford (59), and adapted to a standard MTS test frame, was utilized for this study. The mechanical testing frame shown in Figure 4.7 was outfitted for stretch-forming experiments in which a modified curved punch seated in the Marciniak punch, and was connected to the 50 kip load cell, shown in the diagram. The upper and lower dies in this system conformed to a standard LDH system. Test samples were 8" X 8" panels sheared from the as-received sheet.

Figure 4.7: MTS set-up designed and built by Burford (59)

4.4.3 Strain Calibration

Circle grids were applied to selected samples and were used to calibrate actuator displacement to imposed strain. The calibration curve was required, as circle grids were not desired on the test samples used for critical surface analysis.

Circle grids were applied to 8" X 8" panels with a commercial Lectroetch electrochemical system. A pattern of four 2.54 mm diameter circles in a 6.35 mm square area (0.1 inch circles, 0.25 inch square) was applied to the surface of each zinc coated steel sheet by electrochemical marking. Optimal etch contrast was achieved with a Lectroetch V45A power source and Lectroetch number 112-A electrolyte with the power source operated at 45 Ohmites for 5 seconds. Strain was measured along one of the principal strain axes, by monitoring the total displacement across eight 6.35 mm etched squares. Displacements were measured with a flexible scale with minimum divisions of 0.254 mm (0.01 inches). Uncertainty in the strain measurement increased slightly with strain, and averaged $\pm 0.29\%$.

Figure 4.8 shows the calibration curve that relates actuator displacement to measured strain in one of the two equi-biaxial strains in the plane of the sheet. One layer of polyethylene sheet and LPS1 lubricant was used to lubricate the punch/sample interface. This was found to be the optimal lubrication scheme, and was utilized to minimize friction, thereby increasing the stretching capability.

Figure 4.8: Strain calibration curve relating actuator displacement to biaxial principal strain.

4.5 Creation of Controlled Imperfections

Imperfections were created in the laboratory with dent-type and raised morphologies. The imperfections here were created by slightly different methods than described in Section 4.1.2 above, and the following sections describe the experimental methods used to produce the imperfections and the corresponding test matrices.

4.5.1 Indenter Design / Hard Particle Selection

Dent-type imperfections were simulated in the laboratory by indenting normal to the sheet surface with modified hardness indenters. Conical indenters with face angles of 2° and 4°

were machined, lap finished, and mounted on a standard macrohardness machine using a superficial “F” scale, and a 15 kg applied load. Figure 4.9 shows a schematic drawing of the modified indenter. These indentations were desired to be very shallow, and angle “ θ ” is not shown to scale in the schematic drawing.

Figure 4.9: A schematic drawing of an indenter with machined face angles (θ) of 2° and 4° .

Raised imperfections, or “outdings” were created during stretch-forming experiments, in which grade 440 stainless steel balls 0.381 (0.015 in.), 0.508 (0.020 in.), and 0.635 (0.025 in.) millimeters in diameter were placed at the sheet/punch interface. The stainless steel balls were obtained from the Salem Ball Company. The stretch-forming test matrices for the dent-type and raised imperfections are included below in Tables 4.7 and 4.8 respectively. Completed tests are indicated with an “X”.

4.5.2 Test Matrix for Dents / Outdings

Two different types of tests were conducted in the laboratory. Imperfected samples were stretch-formed in a series of fixed strain experiments in addition to strain accumulation experiments where a single sample was incrementally strained to failure. The strain matrices that follow in Tables 4.7 and 4.8 detail the laboratory experiments conducted at fixed strain values.

Table 4.7: Strain matrix for dent-type imperfections.

Die Angle	Material	Imposed Strain (pct.)				
		0	2	4.5	5.75	7
2°	GA	X	X	X		X
	HDG	X	X	X		X
	EG	X	X	X	X	X
4°	GA	X	X	X		X
	HDG	X	X	X		X
	EG	X	X	X	X	X

Table 4.8: Strain matrix for laboratory-controlled raised imperfections.

Ball Diameter (mm)	Material	Imposed Strain (pct.)			
		2	4.5	5.75	7
0.381	GA	X	X	X	X
	HDG	X	X	X	X
	EG	X	X	X	X
0.508	GA	X	X	X	X
	HDG	X	X	X	X
	EG	X	X	X	X
0.635	GA	X	X	X	X
	HDG	X	X	X	X
	EG	X	X	X	X

4.6 Industrial Imperfections

Participating steel producers also supplied samples of galvanealed and hot dipped galvanized coated sheet steels, selected to include a variety of commercially produced surface imperfections. Imperfection origin ranged from mechanical, to chemical, and processing causes. Sheets containing features referred to as dross lines, sink roll marks, white spots, and small white spots were analyzed for forming response and subsequent appearance after painting. A series of

three independent experiments was conducted for each imperfection as outlined in the following section.

4.6.1 Test Matrix for Industrial Imperfections

1. Form a panel to 4% (principal) strain, and characterize the forming effects on an imperfection by collecting surface data before and after forming.
2. Form a panel to 4% strain, and characterize the combined effects of forming and painting on a imperfection by collecting surface data before forming, after forming, and after painting.
4. Incrementally strain a sample and monitor the topographical change in an imperfection at forming strains until fracture.

Finally, one sample containing each imperfection type was saved to maintain an example of the as-received condition.

4.7 Painting

The as-received commercial samples were visually inspected to locate and characterize the commercial imperfections. These sample blanks were sheared such that the imperfection was located at the center of an 8" X 8" sample. One sample representative of each imperfection provided by participating steel producers was formed and painted. In addition, strained panels consisting of laboratory-induced dent-type imperfections, and microcracks in galvanized coatings were also painted. A summary of the samples selected for painting is shown in Table 4.9, and the painting specifications, as provided by ACT Laboratories are included in Table 4.10. These samples were painted at the same time as the commercial imperfection samples discussed earlier in Section 4.2.2. Only the high-build (powder primer surfacer) was used for the strained imperfections, however.

Table 4.9: Painting test matrix.

# of Samples	Material	Imperfection	Forming Strain (%)
1	HDG	Dross Line	4
1	HDG	Sink Roll Mark	4
1	GA	White Spot	4
1	GA	Small White Spot	4
3	GA	Dent *	2, 4.5, 7
1	GA	Microcracks	4

* Laboratory dents created with a 4^o indenter.

Table 4.10: Painting specifications provided by ACT Laboratories.

Pretreatment	PPC©Zinc Immersion Phosphate	250-525 mg/ft ²
Organic Coating	Coating Specification	Thickness (μm)
ED-Coating	PPG© Cathodic Epoxy	28-32
Powder Primer	PPG© Polyester (hybrid)	64-89
Base Coat	HWB – acrylic-melamine	20-25
Clear Coat	DCT500Z – melamine	46-56

4.8 Characterization Techniques

4.8.1 Profilometry

Surface morphologies were evaluated with the Wyko NT2000 Surface Profiler. Profilometry was utilized to capture and monitor imperfections before forming, after forming, and following subsequent painting. A methodology was developed to characterize strain response of dent-type imperfections in terms of trends in average roughness (R_a) and imperfection area. Roughness data, isolating the local imperfection area from an area unaffected by the imperfection (background), was utilized to examine imperfection evolution as a function of strain. The software eliminated surface tilt from each profile, and curvature from dent-type imperfections (only). Additional filtering was not performed in analyzing roughness, because the measurement lengths were quite small relative to the typical 1-inch traces used in industry.

Laboratory produced outdings were systematically analyzed from two-dimensional (2-D) plots. Repeatable measurements were produced using a two parameter analysis method where the height, h , and width, w , given by the 2-D trace as depicted in Figure 4.10, were measured with reference to the zero line (mean line) determined by the software.

Figure 4.10: Schematic of the measurement variables used for characterizing outdings with surface profilometry.

Endpoints of the measurement were systematically extracted from the intersection of the 2-D trace, and the zero line (mean line) given by the software. This data analysis procedure provided a reproducible relationship between imperfection dimension and forming strain.

The characteristics of industrial imperfections after forming and painting were evaluated in a similar fashion, by recording imperfection width change as a function of strain, in addition to a visual inspection of the contour plots and corresponding 2-D traces through the stages of forming and painting.

Uncertainty in the profilometry data was estimated by measuring the same area of a repeatable surface. Twenty-three repetitions were performed on a cold rolled steel sheet at 5X, where R_a measurements resulted in an uncertainty of ± 38 nm for each measurement.

4.8.2 Microscopy

Surface morphologies of the as-received zinc coated sheet steels were evaluated in the JEOL scanning electron microscope (SEM), and the surfaces were photographed at 100X and 1,000X. Surfaces of dent-type imperfections were also captured in the SEM at equivalent magnifications following strain accumulation experiments in which a single sample was incrementally strained to failure.

Cross-sectional light metallography was employed to better understand deformation behavior at the substrate/coating interface. Zinc coatings that experienced failure during strain accumulation experiments were sectioned across the through-thickness of the sheet. These samples were cold mounted with Leco low viscous epoxy resin and ground with 240, 320, 400, and 600 grit paper. Electrogalvanized and galvanized coatings were polished with 6 and 1 μm diamond compound and 0.05 μm colloidal silica. The softer hot dipped galvanized coating was only polished with 6 μm diamond compound and 0.05 μm colloidal silica. The tint etchant developed by Kilpatrick (60) was used for all three coatings, revealing the phases present in each coating as viewed with light microscopy at 750X. Each substrate/coating interface was also captured in the SEM at 1,000X.

4.8.3 Powdering Tests

Powdering is defined as a deformation behavior where decohesion occurs by intracoating failure, producing particles with dimensions less than the coating thickness (61). Powdering tests were performed following procedures outlined by Deits (61), where adherence tests were employed using scotch tape, qualitatively measuring zinc particle removal against a standard color rating scale. Likewise, following laboratory production of raised imperfections, double-sided carbon tape was applied to a SEM mount, and lightly pressed to the locally raised area of a formed galvanized sheet, and placed into the SEM for analysis. Zinc particles were examined at 500X and 2,000X for two formed galvanized panels experiencing 5.75% and 7% strain, and the particle composition was confirmed as zinc by Electron Dispersive Spectrometry (EDS).

4.8.4 X-Ray Diffraction

Diffraction patterns were generated with a Siemens θ - 2θ diffractometer over the range of 20 to 80 degrees 2-theta using a reflection technique. Since the reflection technique was employed, only planes that were parallel to the coating surface contributed to the resulting diffracted peak intensities. Filtered copper radiation was used with monochromatic x-rays of 1.5406 angstrom wavelength. The intensity for a random zinc powder was used in this analysis to normalize the diffracted intensities of the galvanized, hot dipped galvanized, and electrogalvanized coatings. The “percentage of planes technique” developed by Shaffer et al, (62, 63) and discussed by Wenzloff (64) was used to analyze the diffraction pattern of all three zinc coatings before and after strain accumulation experiments.

4.8.5 Photography

Photographs were taken of the commercially produced samples containing surface imperfections in the as-received condition, and of the formed and painted samples following ED-coating. As-received samples containing dross lines, sink roll marks, white spots, and small white spots were captured at 2X with a standard 35 mm camera equipped with a micro lens for improved depth of field. Photographs of samples containing white spots and small white spots were also taken at 21X to gain a better perspective of the imperfection morphology. Additional photographs were taken of imperfections that were visible to the naked eye following forming and subsequent ED-coating with a Nikon 990 CoolPix digital camera. Dross lines and sink roll marks were captured at 1X and 3X and compared to a surface free of imperfections. Photography documented imperfection appearance in the middle of the painting process, before top-coating.

5.0 RESULTS – PAINTING STUDIES

5.1 Laboratory Induced Imperfections

5.1.1 Substrate Materials

5.1.1.1 Metallography

Imperfections were created in the laboratory on cold rolled (uncoated) sheet, and sheet steels having three different metallic coatings. Figures 5.1-5.6 show SEM micrographs that depict microstructures of the three experimental coated sheet steels at 100 X and 1000 X. The galvanized (GA) coating is intermetallic in nature and is comprised of a needle-like microstructure. The hot dip galvanized (HDG) coating is primarily zinc with an intermetallic phase at the substrate/coating interface and has a pancake-like grain morphology. The electrogalvanized coating (EG) coating is nearly pure zinc and has a flat, faceted microstructure.

Figure 5.1: SEM micrograph of the as-received galvanized coating.

Figure 5.2: SEM micrograph of the as-received galvanized coating.

Figure 5.3: SEM micrograph of the as-received hot dip galvanized coating.

Figure 5.4: SEM micrograph of the as-received hot dip galvanized coating.

Figure 5.5: SEM micrograph of the as-received electrogalvanized coating.

Figure 5.6: SEM micrograph of the as-received electrogalvanized coating.

5.1.1.2 Profilometry

Figures 5.7-15 show contour plot, 2-D trace and 3-D plot of three experimental steel sheets. Profilometric data are presented in a series of contour plots, corresponding 2-D traces and 3-D plot.

A contour plot shows the surface relief of the material, plotting the x and y dimensions across the surface, and the associated peak or valley height. The peak and valley heights appear on a z-axis which is shown here as a height difference from zero, and is measured in micrometers (μm) and represented by color variations according to the legend. Each height scale has a maximum and a minimum that corresponds to the highest and the lowest points on the surface, respectively. Therefore, height scales may vary from plot to plot depending on variations in the material surface. The corresponding 2-D trace represents the surface height across a single trace (in the x or y direction) in the contour plot. The 2-D trace shows both macroscopic and microscopic features in the form of the curvature and roughness features of the coating surface. Also, important surface parameters, such as R_a , R_q and R_t are listed next to the 2-D trace. The definitions of these parameters are provided in Appendix III. The 3-D plot shows a 3-dimensional rendering of the surface, showing the x and y dimensions across the surface, and the associated peak or valley height at each point.

Figure 5.7: Contour plot of GA surface.

Figure 5.8: 2-D trace of unpainted GA surface.

Figure 5.9: 3-D plot of unpainted GA surface.

Figure 5.10: Contour plot of unpainted HDG surface.

Figure 5.11: 2-D trace of unpainted HDG surface.
X Profile

Y Profile

Figure 5.12: 3-D plot of unpainted HDG surface.

Figure 5.13: Contour plot of unpainted EG surface.

Figure 5.14: 2-D trace of unpainted EG surface.

Figure 5.15: 3-D plot of unpainted EG surface.

Table 5.1 lists roughness, R_a , from the manufacturer and unfiltered profilometry results measured here. The roughness of the GA sample shows the highest value compared to the other coating surfaces (see Table 5.1), while HDG has the lowest values. Roughness results from optical profilometry show similar results; particularly that the HDG coating has the smoothest coating surface.

Table 5.1: Surface roughness of three coated sheet steels used to examine laboratory-induced imperfections. (CR samples were received only after painting.)

Roughness ($R_a = \mu\text{m}$)	GA	HDG	EG
From as-received data	0.94	0.76	0.86
Profilometry results (unfiltered)	X*: 1.40 Y**: 1.55	X profile: 0.89 Y profile: 0.78	X profile: 1.15 Y profile: 1.07

*X: R_a from X-direction 2-D trace

** Y: R_a from Y-direction 2-D trace

5.1.2 Profilometry of Imperfections Before Painting

5.1.2.1 Dent Type Imperfections

Creation of dent, scratch, and raised imperfections in the laboratory was described earlier. Examples of profilometric data for two different dent-type imperfections are shown in Figures 5.16-19 for a GA material. 3-D plot, contour plot and corresponding 2-D traces are included for each dent. Indenters with face angles of 110° , 70° and 40° were used to produce the dents. In this section, only data produced from the 110° and 40° indenter with GA are presented. As shown in Figure 5.16 and 5.17, a 40° indenter produced a dent with a lateral dimension of $536\ \mu\text{m}$ and a depth of $13.6\ \mu\text{m}$ for the GA material. In Figure 5.18 and 5.19, a 110° indenter produced a dent with a lateral dimension of $1,239\ \mu\text{m}$ and a depth of $104\ \mu\text{m}$. The dent characteristics on the HDG and EG materials are similar to the GA material.

Figure 5.16: 3-D plot of dent (GA, indenter 40° , unpainted).

Figure 5.17: Contour plot and 2-D trace of dent (GA, indenter 40° , unpainted).

Figure 5.18: 3-D plot of dent (GA, indenter 110° , unpainted).

Figure 5.19: Contour plot and 2-D trace of dent (GA, indenter 110° , unpainted).

5.1.2.2 Scratch Type Imperfections

A scratch tester was used for creating controlled scratch-type imperfections. The slopes, widths and depths of surface features were thought to be potentially important, and different surface imperfection characteristics were obtained by changing the indenter tip geometry and the load on indenter (0 – 25N). Indentation lengths up to 100mm were made so that the visibility of these long indentations would be relatively straightforward to assess after painting. Three different indenters were prepared, having conical tips with varying cone angles, as shown schematically in Figure 4.3. The indenter geometries were designed to assess the painting response of deeper features. With these indenters, a variety of different imperfections were created using the scratch tester. Figures 5.20 and 5-21 show a contour plot, 2-D trace and 3-D plot for scratch-type imperfection with the $\theta = 40^\circ$ tip using an indenter load of 25N. Figures 5.22-25 show the 3-D topography of the galvanized surface after scratch testing with the $\theta = 40^\circ$ tip using indenter loads of 20N, 15N, 10N, and 5N. A clear progression in the features of the imperfections is observed; at an indenter load of 25N (Figure 5.21) a relatively deep scratch is obtained, at an intermediate load of 15N (Figure 5.23) the scratch is not quite as deep as some of the original features in the surface, and at a light load of 5N (Figure 5.25), the surface is barely changed by the indenter. The light scratch is clearly visible as a slight smearing in the image, and this region is more reflective to the naked eye. Two-dimensional surface profiles through the entire series of imperfections from Figures 5.21-5.25 are presented in Figure 5.26. Similar profiles are presented in Figure 5.27 and 5.28 for the two other indenters ($\theta = 70^\circ$ and 110° , respectively). Comparison of these three figures illustrates the substantial changes in imperfection depth and width which were obtained by this methodology. The 3-D topography of the indentation obtained from the 70° indenter at a 20N load is shown in Figure 5.29; comparison of this figure with the corresponding indentation from the 40° indenter (Figure 5.22) illustrates the substantial difference in width of imperfections which can be obtained. Finally, it should be noted that the different indenters result in markedly different slopes of the sidewalls of the imperfections, and the software provided with the profilometer is able to easily quantify these slopes.

Figure 5.20: Contour plot and 2-D trace of galvanized surface containing a scratch-type imperfection created with a scratch-tester using the 40° indenter and 25 N load.

Figure 5.21: 3-D plot of galvanized surface containing a scratch-type imperfection created with a scratch-tester using the 40° indenter and 25 N load.

Figure 5.22: 3-D plot of galvanized surface containing a scratch-type imperfection created with a scratch-tester using the 40° indenter and 20 N load.

Figure 5.23: 3-D plot of galvanized surface containing a scratch-type imperfection created with a scratch-tester using the 40° indenter and 15 N load.

Figure 5.24: 3-D plot of galvanized surface containing a scratch-type imperfection created with a scratch-tester using the 40° indenter and 10 N load.

Figure 5.25: 3-D plot of galvanized surface containing a scratch-type imperfection created with a scratch-tester using the 40° indenter and 5 N load.

Figure 5.26: Series of 2-D traces for scratch-type imperfections created with different loads on the 40° indenter.

Figure 5.27: Series of 2-D traces for scratch-type imperfections created with different loads on the 70° indenter.

Figure 5.28: Series of 2-D traces for scratch-type imperfection created with different loads on the 110° indenter.

Figure 5.29: 3-D plot of galvanized surface containing a scratch-type imperfection created with a scratch-tester using the 70° indenter and 20 N load.

5.1.2.3 Raised Imperfections

The raised imperfections were created in the laboratory using a hardness tester, specially designed indenters, and by penetrating from the opposite side of the sheet. Table 5.2 lists a summary of profilometry results from 24 raised imperfections on 4 different coating surfaces. These raised imperfections were made by changing indenter and load. From the profilometry data, the smallest raised imperfection shows a lateral dimension of 896 μm and a height of 56 μm on the CR surface. This raised imperfection is bigger than the deepest scratch imperfection. In this section, results from one R1 raised imperfection on GA coating are provided. Figures 5.30 and 5.31 present a 3-D plot, contour plot and 2-D traces for the R1 raised imperfection on GA. The raised imperfection appears white or light in the contour plot. This particular “outding” has a lateral dimension of 944 μm and a height of 71 μm . This is the smallest one among the 6 raised imperfections on the GA coating surface. Similar results are provided in Appendix V, for raised imperfections on HDG, EG and CR surfaces, and for the other raised imperfections on GA.

Table 5.2: Summary of profilometry results for raised imperfections.

Imperfection ID		R1	R2	R3	R4	R5	R6
GA	Height (mm)	0.071	0.240	0.950	0.084	0.230	0.930
	Width (mm)	0.950	1.350	2.980	0.945	1.260	2.901
HDG	Height (mm)	0.065	0.265	0.942	0.063	0.256	0.924
	Width (mm)	0.980	1.356	2.970	0.998	1.345	2.790
EG	Height (mm)	0.074	0.251	0.965	0.075	0.243	0.945
	Width (mm)	0.968	1.421	2.845	0.967	1.421	2.786
CR	Height (mm)	0.056	0.214	0.890	0.065	0.241	0.857
	Width (mm)	0.892	1.251	2.680	0.845	1.241	2.560

Figure 5.30: 3-D plot of raised imperfection (GA, unpainted).

Figure 5.31: Contour plot and 2-D trace of raised imperfection (GA, unpainted).

5.1.3 Visual Inspection Results

Figure 5.32 provides a summary of the appearance of the numerous laboratory-induced imperfections after painting with the paint system described in Table 4.3 applied by PPG. Each of the schematic features represents a different imperfection, and the “boxes” indicate which imperfections are completely invisible after painting. These visibility tests show that only a few of the least severe imperfections on each sample panel disappear after painting with this system (based on human visual inspection). The geometric characteristics of the 66 imperfections simulated on each sample were discussed in the previous section. Table 5.3 shows the summary results of visual inspection for the painted laboratory samples with different imperfections geometries. The “invisible imperfection counts” presented in the table represent the number of imperfections that were not visible upon close inspection after BC/CC (base coat plus clear coat) top coating; i.e. that essentially disappeared after painting. In the case of dents, raised imperfections and deep scratches, none of the imperfections created in this work disappeared after top coating. However, some of the shallow scratches did disappear after top coating.

While the primary focus of this project is on GA, it is useful to examine possible differences between the substrates, including galvanized steel sheets (GA), hot-dip galvanized steel sheets (HDG), electrogalvanized steel sheets (EG) and cold rolled steel sheets (CR). There were not significant differences between the substrates, except for GA. In the GA material, the number of imperfections that were invisible after painting was one less than for the other substrates. To put the results in a quantitative context, it should be noted that the deepest scratch that was invisible after painting began with a depth of about 3 μm prior to paint application, although it will be shown in later sections that deeper features of commercial imperfections were sometimes found to disappear after painting. It should also be noted that many of the laboratory induced imperfections were sometimes quite substantial in height or depth, and thus it was not surprising that they remained visible after painting. Since the largest imperfection amplitude that could be rendered invisible was unknown prior to this work, it was important in the methodology employed here to include imperfections having a wide range of characteristics.

Figure 5.32: Visibility test result after painting of laboratory-induced imperfections.

Table 5.3: Summary of visual inspection results for different imperfection geometries.
(After final BC/CC top coating with a conventional painting system)

Substrate	Invisible imperfection counts*			
	Dent	Raised Imperfections	Scratches	
			shallow	deep
GA	0/30	0/6	3/15	0/15
HDG	0/30	0/6	4/15	0/15
EG	0/30	0/6	4/15	0/15
CR	0/30	0/6	4/15	0/15

*Invisible imperfection counts: imperfections invisible after painting / total imperfections examined

5.1.4 Evolution of Imperfections During Painting

To evaluate the painting response of unstrained sheet surfaces, multiple replicate samples having controlled imperfections were prepared and painted. Seven replicates of each of four substrate materials were prepared. The materials examined included hot-dip galvanized steel sheets (HDG), electrogalvanized steel sheets (EG), galvanized steel sheets (GA) and cold rolled steel sheets (CR). While the primary focus of this project is on GA material, it is also useful to examine differences between the substrates. Each replicate sample panel has 30 dents (pits), 30 scratches, and 6 “outding” (raised) imperfections.

5.1.4.1 Evolution of Dent

Samples were painted to investigate dent appearance after painting. The visual inspection results show that all 30 dent-type imperfections were visible after top coating. The “degree” of visibility is indicated in the captions to some of the figures below. Profilometry results from two dent-type imperfections are given below; one is the shallowest one, the other is the deepest one. Figures 5.33-5.38 contain example 3-D plots, contour plots and corresponding 2-D traces of these dent-type imperfections after individual painting steps including ED-coating, primer surfacer application and following top coating (base coat and clear coat). These particular dent imperfections were formed with a 40° indenter with load of 15 N prior to painting. The 3-D plots and 2-D traces show that the dent is visible after ED-coating and primer surfacer application with residual depths of 5.2 and 1.3 μm (Figures 5.36 and 5.37), respectively, and are nearly invisible following top-coating with a depth of 0.6 μm (Figure 5.38). The original (unpainted) dent depth was approximately 13.6 μm .

For comparison, Figures 5.39-5.44 contain 3-D plots, contour plots and corresponding 2-D traces showing the behavior of a large dent formed with the 110° indenter with a load of 55 N prior to coating. The 3-D plot and 2-D traces after top coating show that this dent is still evident as a small valley in the surface profile having a depth of approximately 2.6 μm (Figure 5.44). The original depth of this particular dent was 104 μm .

Substrate

After ED

Figure 5.33: 3-D plot of dent evolution during painting.
(GA, nearly visible after painting, 40° indenter, 15N)

After primer surfacer

After BC/CC

Figure 5.34: 3-D plot of dent evolution during painting (GA, nearly visible after painting, 40° indenter, 15N)

Figure 5.35: Contour plot and 2-D trace of dent.
(GA, unpainted, 40° indenter, 15N)

Figure 5.36: Contour plot and 2-D trace of dent.
(GA, after ED, 40° indenter, 15N)

Figure 5.37: Contour plot and 2-D trace of dent after primer surfacer.
(GA, nearly visible, 40° indenter, 15N)

Figure 5.38: Contour plot and 2-D trace of dent after BC/CC.
(GA, nearly visible, 40° indenter, 15N)

Figure 5.39: 3-D plot of dent evolution during painting.
(GA, visible after painting, 110° indenter, 55N)

After primer surfacer

After BC/CC

Figure 5.40: 3-D plot of dent evolution during painting.
(GA, visible after painting, 110° indenter, 55N)

Figure 5.41: Contour plot and 2-D trace of dent.
(GA, unpainted, 110° indenter, 55N)

Figure 5.42: Contour plot and 2-D trace of dent. (GA, after ED, 110° indenter, 55N)

Figure 5.43: Contour plot and 2-D trace of dent (GA, after primer, 110° indenter, 55N)

Figure 5.44: Contour plot and 2-D trace of dent after BC/CC.
(GA, visible, 110° indenter, 55N)

5.1.4.2 Evolution of Scratches

Sixty scratch-type imperfections were painted to investigate their appearance after painting. The visual inspection results show that only a few shallow scratches were invisible after top coating. Profilometry results from three representative scratch-type imperfections are provided here; including an invisible scratch, one invisible by the naked eye but visible by topography, and finally a visible scratch.

Figure 5.45-5.50 shows the surface evolution of a shallow scratch having a gentle sidewall angle, after each coating step (GA). The depth of this scratch is $2.6\mu\text{m}$ in the unpainted condition. This scratch was completely invisible after final painting, as indicated in the caption. Figure 5.51 shows a summary plot of the 2-D profiles. It should be noted that the y-axis scale reflects the scale of the cross-section features, but the relative (vertical) position of the individual profiles is arbitrary in Figure 5.51. After ED coating, most of the scratch has disappeared, although the scratch still remains visible as shown by the small depression in the ED profile of Figure 5.44 and 5.47, and confirmed by visual inspection. After top coating (BC/CC condition), the shallow scratch has completely disappeared, based on visual inspection and Figure 5.45, as well as the surface contour plot and 2-D trace of Figure 5.49.

Figures 5.52-5.58 provide an example of surface evolution of a scratch having a somewhat greater depth. The scratch depth is $8.1\mu\text{m}$ in the unpainted condition. After primer surfacer application, the scratch is nearly gone. The imperfection was invisible after BC/CC coating, as indicated by visual inspection, but was slightly visible by the (instrumental) surface profile. It is perhaps noteworthy that the vertical dimension of the imperfection is on the order of only a few hundred nanometers after painting, and yet is still visible by 3-D profilometry.

In contrast to the shallower scratches above, Figure 5.59-5.65 shows the surface evolution during painting for a much deeper scratch having a sharp sidewall angle. The depth of the scratch is $37.7\mu\text{m}$ in the unpainted condition. In this instance, it is clear that much of the imperfection depth still remains after ED coating, and Figure 5.60 and 5.64 show that it also remains detectable after top coating (note the linear feature in the contour map). This feature was also evident upon visual inspection. This section illustrates the general differences between imperfections, whereby larger imperfections were detectable after painting.

Figure 5.45: 3-D plot of scratch evolution during painting.
(GA, invisible after painting)

After primer surfacer

After BC/CC

Figure 5.46: 3-D plot of scratch evolution during painting.
(GA, invisible after painting)

Figure 5.47: Contour plot and 2-D trace of scratch (GA, unpainted).

Figure 5.48: Contour plot and 2-D trace of scratch (GA, after ED).

Figure 5.49: Contour plot and 2-D trace of scratch (GA, after primer surfacer).

Figure 5.50: Contour plot and 2-D trace of scratch after BC/CC.
(GA, invisible after top coating)

Figure 5.51: 2-D topography of scratch.
(GA, invisible after painting)

Substrate

After ED

Figure 5.52: 3-D plot of scratch evolution during painting.
(GA, invisible after painting, visible in topography)

After primer surfacer

After BC/CC

Figure 5.53: 3-D plot of scratch evolution during painting.
(GA, invisible after painting, visible in topography)

Figure 5.54: Contour plot and 2-D trace of substrate (GA, unpainted).

Figure 5.55: Contour plot and 2-D trace of scratch (GA, after ED).

Figure 5.56 Contour plot and 2-D trace of scratch (GA, after primer).

Figure 5.57: Contour plot and 2-D trace of scratch after BC/CC.
(GA, invisible after painting ,in visible in topography)

Figure 5.58: Series of 2-D topography of scratches.
GA, invisible after painting, visible in topography)

After ED

Figure 5.59: 3-D plot of scratch evolution during painting.
(GA, visible after painting)

After primer surfacer

After BC/CC

Figure 5.60: 3-D plot of scratch evolution during painting.
(GA, visible after painting)

Figure 5.61: Contour plot and 2-D trace of scratch (GA, unpainted).

Figure 5.62: Contour plot and 2-D trace of scratch (GA, after ED).

Figure 5.63: Contour plot and 2-D trace of scratch (GA, after primer).

Figure 5.64: Contour plot and 2-D trace of scratch after BC/CC.
(GA, visible after painting)

Figure 5.65: Series of 2-D topography of scratch.
(GA, visible after painting)

5.1.4.3 Evolution of Raised Imperfections

The raised imperfections were created using a hardness tester, specially designed indenters, and by penetrating from the opposite side of the sheet. Profilometry results from the smallest raised imperfection (R1, in Table 5.2) are provided in this section. Figures 5.66-5.72 contain 3-D plots, contour plots and corresponding 2-D traces of raised imperfections after ED-coating, primer surfacer application and following top coating (base coat and clear coat). The height in the unpainted condition was about 71 μ m. The 3-D plots and 2-D traces show that raised imperfections are visible after ED-coating, primer surfacer, and top coating with a heights of about 71, 31 and 17 μ m (Figures 5.66 - 5.68), respectively

Substrate

After ED

Figure 5.66: 3-D plot of raised imperfection evolution during painting.
(GA, visible after painting)

Figure 5.67: 3-D plot of raised imperfection evolution during painting.
(GA, visible after painting)

After primer surfacer

After BC/CC

Figure 5.68: Contour plot and 2-D trace of raised imperfection (GA, unpainted substrate).

Figure 5.69: Contour plot and 2-D trace of raised imperfection (GA, after ED).

Figure 5.70: Contour plot and 2-D trace of raised imperfection (GA, after primer surfacer).

Figure 5.71: Contour plot and 2-D trace of raised imperfection (GA, after BC/CC).

Figure 5.72: 2-D plot of surface evolution after each painting step.
(GA, raised imperfection)

5.1.5 Summary Results from Laboratory Induced Imperfections

The geometrical characteristics of the various imperfections are summarized along with the conditions used for their creation in Table 5.4. The summary includes the width and depth or height in the unpainted condition, and after various steps in the painting process. Some of the results are plotted in the figures that follow to provide additional insight into the controlling relationships between the important parameters. From the topographical analysis of scratch imperfections on GA material in Figure 5.73, it is clear that the invisible imperfections have very small values of initial width and depth, less than about $3\mu\text{m}$ in depth and less than about $0.25\mu\text{m}$ in width. Figure 5.74 shows that final residual depth is closely related to the initial depth of the imperfection, and the residual depth decreases with decreasing initial depth. Figure 5.75 shows the change in the residual scratch depth with each coating step for four different initial depths. The electrodeposition (ED) primer attenuates about 80% of the initial depth, and after the primer surfacer, painting attenuates about 90% of the initial depth. Final top coating covers almost 100% of the depth after base/clear coating (BC/CC). The width change of the imperfection during painting is minimal, however, compared to the change in depth (Figure 5.76).

Table 5.4: Summary of imperfection creation conditions and results.

Imperfection Type	Instrument	Indenter	Width (mm)	Depth (μm)
Dent-type	Hardness tester & Scratch tester	40° , 70° , 110°	1.0 – 1.3	13.6 - 104
Scratch-type	Scratch tester	40° , 70° , 110°	0.2 – 0.9	0.13 – 45.00
Raised type	Hardness tester	40° , 70°	0.065 – 2.9	74 - 95

Figure 5.73: Visibility results correlated with topography results. (Galvannealed, scratch imperfections)

Figure 5.74: Residual vs. initial depth of scratch imperfections on galvanized substrate. Results are plotted separately for the different indenter geometries.

*Attenuation ratio = $\frac{(D_i - D_f)}{D_i} \times 100$; D_i = Imperfection Depth on Substrate;
 D_f = Imperfection Depth after each coating strp

Figure 5.75: Residual depth change for scratches with different initial depths.

Figure 5.76: Width change of scratch imperfections having different initial depths on a galvanized substrate after each painting step.

5.2 Results of Panels with Laboratory Induced Scratches Painted Simultaneous with Industrial Imperfections

5.2.1 Effect of Different Primer Surfacers

Sixteen test panels having laboratory-induced scratches originating from 40° indenter tips and different loads were included in the paint application (over industrial imperfections discussed in a later section) at ACT to serve as a reference for comparison of the two different painting studies in this work. These samples underwent the same painting procedures as the commercially produced imperfections (Table 4.6). Two types of primer surfacer (liquid and powder) are used to compare painting response of the laboratory-induced scratches. The powder (thickness: 63.5 - 88.9µm) and liquid (thickness: 40.6 - 45.7µm) primer surfacers represent high and low-build systems, respectively. A summary of results from visual inspection and profilometry analyses are listed in Table 5.5.

After top coating, all of the scratches were visible or barely visible using either visual inspection or profilometry. Scratches painted with a powder primer surfacer become less visible compared to scratches with a liquid primer surfacer, based on the visual inspection results. Figure 5.77 shows residual depth changes for laboratory-induced scratches and “a small white spot” (identical to Figure 5.132, pit type imperfection) after each coating step. From Figure 5.139, residual depths of laboratory-induced scratches (GA 5: GA, load 5N) have similar changes to those of the commercially produced “small white spot”. Therefore, laboratory results are confirmed to predict surface evolution behavior during painting of commercially produced samples containing similar imperfection characteristics.

Table 5.5: Visual inspection and profilometry analysis results for laboratory-induced scratches after ED coating and top coating.

	Stylus* Load (N) (5 Shallow ↔ 15 deep)	Visual inspection			Profilometry		
		After ED coating	After top coating		After ED coating	After top coating	
			Liquid primer	Powder primer		Liquid Primer	Powder primer
GA	5	O	O	△	O	O	O
	10	O	O	O	O	O	O
	15	O	O	O	O	O	O
HDG	5	O	O	△	O	O	O
	10	O	O	O	O	O	O
	15	O	O	O	O	O	O
EG	5	O	O	△	O	O	O
	10	O	O	O	O	O	O
	15	O	O	O	O	O	O
CR	5	O	O	△	O	O	O
	10	O	O	O	O	O	O
	15	O	O	O	O	O	O

O: visible, △: barely visible, X: invisible

GA: Galvannealed, HDG: Hot-dip galvanized, EG: Electro-galvanized, CR: Cold rolled

* 40° Indenter

Figure 5.77: Residual depth of imperfections after electrodeposition (ED) and base coat/clear coat (CC) top coating.

5.2.2 Developmental Coating System

A proprietary 2-coat electrolytic primer system was also examined. The goal of this system is to replace conventional primer systems (ED + Primer Surfacer). The primer thickness is 50 μ m compared to 75 μ m for the conventional coating system in our study. The conditions are listed in Table 4.3.

Table 5.6 shows the summary results of visual inspections after each painting step. For the conventional painting system, some of the imperfections disappeared after the primer surfacer coating. The number of invisible imperfections increased after top coating. For the developmental painting system, there were no invisible imperfections after the primer application, and the number of invisible imperfections after top coating was less than that of the conventional coating system. Again the number of invisible imperfections for the GA substrate was less than that of other substrates.

Table 5.6: Invisible imperfection counts after different painting steps.

Substrate	Invisible imperfection counts (scratches)					
	General coating system				Proprietary system	
	Phosphate	ED	Primer	BC/CC	Primer	BC/CC
GA	0/30	0/30	1/30	3/30	0/30	0/30
HDG (or GI)	0/30	0/30	1/30	4/30	0/30	2/30
EG	0/30	0/30	2/30	4/30	0/30	1/30
CR	0/30	0/30	4/30	4/30	0/30	2/30

Figures 5.78 and 5.79 are examples of surface evolution of laboratory induced scratches with the developmental coating system, obtained for the same type of imperfection geometry as examined in Figure 5.45. After primer coating, most of the scratch is attenuated, but the imperfection was still visible after BC/CC coating, as indicated by the surface profile as well as visual inspection. Figure 5.80 presents imperfection depth changes after painting. The developmental system shows a larger residual depth value and less coverage compared to the normal coating system after each coating step. It is believed that this difference results from either a difference in the coating thickness, or the application process. (Since the developmental primer is entirely electrodeposited, it might be expected to follow the substrate topography more closely, reducing the imperfection coverage.)

Figure 5.78: 2-D topography of shallow scratch having gentle sidewall angle.
(GA sample, developmental coating system)

Figure 5.79: Surface contour plot of shallow scratch having gentle sidewall angle, after painting.
(GA sample, after BC/CC painting, developmental coating system)

Figure 5.80: Residual depth change of scratch imperfections on different substrates with different primer systems after BC/CC application.

5.3 Painting Response of Industrial Imperfections

5.3.1 Characterization of Imperfections

The description of 13 different imperfections obtained from steel producers are listed in Table 4.4 (see Section 4.2.1).

5.3.2 Topography of Imperfections Before Painting

The dross type imperfections are raised imperfections, $2.6\text{ }\mu\text{m}$ to $4.6\text{ }\mu\text{m}$ in height and 0.1mm to 0.9mm in width. Arrangement of the dross particles varied with imperfection type. Light sink roll dross marks, light dross and mid/heavy dross contain dross particles that are distributed randomly throughout the surface. Dross particles are arranged in a line for both light sink roll line and light drag dross line imperfections. Figures 5.81 – 5.83 show some typical contour, 2D and 3D plots of these defects.

Figure 5.81: 3D plot of “Light sink roll dross mark” on galvanized coating

Figure 5.82: Contour and 2D trace of "Light sink roll cross mark" on galvanized coating

Figure 5.83: 3D plot of “Light sink roll line” on galvanized coating.

5.3.3 SEM/EDS analysis of 3 selected Imperfections

SEM/EDS analysis was conducted to provide further characterization of the industrial imperfections. Three imperfections were selected for examination on GA coatings; white spot (oudting type), cold rolled spot and small white spot (pit-type). The imperfections on HDG coating have been well characterized by others (10, 14).

5.3.4 Visual Inspection Results

The results from both the visual inspection and profilometry are listed in Table 5.7, summarizing the response to painting of the 13 industrially produced imperfections with two paint systems. This table provides painting conditions for each imperfection and presents some of the key outcomes of the project. All the imperfections were painted using both a powder (high build) and a liquid (low build) primer surfacer. Imperfection samples H4-H7 were painted only with powder primer surfacer because of limited imperfection sample numbers. After ED-coating, most imperfections remained visible, but the “dents” and “cold rolled spots” disappeared entirely. After top coating with a liquid primer surfacer (low build system), imperfections are less visible than after ED coating, but all of the imperfections except “dents” and “cold rolled spots” remained slightly visible. Imperfections painted using the high build system incorporating a powder primer surfacer were substantially less visible after top coating compared to imperfections with the liquid primer surfacer.

Table 5.7: Visual inspection and profilometry results after ED coating and top coating.

	Imperfection type	Visual inspection			Profilometry		
		After ED coating	After top coating		After ED coating	After top coating	
			Liquid primer	Powder primer		Liquid primer	Powder primer
H*1	Light sink roll dross mark	O	◇	X	O	O	X
H-2	Light sink roll line	O	◇	X	O	O	X
H-3	Light drag dross line	O	◇	X	O	O	X
H-4	Splash mark	O	N/A	◇	O	N/A	◇
H-5	Light dross	O	N/A	X	O	N/A	X
H-6	Mid/heavy dross	O	N/A	◇	O	N/A	◇
H-7	Rough surface	O	N/A	X	O	N/A	X
H-8	Rub/Gouge dross	O	O	X	O	O	X
H-9	Chatter mark	O	O	O	O	O	O
G**-1	White spot	O	◇	X	O	O	◇
G-2	Dent	X	X	X	X	X	X
G-3	Cold rolled spot	X	X	X	X	X	X
G-4	Small white spot	O	◇	X	O	O	X

* H: Hot-dip galvanized ** G: Galvannealed

O: visible, ◇: barely visible, X: invisible,

6.0 RESULTS – FORMING STUDIES

6.1 Profilometry and Analysis of Dents

Two types of indenters were fabricated to produce dent-type imperfections in the laboratory. Indenters were machined with face angles of 2° and 4° . Profilometry of the two types of dents showed that a 2° indenter produced a dent with a lateral dimension of $620\text{ }\mu\text{m}$ and a depth of $7\text{ }\mu\text{m}$ for the HDG material. A 4° indenter produced a similar dent with a lateral dimension of $600\text{ }\mu\text{m}$ and a depth of $8.5\text{ }\mu\text{m}$. That is a 20% difference in imperfection depth between the two indenters, holding the material and hardness testing conditions constant. Both types of dents experience similar response to stretch-forming, a summary plot of the forming effect on an imperfection created from a 2° indenter is presented in Figure 6.1. Dent depth decreases as a function of strain, and is apparent when comparing the surface profiles through the center of a dent for the 0% and 7% strain conditions. Similar deformation response was found for a dent originating from a 4° indenter. Consequently, only data produced from the 4° indenter are presented for discussion.

Profilometric data are presented in a series of contour plots and corresponding 2-D traces. A contour plot shows the surface relief of the material, showing the x and y dimensions across the surface, and the associated peak or valley height. The peak and valley heights appear on a z-axis that is shown here as a height difference from zero, and is measured in micrometers (μm). Each height scale has a maximum and a minimum that corresponds to the highest and the lowest points on the surface, respectively. Therefore, height scales may vary from plot to plot depending on variations in the material surface. The corresponding 2-D traces represent the surface height across single traces in the x and y directions in the center of the dent pictured in the contour plot. The 2-D trace shows both macroscopic and microscopic features in the form of the dent curvature produced from a conical indenter, and the roughness features of the coating surface. Dent width in the x and y directions is approximated by the Vision 32TM software program for each data pair, and is designated by arrows for each Figure.

A compilation of profilometric data for dent-type imperfections was generated for the GA, HDG, and the EG materials. For each set of data, there are data pairs that represent a dent-type imperfection before and after forming for both the 2% and 7% strain conditions, originating from the series of fixed strain experiments. Each strain condition is representative of a different imperfection created from the same loading conditions with a 4° indenter. These data were depicted in a series of contour plots, and 2D & 3D traces. Typical results are shown in Figures 6.2 and 6.3. These generated figures represent three independent observations of unstrained samples for the GA, HDG, and EG materials, and provide an assessment of the variability in creating dent-type imperfections in the laboratory.

The evolution of dent-type imperfections on a GA surface, as shown in these generated data plots, indicates that, as strain increases, the initial (“macroscopic”) profile associated with the indentation flattens, while crack density and depth increase.

For the HDG and EG surfaces, it has been observed that, as strain increases, the imperfection on the HDG surface tends to flatten while the surrounding material simultaneously roughens.

Similar data plots show dent evolution as a function of strain for the EG material. Dent geometry on the EG surface behaves in a similar fashion to HDG, as strain increases the dent profile tends to flatten.

Comparing the deformation response of the three materials, the GA and HDG samples had less resistance to localized deformation than the EG sample during the indenting process. 2-D profiles show that dent depth increases from 6 μm for the EG coating, to 8 μm and 10 μm for the HDG and GA materials, respectively. This may reflect differences in resistance to plastic flow of the three materials, consistent with the small differences in bulk mechanical properties of the three materials detailed in Table 4.2. Specifically, when considering depth of a dent, the yield strengths of the three steel substrates are of particular interest. The yield strength of the EG material is the highest (194 MPa), the HDG material follows (164 MPa), with the GA material having the lowest yield strength (155 MPa).

During the series of fixed strain experiments, the average roughness (R_a) from the local area containing the dent-type imperfection and the area unaffected by the imperfection (background area) was collected as a function of strain by systematically capturing each local area within the same x and y dimensions from the contour plots. An example of the imperfection and the background areas that were captured with the Vision 32TM software are denoted in Figure 6.1 with two boxes. Table 6.2 compares the local roughness of the imperfection area to the background for each of the three experimental materials. Local roughness changes associated with the imperfection area are a function of dent depth and the local variations in the surface profile across the center of the dent. It is important to note that the “local imperfection roughness” is not a true roughness value, but instead is largely a measure of the change in dent dimensions, which is a strong function of dent depth. It is evident that with strain the roughness of the background area increases, while the roughness of the area that includes the imperfection decreases, becoming shallower in depth and less prominent after straining, consistent with a decrease in imperfection area.

In addition to the series of fixed strain experiments, the effects of strain accumulation on surface imperfections were evaluated for the three experimental sheet surfaces. A second set of data shows contour plots and corresponding 2-D traces of dent-type imperfections before and after strain accumulation to failure for the GA, HDG, and the EG materials. One sample of each coating containing a single indentation from the 4° indenter was strained in increments of 0.02 strain. This incremental strain procedure was continued until straining caused fracture of the sample. Typical plots are shown in Figures 6.4 – 6.7.

The average roughness (R_a) was collected in a similar fashion to the fixed strain experiments outlined previously for both the background and imperfection areas and values are plotted in Figure 6. 8 and 6.9, respectively. Figure 6.8 shows that in all three materials the surfaces away from the imperfection roughen in response to forming. This approximately linear relationship between R_a and strain is more prominent in the galvanized material where the slope of the curve is higher by an order of magnitude due to cracks that form in the coating to accommodate substrate strain. The HDG and EG materials have curves in Figure 6.8 that behave in a like manner, and are almost parallel indicating similar deformation responses. Figure 6.9 shows the imperfection response to strain accumulation. Initially at low strains the imperfection

becomes shallower in depth and the average roughness correspondingly decreases until it reaches a minimum. For the GA material, at higher strains the increase in roughness is consistent with the effect of strain on the local background roughness. It is important to note that for each material this roughness behavior as function of strain is consistent with the roughness data presented in Table 6.1.

Repeatability for creating laboratory dents and measuring the associated response to forming with optical profilometry can be seen by comparing the contour plots and 2-D traces for the zero strain condition in a material. For example, a comparison of the 2-D traces for the HDG material shows little to no variability in the depth of the dent (8 μm), with slight variations in the lateral dimensions (474 μm –506 μm).

Figure 6.1: Evolution of a dent (2° indentation) on a HDG coating through the stages of forming, manipulated from output of 2-D traces during optical profilometry.

Figure 6.2: GA coating contour plot (4° indentation, 0 strain).

Figure 6.3: GA coating 2-D trace (4° indentation, 0 strain).

Table 6.1: Local roughness of the imperfection (geometrical measure of the surface)* and background areas as a function of strain. Data were collected during the series of fixed strain experiments.

Material	Imposed Strain (pct.)	Imperfection Roughness (R_a) (μm)	Background Roughness (R_a) (μm)
GA	0.0	1.99	1.45
	2.0	1.46	1.58
	4.5	1.61	1.90
	7.0	1.61	2.09
HDG	0.0	1.85	1.09
	2.0	1.20	1.10
	4.5	1.23	1.15
	7.0	1.23	1.27
EG	0.0	1.52	1.01
	2.0	1.20	1.16
	4.5	1.24	1.26
	7.0	1.24	1.35

* Imperfection originated from a 4° indenter. Imperfection roughness is a function of dent depth plus the local variations in the surface profile across the center of the dent (dent depth >>> surface profile variations).

Figure 6.4: GA coating contour plot (4° indentation, 0 strain).

Figure 6.5: GA coating 2-D trace (4° indentation, 0 strain).

Figure 6.6: GA coating contour plot (4° indentation, 0.1125e).

Figure 6.7: GA coating 2-D trace (4° indentation, 0.1125e).

Figure 6.8: Average roughness as a function of strain for the background region. Data points were collected during deformation accumulation experiments for galvanneal (GA), hot dipped galvanized (HDG), and electro-galvanized (EG) coated sheet steels.

Figure 6.9: Average roughness as a function of strain showing the imperfection deformation response (4° indenter) for galvanneal (GA), hot dipped galvanized (HDG), and electrogalvanized coated sheet steels.

6.2 Profilometry and Analysis of Painted Laboratory-Induced Dents

Galvanneal samples from the series of fixed strain experiments were painted to investigate dent and microcrack appearance after painting. Contour plots and corresponding 2-D traces of dent-type imperfections after ED-coating, and following top-coating, were generated. These imperfections were formed with a 4° indenter and stretched to 7% principal strain prior to painting. Typical results are shown in Figures 6.10 – 6.17.. Imperfections are designated on the contour plots with an arrow. 2-D traces show that dents are visible after ED-coating with depths of 2 and 1 μm (Figures 6.11 and 6.15) for the 2% and 7% strain conditions, respectively, and are invisible following top-coating (Figures 6.13 and 6.17).

For comparison, Figures 6.18-6.25 contain contour plots and corresponding 2-D traces showing a formed galvanneal panel without an imperfection through the stages of forming and painting. Imposed strain for the forming condition was fixed at 0.04e, approximately equal to the strain found in automotive body panels. Cracks are evident in Figures 6.18, 6.20, and 6.22 as black spots and as sharp local variations in profile height in Figures 6.19, 6.21, and 6.23. 2-D traces after ED-coating (Figure 6.23) show that cracks in the brittle GA coating remain evident as small valleys in the surface profile with a depth of approximately 1 μm , becoming invisible after top-coating (Figure 6.25). Curvature in the surface profiles is associated with the curvature of the modified Marciniak punch used for straining the specimens.

Figure 6.10: GA coating contour plot (4° indentation, 0.02e, ED-coating).

Figure 6.11: GA coating 2-D trace (4° indentation, 0.02e, ED-coating).

Figure 6.12: GA coating contour plot (4° indentation, 0.02e, top-coating).

Figure 6.13: GA coating 2-D trace (4° indentation, 0.02e, top-coating).

Figure 6.14: GA coating contour plot (4° indentation, 0.07e, ED-coating).

Figure
coating

ED-

6.15: GA
2-D trace (4°
indentation, 0.07e,
coating).

Figure 6.16: GA coating contour plot (4° indentation, 0.07e, top-coating).

Figure 6.17: GA coating 2-D trace (4° indentation, 0.07e, top-coating).

Figure 6.18: GA coating contour plot (0 strain).

Figure 6.19: GA coating 2-D trace (0 strain).

Figure 6.20: GA coating contour plot (0.04e).

Figure 6.21: GA coating 2-D trace (0.04e).

Figure 6.22: GA coating contour plot (0.04e, ED-coating).

Figure 6.23: GA coating 2-D trace (0.04e, ED-coating).

Figure 6.24: GA coating contour plot (0.04e, top-coating).

Figure 6.25: GA coating 2-D trace (0.04e, top-coating).

6.3 Profilometry and Analysis of Raised Imperfections

Raised imperfections (or “outdings”) were created during stretch-forming experiments, in which 440 stainless steel balls with three different diameters were placed at the sheet/punch interface. Only the imperfections resulting from the 0.381 mm ball are shown, as similar results were found for the 0.508 and 0.635 mm balls. Profilometry results confirmed that imperfection height increased with ball size, and the magnitude of the height decreased with strain after initial punch contact.

Imperfection evolution from 2% to 7% strain is presented in a series of contour plots and 2-D traces for the GA material in Figures 6.26-6.29. Raised imperfections appear white or light in color in the contour plots (Figures 6.26 and 6.28), and as locally raised areas in the corresponding 2-D traces. It is evident from the 2-D traces that outding height decreases from 22 μm at 2% strain to about 16.5 μm at 7% strain. Profilometric data are presented for the HDG and EG materials in a similar fashion in Figures 6.30-6.33, and Figures 6.34-6.37, respectively. Outding height for a HDG material decreases as a function of strain from about 25 μm to 17 μm at 7% strain, and 23 μm to 14.5 μm for the EG material. A plot of the x-dimension as a function of strain is included in Figure 6.38, summarizing the effects of strain on outding width for each material. As strain increases, the imperfection width, as defined in Figure 4.10, increases while the associated height decreases. Forming of laboratory-produced “outdings” essentially stretches the imperfection equi-biaxially, becoming less apparent with strain.

Figure 6.26: GA coating contour plot (0.381 mm ball, 0.02e).

Figure 6.27: GA coating 2-D trace (0.381 mm ball, 0.02e).

Figure 6.28: GA coating contour plot (0.381 mm ball, 0.07e).

Figure 6.29: GA coating 2-D trace (0.381 mm ball, 0.07e).

Figure 6.30: HDG coating contour plot (0.381 mm ball, 0.02e).

Figure 6.31: HDG coating 2-D trace (0.381 mm ball, 0.02e).

Figure 6.32: HDG coating contour plot (0.381 mm ball, 0.07e).

Figure 6.33: HDG coating 2-D trace (0.381 mm ball, 0.07e).

Figure 6.34: EG coating contour plot (0.381 mm ball, 0.02e).

Figure 6.35: EG coating 2-D trace (0.381 mm ball, 0.02e).

Figure 6.36: EG coating contour plot (0.381 mm ball, 0.07e).

Figure 6.37: EG coating contour plot (0.381 mm ball, 0.07e).

Figure 6.38: Imperfection x-dimension as a function of strain for galvannealed (GA), hot dipped galvanized (HDG), and electro-galvanized (EG) coated sheet steels (0.381 mm ball).

The raised imperfections were not subsequently painted, due to their considerable height.

6.4 Microscopy: Coating Microstructures and Cross-Sections

Coating microstructure is directly related to roughness of a material and controls deformation response of the coating. Figures 6.39-6.44 show SEM micrographs that depict microstructures of the three experimental steel sheets at 100X and 1000X (these micrographs were presented earlier, and are reproduced here for comparison to the strained surfaces.). The GA coating is intermetallic in nature and is comprised of a needle-like microstructure, analogous to a higher as-received roughness ($R_a = 1.46 \mu\text{m}$). On the other hand, the HDG coating is primarily zinc with an intermetallic phase at the substrate/coating interface. The HDG coating has the lowest as-received roughness ($R_a = 0.99 \mu\text{m}$), corresponding to a pancake-like grain morphology. The EG coating is nearly pure zinc and has an intermediate as-received roughness ($R_a = 1.15 \mu\text{m}$), corresponding to a flat, faceted microstructure.

These as-received microstructures may be compared to SEM micrographs in Figures 6.45-6.50, which show the microstructures of dent-type imperfections following strain accumulation experiments in which a single sample containing a dent-type imperfection (4°) was incrementally strained to failure. The series of SEM micrographs show the surface of a dent-type imperfection at a low (100X) and high (1000X) magnification. The imperfection fills the field of view at the lower magnification, while only the center of the imperfection is captured in more detail at the higher magnification. It is evident through the SEM that indenting a surface with a conical indenter locally flattens the microstructural features for each of the coatings. The needle-like structure appears to be spreading apart, cracking to accommodate substrate strain.

Cross-sectional metallography was employed to better understand deformation behavior at the substrate/coating interface. In Figures 6.51-6.53, SEM micrographs show the substrate/coating interface for each coating at the strain to failure condition. A tint etchant was used to reveal phases present in the intermetallic microstructure for the GA and HDG coatings, visible with light microscopy at 750X. The GA coating microstructure was confirmed to have a gamma phase (cubic) at the substrate/coating interface, with the delta phase (hexagonal close-packed) which nucleated at the interface and grew towards the free surface of the coating. Very little of the zeta phase (monoclinic) was noticeable with light optical microscopy, however it is possible that a small amount resides at the surface. Crack formation in the GA cross-section is apparent both in the coating and the substrate. Cracks appear to nucleate both at the substrate/coating interface and in the through-thickness of the sheet. Evidence of possible flaking (site "A") and powdering (labeled with arrows) are captured, as free coating particles are seen above the coating, and decohesion of a coating particle is apparent. The HDG coating microstructure (Figure 6.52) was confirmed to be comprised of mostly zinc, with a δ phase at the coating/substrate interface which is approximately 6% iron.

Figure 6.39: SEM micrograph of the as-received galvanized coating.

Figure 6.40: SEM micrograph of the as-received galvanized coating.

Figure 6.41: SEM micrograph of the as-received hot dip galvanized coating.

Figure 6.42: SEM micrograph of the as-received hot dip galvanized coating.

Figure 6.43: SEM micrograph of the as-received electro-galvanized coating.

Figure 6.44: SEM micrograph of the as-received electro-galvanized coating.

Figure 6.45: SEM micrograph of a 4° indentation on a GA surface after failure (0.1125e).

Figure 6.46: SEM micrograph of a 4° indentation on a GA surface after failure (0.1125e).

Figure 6.47: SEM micrograph of a 4° indentation on a HDG surface after failure (0.1425e).

Figure 6.48: SEM micrograph of a 4° indentation on a HDG surface after failure (0.1425e).

Figure 6.49: SEM micrograph of a 4° indentation on an EG surface after failure (0.125e).

Figure 6.50: SEM micrograph of a 4° indentation on a EG surface after failure (0.125e).

Figure 6.51: SEM micrograph of a GA steel sheet after failure (0.1125e).

Figure 6.52: SEM micrograph of a HDG steel sheet after failure at (0.1425e).

Figure 6.53: SEM micrograph of an EG steel sheet after failure (0.125e).

6.5 Powdering Test Results: SEM and EDS

Deformation behavior of coated materials can also be investigated by performing powdering tests. During forming, coating damage is characterized by one of four failure modes: flaking, galling, powdering, and cracking (61).. Powdering was confirmed for stretch-formed samples containing laboratory “outdings” at 6.75% and 7% strain for the GA material, and is also suggested by the cross-section of a GA material formed to failure in Figure 6.51. In Figure 6.54, an SEM micrograph shows extracted zinc particles ranging in size, with the largest particle around 7 μm at 7% strain. This would also suggest that flaking occurred since the particle size is approximately equal to the coating thickness. Particle composition was confirmed as zinc by EDS, and is shown in Figure 6.55. Similar results were observed at 6.75% strain and are not included.

Figure 6.54: SEM micrograph at 1000X of zinc particles originating from an “outding” of a panel stretch-formed to 7% strain.

Figure 6.55: Corresponding EDS pattern for zinc particles originating from an “outding” of a panel stretch-formed to 7% strain.

6.6 X-Ray Diffraction: As-Received vs. Formed Coatings

Crystallographic texture and grain size have been found to influence surface roughness of coatings (66). A linear dependence between grain size and strain has been documented for both uniaxial and biaxial tension (61). In this study, X-ray diffraction (XRD) was performed on samples in the as-received condition and after forming to 7% strain. Figures 6.56-6.58 show the XRD profiles as a function of 2θ before and after forming for the GA, HDG, and EG coatings. The “percentage of planes” technique (62-64) was used to normalize the data and the results, presented as bar charts, are included in Figures 6.59-6.60 for the HDG and EG coatings. This method was not straightforwardly applicable for the GA coating due to the extent of intermetallic phases present in the coating microstructure. Figures 6.59-6.60 plot frequency for the primary diffracted planes within the coatings. The normalization plots show that the HDG material does have a preferred orientation in the [0002] direction before and after forming. A similar preferred orientation was not observed in the EG material before and after forming. Both the (0002) and the (1013) planes were significantly present in the as-received and formed conditions for the EG material. X-ray diffraction showed that there is not a strong texture change after biaxial stretching for the HDG and EG materials. Therefore, possible texture changes in the coating do not significantly contribute to roughening following biaxial stretching.

Figure 6.56: X-ray diffraction profiles for a GA sample (a) before forming, and (b) after forming to 7% strain.

Figure 6.57: X-ray diffraction profiles for a HDG sample (a) before forming, and (b) after forming to 7% strain.

Figure 6.58: X-ray diffraction profiles for an EG sample (a) before forming, and (b) after forming to 7% strain.

Figure 6.59: Normalization for the HDG coating showing the frequency of diffracted planes before forming and after forming to 7% strain.

Figure 6.60: Normalization for the EG coating showing the frequency of diffracted planes before forming and after forming to 7% strain.

6.7 Forming of Industrial Imperfection Samples

Commercially-produced samples containing dross lines, sink roll marks, white spots, and small white spots were stretch-formed to 4% strain, and painted. A series of contour plots and corresponding 2-D traces of dross lines were generated in the as-received condition, after stretch-forming, ED-coating, and following top-coating. Each 2-D profile represents the trace across the center of a dross particle. Imperfection height increased from about 3 μm to 6.5 μm with strain. Dross lines were still visible with profilometry and visual inspection after ED-coating with a height of 6 μm , becoming less visible following top-coating.

In addition, changes in imperfection morphology were documented before and after failure during strain accumulation experiments. Imperfection height varies from about 5 μm , to 11 μm with strain. It is clear that the dross imperfection becomes more apparent with strain, protruding through the coating.

Sink roll marks were observed in the same manner, documenting imperfection appearance through the stages of forming, painting, and strain accumulation. The 2-D trace of the sink roll mark showed that the initial height of the imperfection was about 4 μm , similar to the initial height of the dross line. After forming to 4% strain, the sink roll mark became more apparent with a height of about 7 μm . Following ED-coating, the same imperfection became less apparent with a height of about 3 μm , becoming invisible after top-coating.

Two types of imperfections were analyzed on GA coated samples. Profilometry confirmed that white spots were imperfections of raised geometry while the small white spots were of dent-type morphologies. Profilometric data were generated in a similar fashion, documenting imperfection appearance through the stages of forming, painting, and strain accumulation for the white spot and for the small white spot. Each 2-D profile represented the trace across the center of the imperfection. Imperfection morphologies were evident as local light and dark variations in the contour plots for the as-received conditions. Both types of white spots appeared to be surface artifacts (width $\gg$ height/depth) with evident x and y dimensions, but shallow in depth or short in height. The vertical dimensions of these imperfections were similar to the height variations on the coated surface, on the order of 1 to 2 μm .

Similar changes upon forming in imperfection geometry and topographical features as documented with the dross lines and sink roll marks were undetectable in the GA coating. Microcracks in the GA coating became more prominent after stretch-forming, masking the imperfection of interest. Both the white spots and small white spots were undetectable with profilometry after ED-coating, and top-coating. The radius of curvature in each profile after top-coating was calculated and found to be approximately equal to 11.8 inches, the same radius of curvature for the die (Figure 4.6). All imperfections are designated with an arrow in the figures where appropriate.

6.8 Photography

Photographs were taken of the commercially produced samples containing surface imperfections, and of the formed and painted samples following ED-coating. After top-coating, all imperfections were invisible to the naked eye and were not captured with photography.

6.9 Summary of Results

Raised imperfections that probably formed as a result of non-uniform coating deposition during steel production (e.g. dross lines and sink roll marks) became more apparent after stretch-forming and were visible with profilometry after ED-coating. Likewise, forming-induced imperfections that were a consequence of mechanical response during automotive manufacturing (e.g. cracks) became evident during stretch-forming, and were slightly visible following ED-coating. However, superficial imperfections that formed due to external contact during steel production and automotive manufacturing, and chemical reactions during steel production (dents, stains and spots) were less apparent after stretch-forming, becoming invisible after ED-coating.

Sink roll marks, white spots, and small white spots included in this study were invisible with profilometry following top-coating with a powder primer surfacer. Visibility of dross particles was more questionable, and could be slightly visible due to variation in the curvature of the surface profile following top-coating observed with optical profilometry. The discussion section will offer an explanation to imperfection response to forming, addressing why some imperfections increase in severity after stretch-forming, and others become less visible with stretch-forming. Final thoughts on factors affecting imperfection appearance after forming and painting will also be addressed.

7.0 DISCUSSION

7.1 Evolution of Imperfections During Painting

7.1.1 Effect of Imperfection Geometry

The results of surface analysis and visual inspection of samples (before and after painting) were examined and interpreted to determine quantitative relationships between imperfection geometry and painting response. Figure 7.1 shows a schematic of key geometrical features, such as width, depth and sidewall slopes, that were examined to understand quantitatively the topographical changes of imperfections.

In this study, three types of imperfections were created in the laboratory; dent-type, scratch-type and raised-type. Dent-type imperfections had different depths, shapes and slopes. Scratch-type imperfections had different sidewall slopes, depths and widths. Raised imperfections had different heights. And 13 different commercially produced imperfections selected by steel producers were also tested for comparison.

It was shown earlier (Figure 5.73) that only a few of the least severe imperfections disappear after painting of the laboratory induced imperfections. None of the dents, raised imperfections or deep scratches disappeared after top

Figure 7.1: 2-D profile showing key geometrical features used to quantify imperfection characteristics.

coating. However, some of the shallow scratches did disappear after top coating (Table 5.3). From the topography analysis, it was found that the invisible imperfections had very small values of initial width and depth, less than about $3\text{ }\mu\text{m}$ in depth and less than about $0.25\text{ }\mu\text{m}$ in width (Figure 5.73). Final depth was closely correlated with the initial depth of the imperfection, and the final or residual depth decreased with decreasing initial depth. However, the width change of each imperfection during painting was minimal. The imperfection depth was filled or attenuated about 50% to 85% by the coating after ED coating, about 90% after primer surfacer application and about 98% after top coating.

From the above analysis of both laboratory and industrial imperfections, it is clear that initial depth/height of imperfection is the characteristic most influenced by painting. However, it is believed that the width also influences visibility, and further analysis is ongoing to clarify this issue.

7.1.2 Effect of Coating System and Materials

In this study, three different coating systems (conventional liquid and powder primer systems, and a developmental primer system) were used to evaluate coating system effects on visibility. In Section 5.2.1, conventional liquid (low build, thickness $45\mu\text{m}$) and powder (high build, thickness $88\mu\text{m}$) systems were evaluated. Analysis showed that the powder primer system covers more imperfections than the liquid primer system (Table 5.5). In Section 5.2.2, a developmental 2 layer electrolytic primer system was examined. This developmental primer system has fewer coating layers (3 layers), and a reduced coating thickness (total of about $120\mu\text{m}$) compared to the conventional paint system (4 coating layers and a total of about $140\mu\text{m}$). The developmental system shows a smaller imperfection coverage or attenuation ratio compared to the conventional system. Analysis results from three different organic coating systems suggested that the high build powder system (thicker coating layer) and increased numbers of coating layers improve the coverage of imperfections.

With respect to substrate material considerations, four different materials were used in this project; GA, HDG, EG and CR, described in Tables 4.1 and 4. 2, and Figures 5.1 through 5.6. Figure 7.2 shows residual depth changes for the different materials during painting. After ED coating, the galvanized material shows slightly greater residual depth compared to the other materials. Visual inspection showed that imperfections on painted galvanized materials are slightly more visible compared to the other materials (as shown in Figure 5.32, and Tables 5.3 and 5.5). In this study, a more detailed examination of the mechanisms associated with material effects on visibility was not conducted. However, it is presumed that differences may come from different phosphate microstructures or ED-coating responses on galvanized materials, based on results of other studies (23).

Figure 7.2: Residual depth changes with different substrate materials. (liquid primer system)

7.2 Evolution of Imperfections

Figure 7.3 is a schematic diagram considering the important factors controlling evolution of imperfections in this project. First, the “surface transfer function” controls the evolution of imperfections during painting. Then, the “optical transfer function” determines the visibility of imperfections after each coating step.

7.2.1 Surface Transfer Function

To understand imperfection evolution during painting, first the coating layer formation process during painting must be understood. Film formation processes for liquid coatings (Figure 7.4) are reported to include: 1) wetting – bulk liquid is brought into contact with the substrate, 2) motions of the coating device/substrate which cause new surface formation in the

liquid film, 3) dilation/extensional flow, 4) equilibration or leveling, and 5) drying/curing (53). The film formation processes for powder coatings are: 1) wetting – powder is brought into contact with the substrate, 2) curing- coalescence of molten individual powder particles to form a continuous film (Figure 7.5), and 3) flow of that continuous film from an irregular surface to a smother surface (Figure 7.6) (29). From these film formation processes, it is hypothesized here that shrinkage may be the most important factor controlling imperfection evolution. Generally, shrinkage occurs at the drying/curing stage for liquid coatings and at the curing stage for powder coatings. Evaporation of solvents and crosslinking reactions during curing are the main causes of shrinkage in liquid coatings. Coalescence and crosslinking reactions are the main factors for shrinkage in powder coatings. The influence of shrinkage is discussed in detail below.

Generally, the shrinkage ratio is expressed;

$$\varepsilon = \frac{\Delta l}{l} = \frac{l_{uncured} - l_{cured}}{l_{uncured}} \quad [7.1]$$

where l is the length dimension before or after drying /curing. The shrinkage can be either in the lateral direction (width or length) or in the vertical direction (thickness) if substrate surface is flat. Here, we consider only shrinkage in vertical direction (thickness). The shrinkage ratio varies with coating type, and it has been reported that the shrinkage ratio of liquid coatings is about 4-10%, while the shrinkage ratio for powder coatings is less than 2% (18, 34). In our analysis below, the electrodeposited coating was excluded from the modeling of surface evolution, because ED coating has different film formation mechanisms.

Figure 7.3: Schematic diagram explaining the factors important for evolution of imperfection surface.

Figure 7.4: Schematic diagram of a liquid coating process (29).

Figure 7.5: Schematic illustrating the coalescence of molten powder particles (30).

Figure 7.6: Schematic diagram of the flow of a sinusoidal surface of a continuous fused film (30).

Imperfection topography evolution for one coating layer is proposed to be described by the following expression, when controlled by vertical shrinkage of an initially organic film:

$$PS_{\text{cured}} = PS_{\text{wet}} \epsilon - US (1-\epsilon) \quad [7.2]$$

Where ϵ is the shrinkage ratio, and other parameters are discussed below. This expression applies at every point on the surface and is used to generate the painted surface (PS_{cured}) profile from the underlying surface (US) profile. In this expression, the position of the liquid paint film (PS_{wet}) is first set by adding the nominal paint thickness to the highest vertical point on the underlying surface. The underlying surface profile is obtained here by profilometry, and the other profiles are then calculated assuming a given film thickness and shrinkage ratio. It is also easily demonstrated that the residual imperfection depth after curing is given by;

$$D_{\text{residual}} = D_{\text{initial}} * \epsilon \quad [7.3]$$

where

D_{residual}	= residual depth
D_{initial}	= initial depth
ϵ	= shrinkage ratio

This expression is true for each individual liquid layer application, independent of the film thickness, for the conditions assumed in this simple model. Figure 7.7 shows a schematic which helps define the parameters used in these expressions. Using Equation 7.2 and assuming a perfectly uniform (flat) liquid paint film after application, the 2-D trace after painting is predicted from the 2-D trace before painting (i.e. after ED coating). Figure 7.8 shows a calculated 2-D trace for a scratch type imperfection after one layer application (using a paint thickness of 23 μm and shrinkage ratio of 10%) along with an actual profile (after primer surfacer). The 2-D traces from both calculated and experimental results show very similar properties. Figures 7.9 and 7.10 show a series of 2-D traces for the same imperfection after multiple coating steps, including both calculated and measured data. Figures 7.11 and 7.12 show magnified 2-D traces from Figure 7.9 and 7.10 after primer surfacer and top coat application and curing. Table 7.1 shows the conditions used for these calculations. The assumed shrinkage ratio was 5% for each liquid coating layer.

Figure 7.7: Schematic illustration of parameters used for modeling topography changes.

Figure 7.8: Series of 2-D traces showing change of imperfection profile after primer surfacer. (40⁰ indenter, 25N, GA)

Figure 7.9: Series of 2-D traces showing change of imperfection profile after painting (calculated, based on 2-D profile for scratch, 40° indenter, GA).

Figure 7.10: Series of 2-D traces showing change of imperfection profile after painting (experimental data, scratch, 40° indenter, GA).

(a) Calculated

(b) Actual

Figure 7. 11: 2-D traces showing enlarged imperfection profile after primer surfacer.

Figure 7.12: 2-D traces showing enlarged imperfection profile after top coating.

(a) Calculated

(b) Actual

Table 7.1: Conditions for simulating topography evolution

Painting	Thickness before curing	Shrinkage ratio
ED-Coating	As-received 2-D trace	-
Primer Surfacer	45 μ m	5%
BC	51 μ m	5%
CC	20 μ m	5%

Figure 7.13 shows the effect of shrinkage ratio variations on calculated profiles after painting. Increasing the shrinkage ratio increases the residual imperfection depth. Figure 7.14 shows the effect of paint thickness on calculated residual imperfection behavior. Paint thickness changes do not affect imperfection evolution when a constant shrinkage ratio is used. However, experimental data show that increasing paint thickness decreases residual imperfection depth after painting. Thus, it is clear that other factor (resin properties, curing time, etc.) affect shrinkage of the paint and topography evolution. It is also noted that residual stresses in the paint film are ignored in this analysis, and thicker films are likely to distribute the shrinkage strains over a greater lateral dimension, thereby diminishing visibility somewhat.

From the model presented above, it was shown that shrinkage considerations predict 2-D profile changes after each coating step quite successfully. These results are extremely important, and make it clearer why a 100 μ m paint film is unable to hide surface imperfections that are only several μ m in height or depth. The implications to painting process development are also considered to be potentially important, indicating that the number of layers, and the shrinkage, are potentially much more important than the film thickness. It is expected that this analysis will help quantify the relative importance of width vs. depth features of the imperfections after painting.

Figure 7.13: Enlarged 2-D traces showing effect of shrinkage ratio on imperfection profile after BC/CC. (Substrate profile of Figure 7.7)

Figure 7.14: Enlarged 2-D traces showing effect of paint thickness on imperfection profile after primer surfacer. (Shrinkage ratio 5%). (Substrate profile of Figure 7.5).

7.2.2 Optical Transfer Function

To understand why imperfections having a size smaller than the resolution of the human eye ($88.6\mu\text{m}$) are visible after painting, we are currently considering optical theories developed by researchers studying imperfections on smooth surfaces (43,51). The overall appearance of a surface having an imperfection is a combination of spectral and geometrical factors, seen by the eye and interpreted by the brain. Spectral characteristics depend on the wavelengths of light incident on and reflected from the object. Geometrical factors are related to the location of light sources illuminating the objects, the surface characteristics of the objects being viewed, and the location of the eye of the observer. In this project, only one light source and one paint color were used. Thus, we can exclude spectral issues from the consideration of overall appearance of the surface.

Variations in reflected intensity control appearance. Reflected intensity is a strong function of the amount of illumination, viewing angle, imperfection topography, and material. Mundi et al. (50) reported that several phenomena play a role in the distribution of light reflected (scattered) from a surface. The most important effect is the variation of surface orientation and shadowing. For surface imperfections after painting, the surface is dominated by the gradual variation corresponding to the surface distortion associated with the imperfection. However, the quantitative relationships between these variations and their visibility is not yet understood. Consequently, this analysis is continuing beyond the project completion, and hopefully will provide further understanding of the factors controlling appearance of a painted surface. It is expected that this analysis will help quantify the relative importance of width vs. depth features of the imperfections after painting.

7.3 Evolution of Laboratory Imperfections During Forming

In the forming portion of this study, two types of imperfections were created in the laboratory. Dent-type imperfections were induced normal to the surface of the zinc coating with modified hardness indenters. Outdings were simulated by placing hard particles of varying diameters at the sheet/punch interface during stretch-forming operations, resulting in an imperfection which protruded through the thickness of the zinc coated sheet.

Dent depth and outding height are critical parameters when evaluating appearance of imperfections after forming and painting. Depth and height of an imperfection is dependent on the mechanical properties of the material and the geometry of the object that created the imperfection. For a specific imperfection type, imperfection height and or depth varied according to the yield strength of the material, as depicted in Figure 7.15. Outding height is shown with a 0.381 mm diameter ball and dent depth is shown in the Figure with a 4° indenter for the unstrained sheets. An anomaly is noted with a circle, distinguishing the GA data point from the rest of the data series for outding height as a function of strength. The GA sample was considerably thicker than the other two materials (0.889 mm versus 0.701 mm), suggesting a thickness effect on producing outdings in the laboratory. Dents and outdings on the (stronger) EG coated steel sheet were characteristic of the smallest depth and height, while dents and outdings on the GA coated steel sheet were the most severe in depth and height (disregarding the

anomaly). The following sections will focus on the effects of biaxial strain of dents and outdings, addressing evolution mechanisms for each type of imperfection.

Figure 7.15: Dent depth and outding height ($\epsilon=2\%$) as a function of yield strength.

7.3.1 Evolution of Dents

Loading an indenter normal to the sheet surface, as shown in Figure 2.3 produces dents. The material directly beneath the indenter plastically deforms, and the surrounding material deforms elastically. Optical profilometry was utilized to monitor dent evolution with strain. Zinc coated steel sheets containing laboratory-induced dents were characterized with profilometry by defining a local area unaffected by the imperfection, referred to as the background roughness, and a local area containing the imperfection that was defined as the imperfection roughness. For the purpose of this discussion, dent evolution will be addressed according to the variation in background and imperfection (dent depth) roughness with strain, respectively.

The background region that was defined with profilometry consisted of the local region surrounding the imperfection in Figure 2.3 that deformed elastically during indentation. This background region responded to subsequent forming by roughening (Figure 6.8). Roughness is believed to increase during stretch-forming operations as a consequence of the different

tendencies of grains to thicken or thin during plastic deformation. Consequently, there are multiple interpretations concerning texture effects of zinc coated steel sheets during forming.

Wichern (67) proposed that roughening and smoothing mechanisms in zinc coated steel sheets were a result of three sequential steps that occur during stretch-forming operations: twinning, slip, and heterogeneous texture roughening. This sequence is dependent on the crystallographic texture of the zinc coating. Zinc coatings that are basal textured will follow this sequence; however, zinc coatings that are predominantly comprised of zinc intermetallic compounds (e.g. galvanneal coating) do not follow the sequence defined by Wichern (67). The surface roughness may also be influenced by the deformation response of the underlying substrate.

It has been suggested that initially, zinc coated steel sheet roughens as twinning is promoted at low strains. As strain increases, additional slip systems become favorable for dislocation glide resulting in a smoothing effect. As plastic deformation ensues, neighboring grains are constrained at the grain boundaries, inducing compatibility issues, thereby increasing roughness. These predicted zinc grain orientation changes with increasing strain are depicted in Figure 7.16. This shows a schematic representation of the proposed behavior of a hot dipped galvanized electron-beam textured (HDG-EBT) zinc coating on a steel substrate (67)

A plot of the corresponding roughness behavior as a function of strain, as observed by Wichern (67), is shown in Figure 7.17. Here, roughness as a function of strain is hypothesized to occur in three stages: twinning roughening, basal slip smoothing, and heterogeneous texture roughening. Initially twinning causes surface roughening, as new slip systems become oriented favorably for dislocation glide there is some smoothing. Finally, when there is no clear favorable mode of deformation, plastic deformation with adjacent grain constraint becomes the roughening mode (67). Samples containing imperfections in this study were strained in the region hypothesized as heterogeneous texture roughening in Figure 7.17 (67)

Figure 7.16: a) Representation of texture at initial strain application. b) Twinning deformation of properly aligned grains begins and new orientations which favor slip deformation become numerous. c) As slip deformation begins, smoothing ensues for a HDG-EBT material (67)

Figure 7.17: S_q versus e_{vme} plot highlighting the mechanisms responsible for the different roughening and smoothing effects observed during Marciniak punch deformation of a HDG-EBT material (67).

A comparison of the background roughness between the three zinc coated steel sheets was shown in Figure 6.8 as a function of strain. The incremental increase in roughness behavior can be analyzed for the three zinc coated materials by comparing the slopes of the lines associated with the GA, HDG, and EG background roughness, respectively. The slope of the GA material is equal to $0.1185 \mu\text{m}$, while the HDG and the EG slopes are equal to $0.0136 \mu\text{m}$ and $0.0146 \mu\text{m}$, respectively. This is a difference of one order of magnitude between the GA material and the HDG and EG materials. The slight difference in background roughness as a function of strain for the HDG and EG materials is consistent with the as-received roughness for the two materials, which varies according to microstructure. The HDG coating was slightly less rough compared to the EG coating due to the difference in coating deposition processes. The associated microstructure is seen in Figure 6.42.

In contrast to the pure-Zn coatings, the GA coating is very brittle in nature due to the intermetallic phases present in the microstructure. As mentioned previously, the delta phase in a GA coating comprises most of the microstructure and is intrinsically brittle due to a limited number of slip systems available at room temperature. The delta phase is accompanied by a gamma layer at the substrate/coating interface. These intermetallic phases are ordered phases and there is an additional slip constraint. Intermetallics create large Burgers vectors and the requirement to create an antiphase boundary with dislocation motion produces higher average flow stresses (56). As a result, the GA coating accommodates substrate strain by the development of an array of through-thickness cracks. These cracks are evident as black linear features in Figure 6.6, and as sharp local variations in profile height in Figure 6.7. Crack depth increases as a function of strain, corresponding to the slope of the line (0.118 versus 0.014) in Figure 6.8 for the GA background roughness.

Concurrently, the local roughness of the area containing the dent-type imperfection (geometrical measure of the surface) steadily decreased as function of strain until about 4% strain, when it leveled off and remained constant for both the HDG and EG materials. This initial decrease in roughness is consistent with the forming operation in which the dent experienced biaxial strain while being pressed towards the free surface of the sheet, losing symmetry as the surface continued to roughen in the process. The parabolic shape of dent created from a 4° indentation on the EG surface clearly begins to flatten as strain increases. This change in dent geometry as strain increases also appears to be a function of the substrate yield strength. The effect of a fixed imposed strain on the apparent flatness is diminished from the EG, to the HDG, to the GA material. An example of the smaller change in dent geometry with strain is shown in Figure 7.18 for the softer HDG material. Figure 7.18 shows the x-profile across the center of a dent before and after forming to 7% strain. A comparison between the two profiles illustrates the variation in dent geometry, consistent with a decrease in the local roughness (i.e. dent depth) of the area affected by the imperfection, along with the increase in the background roughness with strain.

Figure 7.18: Evolution of a dent (4° indentation) on a HDG coating through the stages of forming, manipulated from output of 2-D traces during optical profilometry.

The GA material exhibits similar behavior at low strains in Figure 6.9; however, as strain increases, the R_a of the imperfection area increases rapidly with a slope consistent with that of

the background R_a in Figure 6.8. The slope of the curve in this case is attributed to the increase in crack density and depth with strain. Overall, the decrease in the roughness of the local area containing the imperfection for all three materials is consistent with a decrease in dent area with strain, consistent with the local flattening of the microstructural features in Figures 6.45-6.50.

Dents produced within the GA coating through-thickness were painted following forming at 2% and 7% strain. Dent depth decreased from about 7 μm to 2 μm following ED-coating for the 2% strain condition, and 5 μm to 1 μm after ED-coating (28-32 μm) for the 7% strain condition. Following top-coating (total thickness = 157-201 μm), dents were invisible with profilometry and the naked eye. Imperfections in the paint were noted in Figure 6.16; however, no further analysis was conducted of the paint-induced irregularity.

7.3.2 Evolution of Raised Imperfections

Outdings were simulated by placing hard particles of varying diameters at the sheet/punch interface during stretch-forming operations, resulting in imperfections protruding through the thickness of the zinc coated sheet. This process of producing outdings in the laboratory was utilized to model entrapped particles in the forming process between the forming die and the sheet during automotive manufacturing. Two-dimensional traces across the center of the outdings were utilized to extract lateral dimensions of the imperfection as a function of strain. Outdings were imperfections of raised geometry that stretched mechanistically according to the induced biaxial stretching strain. Imperfection width varied directly with strain (Figure 6.38), while the imperfection height of a similar imperfection varied inversely to strain, and is shown in Figure 7.19 for a HDG coating. The GA and EG materials containing outding imperfections experienced similar behavior as a function of strain.

At 7% strain, the imperfection appeared to have a crumbling effect on the GA surface (Figure 6.28 and 6.29). Powdering tests confirmed that the GA surface had experienced surface damage and was characterized to be powdering and flaking. This is consistent with the cracks in excess on 10 μm in Figure 6.28.

7.4 Evolution of Industrial Imperfections

Imperfections originating from non-uniform coating deposition and chemical reactions during steel production were analyzed for forming response and appearance after painting. Dross lines and sink roll marks were representative of imperfections originating from non-uniform coating deposition, while white spots and small white spots were examples of imperfections that could have originated from chemical reactions or mechanical effects. The following section will address their evolution as a function of forming strain, and appearance after painting.

Figure 7.19: 2-D trace of an “outding” created during a stretch forming experiment on a HDG coating. Laboratory-induced out-dings were created with a 440 stainless steel ball bearing of 0.381 mm in diameter at the sheet/punch interface. Illustrates imperfection evolution from 2% to 7% strain.

7.4.1 Evolution of Sink Roll Marks and Dross Lines

Dross particles are probably a result of alloying and solidification (non-uniform coating deposition), while sink roll marks might be a consequence of entrapped particles that form due to alloying, solidification, and mechanical processes involved in galvannealing (non-uniform coating deposition coupled with mechanical response). Both imperfections occur during steel processing and are probably present within the through-thickness of the zinc coating. As forming strain increased, dross lines and sink roll marks increased in height, and decreased in lateral dimension, becoming more apparent after stretch-forming.

Figure 7.20 summarizes the evolution of sink roll marks through the stages of forming and painting, plotting the 2-D traces. The profiles are summarized in Figure 7.20 and the local area containing the sink roll mark is designated with arrows in the figure. The radius of curvature of the surface profile following top-coating corresponds to the die curvature. Similar behavior was observed for dross lines as a function of strain. However, the imperfection height for a dross line became more pronounced after ED-coating, becoming less visible following top-coating.

Figure 7.20: Evolution of a sink roll mark on a HDG coating through the stages of forming and painting, manipulated from output of 2-D traces during optical profilometry.

Sink roll marks and dross particles can be thought of like inclusions which occur somewhere between the steel substrate/zinc coating interface and the free surface of the coating. Deits (68) proposed a model, illustrated in Figure 7.21, that predicts inclusion migration through zinc coatings. Deits (68) specifically developed the model to depict inclusion migration through electrogalvanized coatings during deep drawing. In this model, the EG coating is in compression rather than tension because of the deep-drawing strain state, and increasing in thickness compared to a decrease in thickness associated with tensile stresses experienced during biaxial stretching. It is assumed that inclusions within HDG coatings behave in a similar manner to EG coatings since the two zinc coatings exhibit similar deformation behavior, and surface properties. This model helps explain the change in imperfection height between the profiles for the as-received and the 4% strain conditions in Figure 7.20.

Deits (68) concluded that inclusions are generally less ductile than the steel substrate, and do not plastically deform during forming. The substrate flows around the inclusion pressing it through the coating through-thickness towards the free surface of the coating as pictured in stage I of Figure 7.21. In stage II the inclusion migrates towards the free surface of the coating, the

coating flattens as it comes in contact with the top platen, and the compressive strain in the hoop direction increases. As drawing continues, the coating slides against the top platen, creating a frictional shear stress at the coating surface. Failure of the zinc coating leads to particle removal that occurs when the shear stress on the zinc coating/inclusion interface reaches a critical value, depicted in stage III of Figure 7.21.

Figure 7.21: Schematic representation of a three stage model depicting migration of substrate surface inclusions towards the free surface of the zinc coating during drawing (68).

7.4.2 Evolution of White Spots and Small White Spots

Optical profilometry confirmed that white spots were imperfections of raised geometry while small white spots were characteristic dent-type morphologies. After forming, both spots became undiscernable with optical profilometry after forming. Changes to the overall profile of the coating surface unassociated with the imperfection dominated over changes to the

imperfection itself, since crack depth in the GA coating was greater than the depth or height of the imperfection. In this case, the GA coating microstructure (Figure 6.40) and deformation response dominated the surface profile. An example of the evolution of spots on a GA surface is shown in Figure 7.22, showing a small white spot through the stages of forming and painting. The small white spot is designated with an arrow for the as-received and formed conditions, while the cracks are more evident as sharp local deviations in the surface profile. The small white spot disappears after ED-coating, while the cracks are still apparent. After top-coating the surface profile is characteristic of the die curvature used to impose biaxial strain in the HDG material.

Figure 7.22: Evolution of a small white spot on a GA coating through the stages of forming and painting, manipulated from output of 2-D traces during optical profilometry.

Deits (68) proposed a model, presented in Figure 7.23, which illustrated the importance of tensile stresses on coating cracking zinc coated steel sheets. Stage I depicts the stresses that develop at the coating to accommodate applied tensile strains in the steel substrate. Tensile stresses are transferred to the coating through shear stresses that develop at the coating/substrate interface. These localized shear stresses create a stress concentration factor at the coating/substrate interface, which is a consequence of the difference in elastic moduli across the coating/substrate interface. The zinc coating eventually reaches a critical stress state where it is unable to accommodate the elastic deformation in the substrate. As a result, cracks initiate at the interface and propagate towards the free surface until the tensile stress is relieved, illustrated in stage II of Figure 7.23. Observations of the cross-section through the deformed GA sheet (Figure 6.51) supports Deits (68) theory of crack initiation at the coating/substrate interface.

When tensile stresses in the coating are relieved, the coating acts as an elastic body. Applied shear stresses at the coating/substrate interface approach a critical state as tensile deformation continues, resulting in coating separation from the interface. This process continues until a small portion of the coating remains attached to the steel substrate. The small region left over at the interface remains attached, preventing flaking, and is shown in stage III.

The increase in crack depth as a function of strain outlined by Deits (68) is best represented in a comparison between the 2-D traces for 2%, 7%, and 11.25% strain. As strain increases, crack depth increases. The increase in crack depth and density with strain dominates the surface profiles for both the white spot and small white spot, suggesting that the imperfection of interest is insignificant compared to the coating microstructure and deformation behavior with strain.

Figure 7.23 Schematic representation of a three stage model depicting zinc coating racking resulting from tensile strains (68).

7.5 Factors Controlling Imperfection Appearance After Forming and Painting

Imperfection appearance after forming and subsequent painting is directly dependent on location within the zinc coating, zinc coating microstructure, and subsequent deformation response, along with the mechanical properties of the steel substrate. Figure 7.24 compares the imperfection location within the zinc coated steel sheet. Figure 7.24a shows an inclusion in the zinc coating that solidified during processing, while Figure 7.24b depicts a hard particle at the die/sheet interface that forms an imperfection as result of external contact during forming. Figure 7.24c depicts a surface morphology change on the zinc coating that occurs due to external contact with a hard object. Imperfections that formed within the coating during steel production, such as dross inclusions, increased in height following forming. In contrast, imperfections that formed from external contact through the material thickness prior to forming, such as dents, (Figure 6.31 vs. Figure 6.33) decreased in height after stretch-forming.

Figure 7.24: Imperfection location according to forming mechanism, (a) non-uniform coating deposition, (b) external contact (Automotive Manufacturing) by a particle at the die/sheet interface, (c) external contact (Steel Production).

In Figure 7.24, imperfection origin is listed according to severity after forming. In general, imperfections that formed during coating as a consequence of non-uniform coating deposition were more apparent after stretch-forming. Dross lines and sink roll marks evolved according to the inclusion model proposed by Deits (68) which was discussed earlier, and were still visible after ED-coating with both profilometry and the naked eye. Imperfections that resulted from external contact during forming in Figure 7.24 b decreased in height as a function of strain. Finally, imperfections that were a consequence of external contact prior to forming in Figure 7.24c (simulated here with conical indenters), decreased in depth as a function of strain (Figure 6.9), and were invisible after top-coating (Figure 6.13 and 6.17).

Coating microstructure controls the deformation response of the material, and plays a significant role in the imperfection response to deformation, along with paintability of the zinc coated steel sheet. A GA coating cracks to accommodate substrate strain (Figure 6.46), and can eventually lead to coating degradation in the form of powdering (Figures 6.54 and 6.55), flaking, or galling. Material degradation significantly increases roughness of the material (Figure 6.8), masking imperfections of interest.

8.0 CONCLUSIONS

Surface imperfections were created in the laboratory and compared with real imperfections on commercially produced coated sheet steel surfaces. The response to painting and forming of these imperfections was analyzed, and critical developments and conclusions are highlighted below.

1. Methodologies were developed to create imperfections in the laboratory having various geometries that simulate important characteristics of industrial surface imperfections, providing repeatable imperfections for systematic evaluation. A scheme for classifying imperfections was also discussed.
2. Three-dimensional optical profiling was demonstrated to be a powerful tool for assessing imperfection topographies, and their evolution during painting or forming. This methodology yielded quantitative information, and was able to assess the geometry of surfaces at much higher resolutions than the capability of the human eye.
3. Many of the commercially produced imperfections were invisible after painting. The range of amplitudes or severities was much greater for the laboratory produced imperfections, however, and only the least severe were invisible after painting. Optical profilometry provided sufficient resolution to detect some imperfections that are not completely eliminated by painting, but which are completely invisible by human inspection.
4. From analysis of both laboratory and industrial imperfections, it was suggested that initial imperfection amplitude (depth or height) is the most important geometrical factor controlling visibility after painting. The visibility of raised imperfections and dent or pit-type imperfections having comparable amplitudes was similar after painting. Imperfections having initial amplitudes greater than about 3? m may remain visible after painting, despite the film thickness of the paint being on the order of 100? m.
5. Three different organic coating systems were evaluated in this work, and the results suggested that the system incorporating a high-build powder primer surfacer improved the ability of the paint to cover or attenuate imperfections. Imperfections were slightly more visible after painting for a galvanized substrate in comparison to cold-rolled, electrogalvanized, and hot-dip galvanized substrates, possibly due to a different electrochemical response of the galvanized coating in the phosphate and e-coat processes.
6. A simple model was developed to predict the “surface transfer function,” or the evolution of topography after each painting step. Shrinkage considerations during curing of liquid layers were quite successful in calculating 2-D profile changes during painting, and make it clearer why a 100? m paint film is unable to cover surface imperfections that are only several micrometers in height or depth. It was suggested that reduced film shrinkage during curing and increased numbers of liquid film layers are potentially more important than overall film thickness.

7. The behaviors of different surface imperfections during macroscopic straining were found to differ dramatically, depending on the characteristics of the initial imperfection. The depth of dent-type imperfections was found to diminish during straining, for example, while dross-type imperfections on coated sheet increased in height during straining, even in the absence of direct contact between the dross particles and the forming tools.
8. The background roughness increased during straining for all of the coatings examined, including electrogalvanized, hot-dip galvanized, and galvanized, although the extent of roughening was much greater for galvanized sheet, due to the extensive network of microcracks that formed through the coating thickness.
9. Some shallow imperfections were found to disappear during straining. The microcrack “imperfections” created during forming of galvanized remained visible after e-coat primer application, but were completely invisible after painting.

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