The coating of steel strip with aluminium powder by roll compaction

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THE COATING OF STEEL STRIP WITH

ALUMINIUM POWDER BY ROLL COMPACTION

by

D.N. Skinner

Supervised by: D. N. Skinner

Director of Research: Professor D. C. Freshwater

B. Scarlett

P. J. Lloyd

Submitted for degree of Doctor of Philosophy of
Loughborough University of Technology.

October 1971
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I also wish to thank the staff of the Particle Technology Group for their conscientious assistance during the past three years, especially my future wife Miss Christine Bostock who has endured myself and my thesis both during the day and in the evenings.

I am grateful to Mrs. C. Grant for typing from the manuscript.
This thesis examines a process in which mild steel strip is coated with aluminium to produce a cheap, strong, corrosion-resistant material. The steel strip is initially wetted and sprinkled with a thin layer of aluminium powder. After subsequent drying the strip is passed through a set of two-high rolls to compact the coating and provisionally bond it to the steel. The product can be coiled and finally sintered.

A survey of the existing literature reveals that one of the main problems with the process is that air is expelled from the powder as it compacts. At higher roll speeds this tends to fluidise the powder entering the roll nip and cause uneven coating. Several methods of preventing this have been suggested but previous workers have tended to concentrate on adding binders to the powder. This work therefore confines itself to the actual mechanical behaviour of powder and substrate in the roll nip.

A technique is presented in which the results from an unusual item of powder compaction equipment can be used to predict pressure profiles and final coating densities for this type of two-component system. Experimental evidence indicates that the analysis is not valid at higher roll speeds even if allowance is made for the additional pore pressure caused by entrained air. However, if more information could be obtained on the behaviour of powders at high strain rates then the technique could be a valuable design tool at all roll speeds for any powder-solid system.
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A Real contact area between two surfaces

$A_c$ Cross section area of compact.

$A_o$ Cross section area of graduated glass tube.

B Ratio of radial to axial stresses in a die.

D Compact diameter.

$D_{co}$ Density of aluminium (2.7 grms/cc.)

$D_c$ Density of aluminium compact.

(Subscripts 1 and 2 for density at entry and exit side of roll nip.)

$D_s$ Density of steel

E Young's Elastic Modulus.

$E'$ Percentage void space in a compact.

F Shearing force at aluminium-steel interface.

H Hydrostatic pressure.

$K_1, K_2$ Constants used in relating transverse to axial stresses in a compact.

$K_3, K_4$ Constants used in defining the stress-strain behaviour of a compact.

$K_a$ Constant defining the air permeability of a compact of known voidage.

L Compact length

N Normal force.

$N'$ Normal force per unit area of real contact.
NOMENCLATURE (cont.)

P  Radial pressure at a point on the roll surface.
P_{av}  Average pressure over contact arc.
P_t  Pressure on top punch of a die.
P_b  Pressure on bottom (static) punch of a die.
P_a  Air pressure.
P_c  Pressure acting on the particles in a compact.
R  True roll radius.
R'  Radius of flattened roll.
S  Shear force between contacted surfaces.
S'  Shear force per unit area of molecular bond.
T  Shear strength.
T_s  Tensile strength of steel.
U  Coefficient of uniformity of strain.
V_a  Air velocity through compact pores.
V_c  Velocity of compact layer.
V_s  Velocity of steel layer.

(Subscripts 1 and 2 refer to velocities at entry and exit side of roll nip.)

W  Powder volume.
Y  Height of element in roll nip.
NOMENCLATURE
(cont.)

\( a_m \)
Smooth-metall surface area.

\( a_p \)
Surface area containing pores.

\( C \)
Constant of deformation for roll flattening.

\( e \)
Total strain.

\( e_1 \)
Strain in soft metal.

\( e_2 \)
Strain in hard metal.

\( f \)
Friction coefficient (general).

\( f' \)
Coefficient of friction between compact and die wall.

\( h \)
Roll gap.

\( h_0 \)
Height of oil in graduated tube.

\( k \)
Yield criterion constant.

\( l \)
Length of steel tensile test sample.

\( l_0 \)
Initial length of test sample.

\( n \)
Viscosity

\( q \)
Average horizontal stress on the vertical faces of an element in the roll nip. (Subscripts \( c \) and \( s \) refer to compact and steel layers.)

\( s \)
Compact solidity or theoretical density.

\( t \)
Time.

\( v \)
Poisson's Ratio.

\( x \)
Distance from line of roll centre.
NOMENCLATURE (cont.)

\( y \) Height of element in roll nip.

\( y_a, y_c, y_b \) Thickness of pure aluminium, aluminium compact or steel layers respectively.

(Further subscripts 1 and 2 refer to entry and exit side of roll nip.)

\( \sigma_1, \sigma_2, \sigma_3 \) Principal stresses.

\( \sigma_a, \sigma_r, \sigma_t \) Axial, radial and transverse stresses respectively.

\( \theta \) Angle of element to line of roll centres.

\( \mu \) Coefficient of friction between sample and roll surface.
CHAPTER 1

Survey of Existing Literature
INTRODUCTION

It is the intention of this thesis to extend present knowledge of the process in which steel sheet is continuously coated by roll compaction of a metal powder on to its surface. The back-ground to this work lies not only in previous research on the coating process but also in the general roll compaction of powders, basic continuum rolling theory, and a sound knowledge of the mechanical behaviour of the materials involved.

POWDER ROLL COMPACTION; HISTORICAL BACKGROUND

In 1843 Henry Bessemer accidentally produced brass strip by rolling fine brass turnings (1) but it was not until 1902 that the first patent was granted on the process. This was taken out by Siemens and Halske (2) in Germany, who produced metal strip by passing powder vertically downwards through horizontally-mounted rolls. Serious research however, was not started until the Second World War when Germany tried to develop the process for the manufacture of shell cases. The results of this work were published by Naeser and Zirm in 1950 (3). Charles Hardy continued this research in America and by 1956 the Americans were acclaiming the process as a great money saver (4).

THE COATING PROCESS

In Britain research seems to have centred around the 'Elphal' process which was a method of coating steel with aluminium. The original patent (5) was for a process in which the aluminium powder was deposited on the strip by electrophoresis in a solution of ethyl alcohol. Nickel nitrate was added to the solution so that the
Figure 1 The Original ‘Elphal’ Process for Coating Steel Strip with Aluminium (Ref.6)
nickel hydroxide thus produced could help to bond the powder to the substrate. After drying, the coated strip was passed through a normal two-high rolling mill to compact the coating before sintering. (6) (Fig.1)

The process was developed by B.I.S.R.A. but taken over by the John Summers Steel Company in 1963.

Until this time, commercially available methods of coating steel with aluminium had been hot dipping, spraying, or cladding. Hot dipping caused the formation of a brittle Fe-Al alloy at the interface and spraying gave very thick uneven coatings, of only moderate adhesion. Cladding is very expensive. The 'Elphal' process therefore seemed a big advance. It was flexible in that the powder could easily be changed and powder mixtures could be used for alloyed coatings. Either one or both sides of the strip could be coated and the coating thickness was easily controlled.

DEPOSITION OF THE LOOSE POWDER

The early work on 'Elphal' concentrated largely upon the initial coating of the strip with loose powder (7). It was observed that a certain amount of electrolytic deposition occurred at the same time as the electrophoresis and it was suggested that the electrolytic coating could be used to fill the voids between individual powder particles. Also a dry electrophoretic process was tried in which the earthed strip was passed through a charged fluidised bed of powder.

The 'Elphal' electrophoretic deposition process became a commercial reality in June 1964 with speeds up to 20 feet/min. But it was soon found that the process had disadvantages (8). Dragout of ethyl alcohol presented a fire hazard and hydrostatic drag put an
upper limit on the coating thickness. The nickel hydroxide had an adverse effect on the quality of the final bond. Research therefore began to concentrate on the development of a dry coating process.

Canning, Davies, and Gibbon (9) degreased laboratory samples of steel strip and pickled them in 5 vol.% nitric acid for a minimum of 30 seconds. The samples were scrubbed and dried and then -120 mesh powder was sprinkled on to the wetted strip. Agglomerates were broken-up by letting the powder fall through a vibrating electrostatic grid from an ingenious metering device called a Meter Roll. This device consisted of a rotating roll with radial grooves and a powder hopper mounted above so that the upper surface of the roll was the floor of the hopper. The powder entered the grooves and was carried to the lower side of the roll where it was brushed out with stiff wire. Unfortunately it has been found (13) that this device forms oversize particles by compaction within the grooves.

It was found that 'pick-up' (powder sticking to rolls) could be reduced in the compaction of the coated strip by maintaining a high surface finish. The rolls used, gave a Centre Line Average roughness reading of 5 micro inches with a 'Talysurf' surface roughness measuring device.

Bullough and Popplewell (10) also experimented with dry coating techniques and claimed that, for good bonding, the pickled steel strip had to be heated at 380°C for 2-3 minutes so that a brown oxide coating was formed before adding the powder. Electrical conductivity tests indicated that there was no metallic bond between the aluminium coating and the substrate and transmission electron microscopy showed that it was possible for alumina and iron oxide to form the spinel FeAl₂O₄ at temperatures as low as 450°C. The free aluminium was reduced by heating the compact in a dry mixture of hydrogen and hydrogen chloride at 500°C for one hour and the resulting
framework of alumina indicated that the oxide coating on the particles had not been broken.

With all the work on dry processes however, by 1968, B.I.S.R.A. was still unable to roll at speeds greater than 25 feet/min. (11)

**MECHANICS OF THE ROLLING STAGE**

In 1965 the Canadians, Lund and Armstrong, published a paper on the coating of steel with zinc by roll compaction (12). They coated 3 ins x 12 ins strips of steel by shaking the powder through a sieve and investigated the bond-strength after rolling, and after both rolling and sintering, as a function of powder thickness, particle size and rolling load. This is the first paper to get away from the problems of the chemistry of the deposition and to think in mechanical terms. Brown and Jackson (6) had previously observed that 'too high' a rolling load caused blistering of the coating during sintering but they had tended to regard this as a chemical, rather than a mechanical, problem.

Lund and Armstrong investigated the adherence of the coating by bending the strip so as to produce a known strain in the coating. An NT bend produces a strain in the coating of approximately $100\times\frac{1}{N+1}\%$. Thus an OT bend produces $100\%$ strain in the coating. Multiple cracks (cracking) in the coating are a sign of good adhesion but if only a few cracks form and the coating flakes between them (flaking) then the adhesion is obviously not as good. If the coating just cracks in the centre and peels off (peeling) then the adhesion is very bad.

Where the adhesion was very good it was found that the cracks corresponded to the particle boundaries. These cracks were not produced in pure zinc at the same strain so it was postulated that the oxide coating on the initial zinc particles remained a weak link in
the compact.

For any particular weight per unit area of powder coating, it was found that a linear relationship existed between total roll force and percentage reduction of steel substrate. A theory was proposed in which it was assumed that the total roll force is roughly proportional to the length of the arc of contact between roll and strip. Thus it was possible to estimate an approximate total roll load for a given powder coating thickness.

The yield stress of pure zinc is less than 10,000 lbs/sq. ins. whilst that of steel is 35,000 lbs/sq. ins. Lund and Armstrong therefore expected considerable deformation of the zinc before the steel began to deform. However, extrusion tests showed that the zinc was considerably strengthened by its oxide and that its yield strength increased tremendously at increased strain rates. At a rolling speed of 3 ft/min. Lund and Armstrong calculated the yield strength of the zinc powder compact to be 58,000 lbs/sq. ins. They also observed that deformation of the steel was essential for successful bonding of zinc. (A similar phenomena has been observed with aluminium (13). Even if the steel is very hard it must be deformed before the aluminium powder will bond to it.)

At 35 ft/min. a feathery pattern was obtained on the coated material which was attributed to strip vibration. It was also found that if the mean particle size was significantly larger than the final coating thickness, then the coating was porous and appeared spotty.

In 1966 Chisholm published a paper on the rolling of presintered copper/lead or copper/lead/tin powder mixtures onto mild steel (14). He concluded that the final density of the coating was not significantly affected by roll speed, in the range 15-48 ft/mins, but that the final density was increased by using larger rolls.
THE JAPANESE PROCESS

The Japanese published a paper on the coating of steel strip with aluminium powder in 1967 (15). Their process, which was developed by Kobe Steel Ltd., uses an adhesive solution to stick the powder to the substrate before rolling. The powder is first made hydrophobic by additions of fatty acids, amines, alcohols, or paraffins so that capillary action does not cause amplification of small thickness variations as the powder is sprinkled on to the strip. The adhesive is dried before rolling. It was noticed that bonding was greater with increased reduction of steel but that too great a load caused blistering during the sintering stage. This was attributed to over-densification of the coating preventing the escape of trapped gases. To overcome this problem it was recommended that two rolling stages be used; one where the reduction is only 5%, followed by a heating stage to degas the coating and then a final roll. Speeds of up to 7 ft/min. are achieved but there is no indication of the reason for this limit.
Figure 2  Tundermann's Arrangement for Experiments on the
Roll Compaction of Iron Powder  (Ref. 26)
TECHNIQUES

The literature on powder-solid rolling concentrates to a large extent on the chemical pre-treatment of the substrate. The work on powder rolling however, is far more concerned with the process mechanics. The various techniques used to roll metal powders can be summarised as below:-

1. Rolling horizontally by temporarily supporting the powder on a substrate (16)

(This was the technique first patented by the American, Charles Hardy, in 1938 and was also used by the Germans in the Second World War (17).)

2. Rolling a pre-bonded powder, produced either from a slurry or by sintering.

(Kalling, Eketorp and Backstrom sintered granulated pig-iron and iron oxide at 100°C and rolled the slab on a multipass basis (18). B.I.S.R.A. have coated a drum with a slurry containing 75 wt.% iron powder in a dilute alcoholic combustible binder and surface active agent (19,20). The binder is volatilised and the bonded slab is then rolled.)

3. Rolling dry powder by passing it vertically downwards through horizontally mounted rolls (3).

It is this last technique on which most research has been carried out (Fig. 2).

THE ROLLING OF DRY POWDER

It must be remembered that with the vertical process the feed mechanism is mainly gravitational whereas with the horizontal system the powder is conveyed to the nip via a supporting solid strip. For
this reason it was possible for Tundermann (21) to propose an equation for powder flow which is based on an empirical flow-rate equation for static hoppers. The area of the hopper opening and the hopper apex angle were taken into one term and compared experimentally with rolling results. It was found that for all the iron powders tested there is one unique relationship between roll speed and the equivalent hopper flow area. Using this relationship the total powder flow could be calculated for any roll speed. The final density and thickness of the strip could not be separated however.

References (3, 22, 23, 24) describe how the powder remains free-flowing and in turbulent motion in the hopper above the rolls but movement ceases as it enters the roll nip. The point at which movement ceases is called the 'Angle of Nip' or sometimes the 'Gripping Angle'. (Fig. 2) Kurtz and Barduhn (23) and Nikolaev (25) propose that the voidage reduction is due entirely to the relative approach of the roll surfaces so that the voidage of the product is entirely a function of Gripping Angle, roll gap, and the powder voidage at the Gripping Angle. A similar analysis is given by Tundermann (26) who experimentally tested the voidage of the strip at various angles by using a Quantitative Television Microscope which measures the ratio of dark to light areas in a section of the partially rolled strip. (Details of the equipment are given by Fisher (27).) Tundermann found that the voidage at the Gripping Angle was slightly more than the tap density of the powder but that the proposed model of voidage reduction seemed to hold above this density. To obtain his samples Tundermann had to stop the rolls and remove the partially compacted powder strip from between them. His conclusions are therefore only strictly true for very low rolling speeds.

At higher speeds the powder density and thickness falls off, either due to a breakdown of the proposed mechanism or due to a
Figure 3  Tundermann's results showing the effect of roll speed on the powder flow rate for two iron powders (Ref. 21)
**TABLE 1 (Ref. 21)**

<table>
<thead>
<tr>
<th>POWDER</th>
<th>TRANS. SPEED (Ft./min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MH40</td>
<td>100</td>
</tr>
<tr>
<td>MH100</td>
<td>55</td>
</tr>
<tr>
<td>MH300</td>
<td>25</td>
</tr>
<tr>
<td>Elec. 100</td>
<td>55</td>
</tr>
<tr>
<td>JJM100</td>
<td>60</td>
</tr>
<tr>
<td>JJM300</td>
<td>22</td>
</tr>
</tbody>
</table>

MH. is Hugan's sponge iron powder  
Elec. is Sintrex electrolytic iron powder  
JJM. is J. and J.Makin sponge iron powder  
The numbers indicate the mesh size: so  
MH40 is -40 mesh powder.
reduction in Gripping Angle. It has been shown (28, 29) that Gripping Angle is reduced by increasing the roll gap.

AIR DISPLACEMENT

When a powder is rolled so that its voidage is reduced, the displaced air has to be squeezed back through the nip counter to the powder flow (24). Vinogradov and Fedorchenko (30) and Worn and Perks (31) carried out a series of experiments with gases of different viscosities to demonstrate the effects of gas viscosity on the flow of powders. They showed that very fine powders produced the best compacts in a vacuum.

Tundermann (21) found that as the roll speed was increased the powder flow rate rose linearly to a transition speed where it began to fall off to a constant value. (Fig.3) The transition speeds for various iron powders are shown in Table 1.

These results show that coarse powders can be rolled at a much higher speed than the fine ones purely due to the effect of expelled air.

Evans (32) attempted to use Leva's (33) correlation for 'Velocity of Incipient Fluidisation' to predict the flow transition speed and pointed out that it should be possible to roll above this speed because incipient fluidisation is only the beginning of fluidisation and may be a long way from true elutriation. It was found that the velocity of incipient fluidisation was smaller than the transition velocity during rolling. This was thought to be due to warming of air in the nip by mechanical action, or due to some of the air passing through the rolls and producing pockets of high pressure gas in the compact. Severdenko and Lozhechnikov (34) showed that the strip was extruded forwards, out of the roll nip by slipping against the roll surfaces. The velocity of the compact relative to the extruded air is there-
fore a variable through the roll nip and Evans could easily have over-estimated his experimental speeds.

By inserting thermocouples into the powder, Tundermann (26) found that temperatures as high as 135°C could be reached during rolling. At the actual particle contact points temperatures could have been even higher. It was observed that: 'high density strip, tightly coiled immediately after compaction, retained this coiled shape after reaching room temperature. It was then impossible to straighten the strip without fracturing it.' Tundermann also found that the expansion of the rolled strip could be as much as 8.5% as it emerged from the rolls. This was too great to be explained by elastic recovery and was thought to be an indication of high-pressure air pockets in the compact.

PRESSURE DISTRIBUTION IN THE ROLL NIP

In 1960 Kurtz and Barduhn (23) suggested that the pressure at any point in the nip is equal to the yield strength of the material at that point. The yield strength can be obtained from a plot of voidage versus pressure from a die compaction test. However, in 1954, Hoar (35) had already pointed out that rolling and static compaction differed drastically because of the wall effect.

In 1970, Aksenov (36) showed that the friction effect of die walls could be eliminated by initially analysing the powder in a set of rolls fitted with gauges to measure the pressure profile. He assumed that the powder would reach a maximum density at the point in the roll nip where the pressure was a maximum and that the density would not then be significantly reduced by the falling pressure on the exit side of the nip. He was therefore able to obtain a stress-strain correlation for the powder in a system where there were no vertical walls. His results were valid for the rolls used, but they
Figure 4  Pressure distribution in the roll nip for iron powder. Showing that the peak pressure is not at the line of roll centres. (Ref.39)
gave no indication of how the maximum pressure could be calculated for another set of rolls (possibly with different diameter and surface roughness) which were not fitted with pressure gauges.

Katashinskii and Vinogradov (37, 38) in 1965 measured the pressure distribution by means of wire resistance gauge dynanometers mounted within the rolls. They showed that the maximum pressure occurred before the line of roll centres and therefore the compact probably slipped forward against the rolls. (Fig.4) These workers also showed (39) that the magnitude of the maximum pressure and length of contact arc are both decreased with a coarser powder.

ROLLING SPEED

Evans and Smith (40) pointed out that if a compact was held at a certain pressure in a die it would slowly become more dense. It is therefore to be expected that low speed rolling would produce denser strip. Their conclusion is born out by Kurtz and Barduhn (22) and by the experimental results of Tundermann and Singer (21).

It has also been suggested that cohesion improves with dwell time at a given pressure (41, 42) so that compact strength should increase with reduced rolling speeds. This again was verified by Tundermann and Singer (21) who found a direct relationship between density and Ultimate Tensile Strength for most of their iron powder samples. It is interesting to note however, that for Högans annealed sponge iron powder, which contains very porous particles, the U.T.S. decreased as roll speed increased for a constant final strip density. It was also found that for all iron powders used, although strip density fell off rapidly above the transition rolling speed, the U.T.S. tended to level-off. Rolled specimens were found to be stronger than die compacted specimens of the same density.
Density distribution across width of MH300 iron powder strip from results of Tundermann (Ref. 26)
Although the stress necessary to plastically deform a solid is greater at higher strain rates (43), Tundermann's results showed a decrease in rolling load at higher speeds. However, in 1970, Satoh (44) published a paper on the die compaction of iron powder at different rates and showed that, within the range 0 - 15 cm./sec. for a filling depth of 5.2 cm., there was a linear relationship between density and compaction speed for a given pressure. This tends to support Tundermann but is a direct contradiction to Evans and Smith (40) who showed that lower speeds produced higher densities at a given pressure. Satoh suggests that the results he obtained are due to the effect of strain rate on the coefficient of friction between compact and die wall. This would explain Tundermann's results if slip occurs between compacted strip and roll surface.

**DENSITY VARIATION**

In 1959, Dougherty (45) had observed that high speed rolling produces strip with high density edges and low density centre. It was thought that this was due to preferred air flow from the strip edges. (Fig. 5. The low density regions on the outer edges of the strip are caused by side flow of the powder. (46)).

Tundermann additionally discovered (26) that across the thickness of the strip the density is always highest at the centre. With thin, low density strip, a low density region was noticed at the centre of the thickness density profile. It was noted that there is a possibility of the high density regions elastically recovering to such an extent as to crack the low density regions.

**PARTICLE DEFORMATION AND ANISOTROPY**

As metal is deformed at room temperatures it undergoes a work-hardening which can be measured quantitatively (47). Matsumura (48)
plotted the microhardness of copper against its degree of strain and then estimated the particulate strain in rolled spherical copper powder from microhardness measurements on the rolled strip. He found that the strain was always less than 20%. By metallographic examination and critical grain growth studies of iron strip, Davies (26) estimated that the maximum average deformation was less than 15%. Armstrong (49) found that die compaction of iron powder at 80,000 p.s.i. produces a strain of less than 6% in individual particles.

Evans and Smith (40) noted considerable anisotropy as a result of rolling copper powder. Tundermann (26) pointed out however, that the microstructure given by these workers is not compatible in the three directions. Repeating their experiments Tundermann found that the elongation of copper particles in the longitudinal direction was negligible even at maximum packing densities. A slight degree of anisotropy was detectable however.
1.03 The Rolling of Solids

INTRODUCTION

Examination of the literature on powder roll compaction and powder coating in the previous two sections, has revealed a great weakness in the understanding of the mechanical processes actually taking place between the rolls. In an attempt to get some idea of what might be going on, the next two sections deal with the rolling of continuum metals and bimetallic strips and include some information on the general mechanical behaviour of plastically deforming solids.

HISTORICAL BACKGROUND

In the first quarter of this century the opinion was current that the longitudinal velocities of elements of metal in any given vertical cross-section were not equal \((50, 51)\). In 1927, Pavlov propounded a new theory which subsequently acquired the name 'The Theory of Rigid Ends' \((52)\), in which the author uses the two rigid, unstressed regions of strip at either side of the rolls as starting points for his analysis. This simplification is similar to the hypothesis that incremental elements in the deformation of a cylinder, for example, are not curved but remain flat and only reposition themselves. The limits of the applicability of Pavlov's theory are only just beginning to be studied.

As a consequence of Pavlov's work several researchers began to consider the possibility that, in certain circumstances, the horizontal velocities of elements of solid in any given vertical cross section could be equal. Sobolevskii \((53)\) in 1933, showed that the velocities were equal if there was slippage between metal and roll. Golovin \((54)\) however, came to the conclusion that slip occurred at
Figure 6  The effect of friction on the pressure profile
during cold rolling  (Ref. 57)

Figure 7  Velocity Profiles in Cold Rolling  (Ref. 56)
(Length of arrows proportional to velocity of element)
either end of the deformation zone but that there was bound to be a sticking region in the middle and Orowan (55) declared that the sticking condition occurs at the point where the friction is equal to the yield strength in shear of the material. (Fig.6) Tselikov (56) published graphs showing the longitudinal velocities of elements of metal in various parts of the zone of deformation in 1944 (Fig.7).

HOT ROLLING

Wusatowski (57) has recently pointed out that sticking between roll and metal always occurs in hot rolling where the yield strength is low, but that in cold rolling, slipping and sticking regions exist with magnitude depending on the roll surface condition and lubrication. Also in hot rolling, work hardening does not occur so the yield strength of the material is constant.

PLASTICITY

Before going into details of the solution of rolling problems it may be useful to include an introductory paragraph on the mathematical treatment of plasticity.

In 1914 Mohr (58) proposed a geometrical representation of two-dimensional stress which came to be called the 'Mohr Circle'. Along the line of action of the normal stresses \( \sigma_1 \) and \( \sigma_2 \) (Fig.8a) no shear stress occurs. However, these two 'Principal Stresses' cause shear stresses in all other directions. Mohr found that the relative magnitude of shear and normal stress in any direction at a point could be represented by a circle on a \( \sigma \) versus \( T \) plot: (Fig.8b). Where \( T \) is the shear stress in any particular direction. The Principal Stresses are the end points of the semicircle where the shear stress is zero.
Figure 8 (a) Stresses on an element in equilibrium

Figure 8 (b) Mohr's Representation of the stress on an element
In 1864, Tresca (59) proposed that the elastic limit of a solid was given by a relationship between the largest and the smallest principle stresses at any time.

\[
\sigma_1 - \sigma_2 = \text{constant}
\]

On a Mohr Circle this is represented by any stress state where the semi-circle touches a line parallel to the normal stress axis (Fig.8b). Mohr extended this yield criterion to cases where yielding was represented by any line above the normal stress axis.

The Tresca yield criterion assumes that the stress in the third direction plays no part in determining the yield condition. In 1913 however, von Mises (60) proposed that failure would occur when a critical strain energy was reached as given below.

\[
(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2 = \text{constant}
\]

This equation was later generalised by Schleicher (61) for cases where the yield condition is a function of hydrostatic pressure.

Hydrostatic Pressure = \( H = (\sigma_1 + \sigma_2 + \sigma_3)/3 \)  

and \( (\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2 = f_N(H) \)

Generally, Tresca's maximum yield criterion is more useful because it is simpler but for most metals von Mises is more accurate.

**SLIP LINE ANALYSIS**

The solution of cold rolling problems is generally very empirical as can be seen from a recent publication in the Ball Bearing Journal (62, 63) where experimental results are published in graphical form and simple equations used to extend their applicability for various degrees of roll flattening or strip tension. However, for metal strips with a width/thickness ratio of at least 10, rolling can be considered as a two dimensional problem because friction between roll and specimen is always great enough to prevent significant lateral expansion (43). It is then possible to study the mechanism by
a technique known as 'Slip-line Analysis'.

A diagram similar to that used by Mohr for stresses, can be constructed to represent the strain conditions at a point; Normal strain is plotted against shear strain. For an incompressible material the centre of the circle must be at the origin because the sum of strains must be zero. The point where normal strain is zero represents the direction of the slip-line and therefore, for incompressible materials, this must lie at 45° to the directions of the principal strains. (64)

A 'slip-line field' is a plot of the slip-line directions for a continuum solid in plane strain. For each element there are two slip-lines which are always perpendicular.

The first slip-line equations were given by Hencky in 1923 (65) and were supplemented by Geiringer in 1937 (66) for states where rate of yielding is important. Prandtl (67) did some early work on slip-line fields and pointed out that for an element in contact with a rough boundary surface the slip-lines are parallel and perpendicular to the boundary, whereas for a frictionless surface they would both be at 45°. Unfortunately, solution of slip-line fields is very complex (68) and even then is limited to systems which are already adequately covered by simple 'technological' theories as in the next section.

THE COLD ROLLING OF THIN STRIP

For thin sheet with a thickness of less than 0.2 inches and a width/thickness ratio of at least 10, the lateral expansion of the strip is not more than 2% so it can be considered as a two-dimensional (plane) strain problem. Also, if the roll radius is about one hundred or more times greater than the strip thickness and there is no sticking between strip and roll, then a simple analysis due to
Figure 9  Diagrams showing the dimensions and Forces acting on an element in a roll nip
von Karman (69) can be used.

Consider an element of isotropic solid of length $y$ and thickness $dx$ in the nip of a roll at angle $\theta$ to the line of roll centres (Fig.9). If the average horizontal stress component across one vertical face of the element is $q$, then a horizontal force balance gives:

$$- d(q.y) + 2.P.R' \sin \theta \cdot d \theta + 2.\mu \cdot P.R'. \cos \theta \cdot d \theta = 0$$

where $R'$ is the effective roll radius allowing for roll flattening.

By geometry:

$$\cos \theta = (2R' + h - y) / 2R'$$

Therefore:

$$\sin \theta \cdot d \theta = dy / 2R'$$

and

$$\sin \theta = x / R'$$

Therefore:

$$\cos \theta \cdot d \theta = dx / R'$$

Substitution of these equations gives:

$$- d(q.y) + P \cdot dy + 2.\mu \cdot P \cdot dx = 0$$

where $\mu$ is the coefficient of friction between strip and roll surface.

As the material is incompressible the strip speed has to increase as it passes through the nip. There is therefore a point in the nip where strip and rolls travel at the same speed. This is called the 'Neutral Point'. At either side of the neutral point the strip is extruded from the rolls so that on the entry side it is travelling more slowly than the rolls and on the exit side it is travelling more rapidly. The direction of the friction force therefore changes as the neutral point is reached. This explains the alternative + or - for the friction term in the above equation.

The elementary theory now assumes that the entire region of strip lying within the roll nip is plastically deforming and that the relationship between the stresses $p$ and $q$ is given by the maximum yield stress condition.
\[ \sigma_1 - \sigma_2 = \text{const.} = 2. T \]

Therefore:
\[ P - q = 2. T \]

where \( P = \sigma_1 \) & \( q = \sigma_2 \)

\( T \) is the yield strength in shear.

Substitution for \( q \) gives:
\[ \frac{1}{2} \cdot y \cdot \frac{dP}{dx} = \mu \cdot P = T \cdot \frac{dy}{dx} \]

The equation for the arc of contact is usually simplified (43) to:
\[ y = h + \frac{x^2}{R'} \]

Substitution gives:
\[ \frac{1}{2} \left( h + \frac{x^2}{R'} \right) \frac{dP}{dx} = 2. T \cdot \frac{x}{R'} + \mu \cdot P \]

This equation was solved by Nadai in 1939 (70).

**DEFORMATION OF THE ROLLS**

It will be noticed that \( R' \) is not the true roll radius but a corrected roll radius allowing for roll flattening. Hitchcock (71) in 1935 proposed this correction because of the very great increase in roll radius caused by elastic deformation at the arc of contact. His equation was:
\[ \frac{1}{R} - \frac{1}{R'} = \frac{16.(1 - v^2)}{\pi.E.x^2} \cdot \frac{P_{av}}{c.x^2} \]

where \( c = 2.9 \times 10^3 \) ton/sq. ins. for steel rolls.

\( v \) is Poisson's ratio for the roll material.

\( E \) is Young's modulus for the roll material.

\( \frac{P_{av}}{c.x^2} \) is average pressure over contact arc.

Since this equation also depends on roll pressure, solution of the problem can only be obtained by successive approximations of \( R' \) until both equations are satisfied.

Elastic deformation of the rolls also takes place along the roll
Figure 10  Axial Bending of Rolls

Figure 11  Pressure Dist. for rolling of solid Aluminium on 7 ins Rolls
Reduction from 0.08" to 0.044"  (Ref.72)
axis, (Fig.10) and this limits the minimum roll size. This is a particular disadvantage because the total roll pressure is usually proportional to the length of the arc of contact and it is therefore desirable to use as small rolls as possible to minimise the total pressure requirement (57).

EXPERIMENTAL EVIDENCE

Application of von Karman's analysis gives two pressure curves for entry and exit of the strip which meet in a peak at the neutral point. In practice however, the so-called 'neutral point' is a region of sticking of finite length. Therefore a true pressure distribution curve is found to be curved at the top. The results of Siebel and Lueg (72) for aluminium are typical of this effect. (Fig.11). Also plotted on the graph is the yield strength of the aluminium which is regarded as the roll pressure at either end of the arc of contact. (Thus the contact arc does not include the regions of elastic deformation at the beginning and end of the plastic deformation zone.) It can be seen that the aluminium is slightly work-hardened during the pass and that its yield strength increases. According to Hill (43) this effect is best allowed for in the von Karman analysis by using the average yield stress for the roll pass as the yield stress (T). However, it would be more accurate to completely change the equation to describe the true relationship between the two principle stresses during work-hardening.

Another factor emerging from Figure 11 is the tremendous pressure required to overcome friction between rolls and strip. Thus, although the yield strength of aluminium is only about 20,000 p.s.i. the maximum roll pressure is more than 100,000 p.s.i. Hill (43) uses a coefficient of friction of 0.08 as typical and this seems to agree with the results of Sims (73) for a roll surface with a
roughness of 10-20 micro inches. Whitton and Ford (74) however, obtained a coefficient of 0.095 for aluminium with dry steel rolls of surface roughness less than 9 micro inches.

The maximum roll pressure would be less for a lower coefficient of friction or if the strip were put under tension so as to increase the horizontal stress component (q).
ROLL PRESSURE

In 1942 Pomp and Lueg (75) claimed that the total roll pressure for clad strip was the sum of the pressures for the same observed reductions of base metal and cladding separately. A graph of roll pressure versus percentage cladding for stainless steel on mild steel, showed a linear relationship. Holmes (76) confirmed this theory but showed that it was only valid for materials of similar yield strength and where friction between roll and material does not change during the compaction.

Gulyaev and Rakov (77) assumed friction at the bimetallic interface to be constant, although they acknowledged that this could not be true due to the gradual formation of a bond between the two layers. They showed that their analysis was valid but only under limited experimental conditions.

Arnold and Whitton (78) carried out a von Karman (69) - type analysis to find a mean yield stress for the composite when cladding a hard metal with a soft metal. They then used this mean value to calculate total roll force.

Their basic assumption in carrying out this analysis was that no slip occurred between the layers. Therefore lateral strains were equal and, for no volume change, longitudinal strains were also equal. Titanium and brass were used as the hard metals and aluminium and copper as the cladding. Surfaces were simply cleaned with emery cloth before rolling took place. Their assumption broke down for hardness ratios (based on Vickers Diamond Pyramid Values) of more than about 3 : 1 and where the hard metal was so much harder than the soft, that it didn't deform at all.
RELATIVE DEFORMATION

Several workers (79, 80, 81) considered the relative deformation of the layers when rolling bimetallic strip. Gulyaev (79) is typical. He describes the degree of non-uniformity of deformation by the coefficient \( U \).

\[
U = \frac{\ln e_1 - \ln e_2}{\ln e}
\]

where

\( e_1 = \) soft metal strain
\( e_2 = \) hard metal strain
\( e = \) total strain

For uniform (equal strains) deformation \( U = 0 \) but Gulyaev found the values of \( U \) to vary between 0.2 and 0.65. Interlayer lubrication increased the values of \( U \), and increasing the total strain reduced the values of \( U \).

An empirical formula has been published (82) for calculation of the initial thickness of aluminium \( (Y_{A1}) \) required to give a certain final cladding thickness \( (Y_{A2}) \) for a known initial thickness of steel \( (Y_{S1}) \) and a total percentage reduction \( (e) \).

\[
\frac{Y_{A1}}{Y_{S1}} = \frac{0.185 + Y_{A2} - 0.285.e}{4.115 - Y_{A2} - 5.465.e}
\]

It can be seen that this signifies non-uniformity of deformation for the cladding of steel with aluminium. No mention is made however, of the surface condition of the strips.

INTERLAYER BONDING

Milner and Vaidyanath (83) have carried out roll bonding tests using aluminium and found that bond strength is proportional to the product of shear strength and area of new surface. The new surface is formed by deformation and lengthening of the interlayer region.
but the actual area depends upon the mechanism of break-up of the oxide film.

Takana and Yosikihi (84) have suggested a geometric relationship to predict the total percentage reduction necessary for bonding and have verified their theory for several different materials.

A review of bimetallic rolling was compiled by Sheppard and Brooks in 1967 (85).
INTRODUCTION

The last two sections, which dealt with the mechanics of rolling of both single layer and bimetallic strips, reveal the importance of two factors which have been virtually ignored by workers in the powder rolling field. These factors are intermetallic friction and bonding. They are examined in more detail in this section.

INTERPARTICLE BONDING

It is interesting to note that when two surfaces are brought together, their true area of contact is very much less than the apparent contact area. Bowden and Tabor (86) found by resistivity measurements between two polished 20 sq. cm. steel plates that, even at a load of 500 kgrms. the true contact area is only 1/400th. of the apparent area. This is due to microscopic roughness of the surfaces.

When two metal particles are pressed together there is an increase in surface energy and a tendency towards local molecular diffusion at both sides of the interface. Eventually the particles bond together by atomic linkages (87). This process (known as 'cold welding') is temperature time and pressure dependent and can be hindered by the presence of a continuous oxide coating (88). Johnson (89) however, has shown that during plastic deformation of contacts, the material at the surface of the contacted regions tends to flow outwards from the centre. This would possibly disrupt any oxide coating. If the temperature at a contact rises above the melting point of the metal, then a 'hot weld' may be formed by normal liquid mixing.

Electrostatic (90) and Van der Waals (91, 92) forces are, except
with extremely clean surfaces, negligible compared with the atomic bonds caused by metallic diffusion and are only relevant in the bulk powder where surface moisture provides a lot of the adhesion.

**PARTICLE INTERLOCKING**

The other major bonding force is Particle Interlocking. This is produced when protrusions on the surface of one particle indent another particle or when the metal of one particle is squeezed into the pores on the surface of another. Bockstiegel (93) proposed that the interparticle bonding force with such a mechanism must be a function of the probability of a pore in one particle being adjacent to a smooth area of surface on the other. Thus:

\[
\text{Force} = \text{constant} \times \frac{a_p}{(a_p - a_m)_1} \times \frac{a_m}{(a_p - a_m)_2}
\]

where

- \(a_p\) is total pore area
- \(a_m\) is total smooth-metal area
- and the subscripts refer to particles 1 or 2.

Bockstiegel found considerable experimental evidence to support his theory.

**INTERMETALLIC FRICTION**

According to MacCurdy (94) Leonardo da Vinci in the Sixteenth Century was the first person to propose that friction is directly proportional to the normal force between sliding surfaces and is independent of contact area. In 1699 Amontons (95) proposed that shear force is proportional to normal load.

\[
S = f.N
\]

where

- \(S = \text{shear force}\)
- \(N = \text{normal force}\)
- \(f = \text{coefficient of friction}\)
Coulomb confirmed Amontons' Laws in 1781 (96) and distinguished between static and sliding friction.

In 1925, Terzaghi proposed that frictional resistance between surfaces is due to molecular bonds over the contact area (97). He assumed that the real contact area was proportional to normal load and shear strength was independent of normal load.

\[ S = A \cdot S' \]

\[ f = \frac{S'}{N'} \]

where

- \( A \) = real contact area
- \( S' \) = shear strength per unit area of molecular bond.
- \( N' \) = normal load per unit area of real contact.

Bowden and Tabor (86) have demonstrated Terzaghi's assumptions for metals and stated that \( N' \) must be the plastic yield strength of the material at each contact. They have also shown that when loading is high, the local temperature at the micro-contact points can exceed the melting point of the metal. This leads to a great divergence from simple Coulomb-type friction.

**SLIDING FRICTION**

Greenwood and Tabor (98) observed that under static loading the mean contact pressure is about six times the yield strength of the material but as sliding commences it falls to about the same order of magnitude due to an increase in contact area.

Dokos (99) claimed that sliding velocity effects friction in three ranges for most materials.

1. **Low velocity:** Coefficient of friction decreases with load.
2. **Intermediate velocity:** Coefficient increases with load.
(3) High velocity: Coefficient decreases with load.

For clean contact surfaces the coefficient always increases with decreasing velocity.
INTRODUCTION

The last three sections have dealt almost exclusively with solid materials so it is now necessary to take a closer look at powders and their peculiarities.

Zapf (100) gives a good account of the differences to be found between various metal powders. The production processes may be grouped under four headings:

1. Reduced
2. Atomized
3. Comminuted
4. Electrolytic

Powder may differ in many properties such as chemical analysis, particle size, particle shape, flow characteristics, apparent density, compressibility, sinterability or green strength, but obviously some of these factors must be linked. In Britain powders are tested according to B.S. 3029 and compressibility is the density of a powder for a given compacting pressure. Compressibility is effected by size distribution so that a powder may have such a wide size distribution that small particles are isolated between large ones and may not be deformed at all (101).

Scarlett (102) points out that size is not a consistently defined quantity. He describes 74 methods of particle sizing which can give many different dimensions such as the diameter of a sphere that would have the same sedimentation characteristics as the considered particle (Stoke's diameter), the side of a square hole through which the particle would just pass, the particle volume, or the diameter of
a sphere with the same particulate volume.

The principles of characterisation of particle shape are dealt with by Heywood (103) who uses the deviation from a sphere, internal porosity, flatness and dendritic nature as his most important criteria.

Characteristics of Bronze, Beryllium and Tungsten powders can be found in the respective references (104, 105, 106).

PARTICLE PACKING

Numerous analyses of packing have shown the importance of particle size distribution upon the maximum packing density (107). McGeary, for example, claims that if spheres of diameter ratio 316:38:7:1 are mixed in the respective proportions 60.7:23.0:10.2:6.1 a packing density of 95% can be achieved with no particle deformation (108).

Smith, Foote and Busang (109) studied experimentally the packing of spheres in a sack and found that the distribution of co-ordination number for the particles was Gaussian with its maximum probability at about 8. As the voidage was reduced however the system gradually became more ordered and close hexagonal and simple cubic arrangements became more common.

Brown and Bennett (110) showed that the relation between voidage and co-ordination number for irregular starch particles is not the same for spheres, and in 1945 Brown and Hawksley (111) made the general observation that the study of sphere packing is not helpful in a consideration of irregular particles.

In 1931 Furnas (112) studied the packing of broken solids and produced a correlation whereby the size distribution of solids could be picked to give the closest packing. This was achieved by fitting each particle size into the voids left in the packing of the next larger size.
DILATENCY

Reynolds published his classic paper on dilatency in 1885 (113). He found that dense sands expand during shear failure whereas loose sands compact during failure. This phenomena of expansion, which is called 'dilatency' is unique to granular materials and is caused by particles having to move sideways before flow can occur.

Reynolds' ideas were taken further by Hvorslev in 1937 (114). He plotted shear stress, normal stress and voidage on a three-coordinate system and found that the failure condition formed a surface subsequently called the 'Hvorslev Surface'.

DIE COMPACTION OF POWDERS

The classic work on powder compaction in a die was published by Unckel in 1945 (115). By a simple static load balance he showed:

\[ \frac{P_t}{P_b} = \exp \left( 4f' B \frac{L}{D} \right) \]

where

- \( P_t \) is pressure on top punch.
- \( P_b \) is pressure on bottom punch (assumed static).
- \( f' \) is coefficient of friction between compact and wall.
- \( B \) is ratio of radial to axial pressure.
- \( L \) is die length.
- \( D \) is die diameter.

Other workers (116,117,118) however, have shown that a distinct distribution of densities (stresses) exists in the die. They conclude:

1. For the same diameter, an increase in height or pressure increases the width of the density distribution.
2. For the same pressure and height larger diameters reduce the density variations.
Figure 12 Results obtained by Long for aluminium on the split die apparatus (Ref.119)
Up to a height to diameter ratio of 2:1 the density at the centre of the base exceeds that at the centre of the top. Above this ratio the opposite is true.

Lubrication of the wall significantly reduces the density variation, but lubrication of the particles has little effect. (In a lubricated die the maximum difference in density was typically 4 - 5%).

Long (119) investigated the relationship between radial and axial pressure in a die, assuming no die wall friction and a perfectly uniform distribution of stress. He pointed out that once the compact had reached such a degree of compaction that the shear strength (T) was constant, then the yield condition would be given by:

\[ \sigma_a - \sigma_r = 2T \]

where \( \sigma_a \) and \( \sigma_r \) are axial and radial stresses respectively. For a non-work-hardening metal at constant density T will be constant so the relationship between \( \sigma_a \) and \( \sigma_r \) will be a straight line of unit gradient. He built equipment to investigate this phenomenon for various materials and obtained curves of the type shown in Figure 12. Relaxation of pressure, once constant density has been achieved, brings the sample back to point C where residual elastic energy leaves a finite value of radial pressure.

In a later paper (120) Long showed that if the radial and normal pressures were normalized by dividing each by the axial pressure required to reach a theoretical density of 90%, then the relationship between radial and axial pressure is almost identical for any material. This indicates that in compaction, beyond the stage where individual particles deform, the strength of the material is more important than the size or shape of individual particles.

In 1948, Balshin (121) proposed a relationship between apparent
powder volume ($W$) and axial die pressure ($\sigma_a$) as shown below:

$$W = K_1 - K_2 \log \sigma_a$$

where $K_1$ and $K_2$ are constants.

Train worked on die compaction of magnesium carbonate and came to the conclusion that three regions of compaction can be observed (118).

1. Particle repacking.
2. Compaction according to the Balshin formula.
3. Fracture or extensive plastic flow.

Long and Alderton (122) have carried out a programme of research into the extrusion of air from die compacts. As would be expected they found that air is expelled more slowly with finer powders at the same pressure but they also found that if a compact with a theoretical density greater than 95% was rapidly formed, some air can be permanently entrained.

**BEHAVIOUR OF A POWDER DURING RAPID LOADING**

Lund and Armstrong (12) noted that the yield strength of zinc is effected by its rate of strain so that even at roll speeds as low as 3ft./min., the strength could be increased from the normal 35,000 lbs/sq.ins. to 58,000 lbs/sq. ins.

In 1948 Casagrande and Shannon (123) demonstrated that, for clay, a 220% increase in compressive strength could be obtained by loading in 0.01 secs. However, only 10 - 15% increase was observed for sand at the same rate. Whitman (124) investigated further and found that for loading below 0.001 secs., a wave was produced which took a measurable time to pass through the sample. Working with Healy (125) he found that lateral strains had to occur before failure took place so that the lateral inertia of the particles at these high loading rates allowed the measured stress to increase beyond the normal failure condition.
THE COATING PROCESS

Early results with 'Elphal' (5) indicated that coating by powder roll compaction could have great economic advantages over other coating techniques. However, it was found to be very difficult to get an even coating of dry powder to stick to the substrate and most research concentrated on finding suitable binders and whether to use electro-phoresis or dry deposition techniques (8). Lund and Armstrong (12) were the only researchers to seriously study the mechanics of the process so the basic problems can be stated quite briefly.

1. The substrate must be plastically deformed or the powder will not bond during rolling.
2. Over-densification of the coating may cause blistering during sinter stage due to the presence of volatile binders in the powder.
3. High roll speeds produce a feathery pattern in the coating.
4. Over-large particles produce a spotty coating.
5. Compact yield strength is increased with strain rate so rolling at higher speeds requires more pressure.

ROLL COMPACTION OF POWDER ALONE

The rolling of a powder alone is slightly different from the coating process in that flow rate is generally controlled by gravity (3). However, research in this field has concentrated to a much larger extent on the mechanism of the rolling and has therefore made observations which are relevant. The most important of these are listed below:

1. Flow rates are limited by the air which is expelled during compaction, fluidising the feed hopper. (Air expulsion
must have a similar effect on the coating process and is probably the cause of the observed feathery patterns produced at higher rolling speeds). The effect is not as noticeable with larger particles since they fluidise less easily.

(2) Temperatures of over 135°C were measured in the compacting powder and it is possible that even higher temperatures exist at the particle–particle contacts.

(3) The recovery of the compact as it emerged from the roll seemed greater than would be expected from a purely elastic recovery. Pockets of entrained air at high pressure were suggested as a possible cause.

(4) Density could vary across the produced strip.

(5) Even at large bulk deformations the strain in individual particles seemed relatively small and there was little sign of anisotropy.

(6) Measurements of pressure profile in the roll nip indicated some degree of slippage between compact and roll surface. This effect means that existing theories for profile prediction are either completely false or of little practical value.

THE ROLLING OF SOLIDS

Conventional rolling theories have long recognised the existence of slip between the sample and the roll surface; especially in cold rolling. Somewhere in the roll nip (just before the line of roll centres) there will be a neutral zone where the perimeter of the roll and the sample travel at the same speed. The sample is effectively extruded from this zone in two directions so that initially it travels
more slowly than the rolls and on the exit side it travels more rapidly (54). The problem is further complicated by the elastic deformation of the rolls themselves which tend to flatten and bend along their axes under load. It is possibly this axial deformation that produces strip with high density edges in the powder process.

Slip-line analysis has been tried as a method of solving the strain mechanism in a roll nip (68) but it is a complex technique which would probably be very difficult to apply to powder systems. More generally, empirical techniques are used (62, 63) but these are even less applicable. The von Karman technique (69) looks promising however, for cases where a thin strip is being rolled. Arnold and Whitton (78) have successfully applied von Karman to bimetallic strips but have used approximations to solve the resulting equations. They point out the importance of knowing whether-or-not slip occurs between adjacent layers.

**BONDING**

For the rolling of bimetallic strip, it has been shown (83) that interlayer bond strength is proportional to the area of new surface produced by the straining of the layers. It could well be this phenomenon which is responsible for the observed necessity for the substrate to be deformed in the powder coating process. If the substrate is not deformed, however great the load, the elastic recovery of the substrate as it emerges from the nip will put the compacted powder layer under a tensile strain which could break it up into a powder again. (Lund and Armstrong (12) noted in zinc the tendency for fairly weak bonding at interparticle contacts). This would explain the facts as observed by Jackson (13).

The literature on bonding seems to lead to the conclusion that cold-welding or particle interlocking would be the main mechanisms at
low rolling speeds. At higher speeds the temperatures at the
contacts may be high enough for hot bonding between particles but there
is no evidence on which to draw conclusions about this. The oxide
coating may be a hindrance to bonding but Johnson (89) has shown
that when two particles are forced together so that they deform
plastically then the regions in immediate contact tend to move outwards
from the centre of the contacted area. This would disrupt any oxide
film.

**FRICTION**

The results obtained by Siebel and Lueg (72) (Fig.11) for rolled
aluminium strip show the importance of friction in raising the roll
pressure above that required for simple yielding of the strip. Bowden
and Tabor (86) have shown that the actual area of contact between
any two surfaces is very much smaller than the apparent area and
Terzaghi (97) has proposed that friction is caused by atomic bonds
at the microcontacts.

It has been shown (99) that coefficient of friction always
decreases for increased velocity of strip so it might be expected that
the excess pressure due to work against friction in the rolling process,
may be less at higher speeds.

**POWDER MECHANICS**

Free-flowing powder is unique in that it exhibits the property
of dilatancy (113) and its behaviour must be described by the complex
three-dimensional Hvorslev Surface (114). However, a powder under
load soon ceases to be free-flowing and begins to behave as a variable-
density solid.

It is most interesting to note that careful selection of particle
size distribution can give very low loose powder voidages (108). This may be of use in the rolling system where air expulsion is a problem.

MECHANICS OF POWDER COMPACTS

Train (118) has pointed out that there seem to be three distinct regions of powder behaviour. The first is the region in which particles repack to their lowest density without particle deformation. This is probably the stress range in which the Hvorslev treatment applies. The next stage involves deformation at particle contacts and is covered by the stress-strain relationship in Balshin (121). The final region occurs where the voidage is so low that the compact behaves as a solid. In this region Long (119) has observed that the yield strength is virtually constant. Roll compaction must take a powder through all these stages just as die compaction would do. Unckel (115) however, has shown that wall friction in a die is a very important, and difficult to measure, parameter. It causes a complex stress (density) distribution within the die.

It is well known that when a solid is plastically deformed it begins to behave like a viscous liquid in that its yield strength increases with strain rate. Lund and Armstrong (12) showed this for compacted zinc powder and also pointed out that the oxide coating on the powder seems to increase the yield strength over that for pure zinc. Whitman and Healy (125) showed that rapid loading of loose powders produces a similar increase in yield strength, possibly because of the lateral inertia of the particles.

CONCLUSIONS

The major problem with the coating process seems to be the speed
limitation due to expulsion of air from the compacting layer. There are several possible ways of dealing with this.

1. Operate in a vacuum or in an atmosphere of low viscosity gas. (The viscosity of a gas is lower at lower temperatures).

2. Use the largest particles possible because larger particles are fluidised less easily. It should be remembered that the particles used should not be larger than the final coating thickness or they will not give a continuous coating.

3. Bond the particles together before rolling, either by pre-sintering or by means of a chemical binder.

4. Use multipass rolling so that not all the air is expelled at any one pass.

The first possibility does not seem very sensible. For vacuum operation the whole plant would have to be sealed. Keeping the rolls at a very low temperature seems a little easier but this might lead to other problems and would certainly be excessively expensive.

Using larger particles seems a reasonable idea, especially if used in conjunction with one of the other techniques. However, the extra work involved in selecting or manufacturing, particles of a specific size, might raise powder costs to an unacceptable level.

Chemical bonding of the powder seems to be the most generally used technique at the moment and a tremendous amount of research has gone into it. However, the existence of a volatile binder in the compact means that there is danger of blistering during the sintering stage. Mori and Inoue (15) have suggested multipass design to overcome this problem. Presintering may be expensive or impossible.

Multipass design looks, on the surface, to be the best solution
to the problem. The compact only has to be deformed until the particles bond together and then it can be completed with one more pass. In practise however, it has been found (13) that if the roll pressure is not high enough the powder will stick to the rolls and be separated from the substrate. Canning, Davies and Gibbon (9) suggest that this effect can be minimised by having a high surface finish on the roll but even this may not be good enough.

It therefore seems that for very high speed rolling it would be best to operate at low temperatures, use the largest particles possible, chemically bond the powder (or presinter if this is metallurgically possible), and roll in several passes. Roll pressure could be saved by using the smallest rolls that would not bend along their axes and so cause a density distribution across the strip. With all this sophistication it will still be necessary to deform the substrate sufficiently so that the compact bonds to it, and to produce a compact which is dense enough to give corrosion protection but not so dense that blistering takes place during sintering.

If such a system is to be designed it is obvious that much more information must be obtained about the effect of roll size, roughness, speed and gap upon the deformation of substrate and coating. The intention of this thesis is to propose a technique by which this information can be obtained.
CHAPTER 2

Experimental Investigation
FIGURE 13  Size Analysis of Aluminium Powder Samples
FIGURE 14  Stereoscan electron microscope pictures of the aluminium powder
Experimental Results

Curve given by:

\[ \ln(\text{Strain}) = 5.3 \ln(\text{Stress}) - 40.44 \]

**FIGURE 15** Tensile strength of mild steel substrate
FIGURE 16 Determination of the coefficient of friction between an aluminium compact (s = 93°) and the unrolled steel substrate.

Gradient = 0.58
FIGURE 17 U.K.A.E.A. Split Die Compaction Equipment

TRANSVERSE LOAD

Axial Load

Powder Compact

Cross-section of one half of the split die
THE ALUMINIUM POWDER

Alcan aluminium powder was used from a commercial sample, superficially graded as - 36 mesh. Chemically it consists of 99.5% min. purity aluminium with principal trace elements as below.

<table>
<thead>
<tr>
<th>Element</th>
<th>Max. %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iron</td>
<td>0.3%</td>
</tr>
<tr>
<td>Silicon</td>
<td>0.25%</td>
</tr>
<tr>
<td>Copper</td>
<td>0.03%</td>
</tr>
</tbody>
</table>

Because it is an atomized powder it has a coating of aluminium oxide making up about 1% of the total weight.

The powder was sized on a Wide Angle Photosedimentometer manufactured by Microscral Ltd. which gives dimensions as Stoke's Diameters. (i.e. The diameter of a sphere that would settle at the same rate as the particle being examined.) The result of this analysis is shown in Figure 13 and it can be seen that the range of sizes is very wide and has its median (50%) value at 32 microns. There is very little powder below about 10 microns, and apparently very little above 110 microns. A sieve analysis was carried out however, and this showed that there were a few very large particles in the powder which had not been caught by the sedimentation technique.

The Alpine Multiplex Laboratory Zigzag Classifier 100 M.Z.R. was used to cut three size fractions from the Alcan powder and these were sized on the Microscral Photosedimentometer as shown in Figure 13. The median (50%) values of these three fractions are 18 microns, 34 microns and 54 microns.

This powder is manufactured by atomization and its irregular shape can be seen in Figure 14. The Stereoscan electron microscope
FIGURE 18 Comparative split-die results for -36 mesh powder at different compact heights

TRANSVERSE STRESS (Kg/sq.cm.)

Initial Compact Height
- 4.2 cm.
- 3.0 cm.

\[ \sigma_t = 0.56 \sigma_a - 196 \]

\[ \sigma_t = 0.215 \sigma_a \]
FIGURE 19
Comparative split die stress results for three aluminium powder fractions
FIGURE 20

Comparative stress-strain results for the split-die equipment

Experimental Results

1. $S = 3.171 - 0.298 \ln\left(\frac{\sigma}{\sigma_0}\right)$
2. $S = 2.479 - 0.198 \ln\left(\frac{\sigma}{\sigma_0}\right)$
3. $S = 1.306 - 0.039 \ln\left(\frac{\sigma}{\sigma_0}\right)$

- 36 mesh
- 18 micron
- 34 micron
- 54 micron
shows in detail the strange formation of the particle surface.

THE STEEL SUBSTRATE

0.035 inch thick mild steel was chosen as the substrate and test pieces were cut for estimation of its yield strength and strain hardening properties. Each test piece was 18 cms. long and had a waisted section 0.9 cms. wide. Stress characteristics were measured using a Hounsfield Tensometer with a Hounsfield Extensometer giving strain values based on a 2 inch gauge length. The results are shown in Figure 15 and it can be seen that they are fairly typical for a mild steel. There was found to be negligible variation between test pieces.

COEFFICIENT OF FRICTION

The coefficient of friction between the steel strip and an aluminium powder compact was measured on the platform of a Jenike Flow Factory Tester. An aluminium compact of theoretical density 96% was vertically loaded onto a sample of the steel strip and the horizontal force required to cause slip at various normal loads was recorded. Results showed (Figure 16) that the coefficient of friction in the load range measured is fairly constant at about 0.6.

YIELD STRENGTH OF POWDER COMPACTS

To obtain as much information as possible about the strength characteristics of aluminium powder compacts, it was decided to make use of the split-die compaction apparatus used by W.M. Long (reference 120) at U.K.A.E.A. This equipment consists of a 1.5 inch diameter cylindrical die which is split down the middle (Figure 17). The two halves of the die are held together by horizontal rams, but at any axial load the horizontal pressure can be reduced until the die
**Aluminium Fractions**

- **1** $-100 + 75$ micron
- **2** $-75 + 53$ micron
- **3** $-53 + 45$ micron
- **4** $-36$ mesh

**Copper Samples**

- **5** irregular
- **6** spherical

**FIGURE 21** Crushing strength of powder compacts
halves just begin to separate. This is indicated by the breaking of electrical contacts at the four corners of the die halves. Transverse and axial loads are measured with proving rings between the die and the system of hydraulic rams which surrounds it. The bottom punch is static but the sections which make up the die walls are suspended from the main framework with pulleys and counterbalance weights so that the compact is effectively strained from both ends.

Results are obtained by increasing the transverse stress to some arbitrary value to hold the split-die together, filling the die with powder, increasing the axial load to some known value and then reducing the transverse stress until one or two of the contacts are broken at the corners of the die halves. (This is indicated by a light being extinguished.) The transverse load and strain are then recorded for that value of axial load. Transverse pressure is then increased to clamp the die together again and axial pressure is increased by another increment for another stress measurement. The final compact is weighed for calculation of density. Figure 18 shows results for the -36 mesh Alcan powder at different compact heights. Comparative results for the three powder fractions are shown in Figure 19 and stress-strain results in Figure 20.

**CRUSHING STRENGTH OF POWDER COMPACTS**

Another advantage of the split die compaction equipment is that the two halves can be separated at any stage to leave a compact of known density. With conventional apparatus it is often found that the compact will crack as it is being pushed out of the die.

Compacts prepared as above were crushed to discover the effect of particle size on crushing strength. (This is effectively the yield strength of a compact at zero radial stress.) So that a wider
distribution of sizes could be tested, these fractions were prepared by sieving.

To examine the effect of particle shape on crushing strength, a spherical and an irregular copper powder were compared. (No spherical aluminium powder could be obtained.) The copper particles were roughly the same size (Figure 21).
FIGURE 22  Sectioned elevation and plan view of the equipment used to form a loose-powder coating.
2.02 Formation and Examination of Loose Powder Coating

INTRODUCTION

The previous section dealt with the examination of aluminium powder, and an investigation of the strength of powder compacts and of mild steel strip. The next problem was to coat a specimen of the mild steel strip with the powder and then roll the product. This section deals with the problem of forming an even coating of loose, dry powder on the steel substrate.

TECHNIQUE

It can be seen from the literature that there are many ways in which an even coating of powder can be achieved. To reduce the number of variables it was decided that the coating should be completely free from chemical additives, so all slurry techniques were discounted. Without binders it is only possible to coat the strip on one side.

Initially, an attempt was made to sprinkle powder from a vibrating table, but it was found to be very difficult to get an even coating across the width of the 3 inch strips. Sprinkling from a sieve would have been messy and difficult because of the wide size distribution of the powder. Dry electrophoresis was the technique finally adopted because it was thought that this would have the added advantage of breaking up agglomerates.

PRE-TREATMENT OF STEEL

Mild steel, with a thickness of approximately 0.035 inches, was cut into strips 10 inches long and 3 inches wide. These were cleaned in 5% nitric acid to remove rust and then degreased with a solution
FIGURE 23
Experimental arrangement for determining the thickness of a dry powder coating.
of Quadralene in a 50% water-isopropanol mixture. The samples were washed with distilled water and left wet so that the powder would stick initially rather than bouncing off.

**DRY ELECTROPHORETIC DEPOSITION**

The loose powder coating was formed by means of a vibrating conveyor with a charged plate set into it. This can be seen in Figure 22, where the hopper and the entire conveying length is constructed of an insulating material but has a 6 inch long steel plate set into it. The sample of steel strip was mounted in a wooden frame for insulation and drawn across the conveyor above the charged plate in a direction perpendicular to the conveying direction. It was moved at a constant velocity by means of an electric motor which was heavily geared down with a worm and thread mechanism. Coating thickness could be controlled by varying the speed of the motor. The mild steel sample had previously been wetted as described above and was earthed. It was found that an adequate coating could be formed with a plate-plate gap of 1 cm. and a steady charge of 4,000 volts. The coated strip was dried in an oven for 24 hours at 80°C.

**EXAMINATION OF DRY COATING**

After drying, a 0.5 cm. wide strip around the perimeter of each sample was cleared of powder with a piece of fine glass tubing attached to a conventional vacuum cleaner. The sample was then clamped into a metal jig so that the powder thickness could be measured. This was done by lowering a pointer onto the surface of the powder, noting the displacement on a strain gauge, then lowering the pointer through the powder until it touched the steel and recording the difference in the two strain gauge readings. This was done at several points
FIGURE 24  Roll gap profiles

a  At rest
b  With steel strip in nip
c  After removal of strip

(Traced directly from photographs)
over the surface of the sample. Figure 23 shows the experimental arrangement and how a powerful light source was used to indicate when the pointer touched the powder surface. By looking at the pointer with the light source behind, the powder and the shadow it threw onto the powder surface looked very dark. As the pointer was lowered it would get closer to the shadow until pointer and shadow could be seen to touch. This occurred at the powder surface. The pointer was lowered with a spring attachment so that it just stopped at the steel surface and did not indent it.

Reproducability tests showed an accuracy of ± 10 microns by this method.
Figure 25 Showing how the sample strips were cut after being rolled from both ends towards the middle.

--- Path of cut

a Strip cut at five places for thickness determination
b Strip cut into five squares for powder wt./unit area determination.
FIGURE 26 Cross-sections of rolled strip as seen under the microscope
2.03 Formation and Examination of Rolled Strip.

THE ROLLS

A two-high mill with 6 inch diameter rolls was used to compact the powder-coated strip. The (C.L.A.) roughness of the roll surface was 15 micro inches and the roll speed was constant at 7 r.p.m.

Figure 24 shows the roll gap at rest and with a piece of steel strip between the rolls. It can be seen that strain in the mechanism is considerable and that any gap measurement would have to be taken with the strip in place and the rolls turning. This would present considerable difficulties and would still not compensate for roll flattening or bending. Tundermann (26) observed that the recovery of a rolled iron compact could be as great as 8%, but the compact thickness on the coated strip is only about 1/30th. of the total so the recovery would only be 0.26% for the coated strip. Elastic recovery of steel (Figure 15) is only 0.1% so it is reasonable to assume that the final thickness of coated strip is equal to the roll gap. (Error less than 0.4%).

The rolls were fitted with pressure gauges but they were not very accurate. Also, it will be remembered, powder had been cleared from around the perimeter of the strip so that it could be clamped into the jig for powder-thickness measurement. Therefore, any pressure recorded on the gauges would be the total pressure for compaction of both coated and uncoated strip. For this reason no attempt was made to improve the pressure recording facility.

ROLLING THE COATED STRIP

Before each rolling operation the rolls were cleaned with a clean piece of dry cloth. It was found that some powder always stuck
to the rolls, especially at the edges of the coated region where densification was reduced by sideways movement of the powder. Each coated sample was rolled from one end to the middle and the rolls were then stopped. Pressure was released and the strip was removed. The rolls were then set at a new gap and the sample was rolled from the other end.

EXAMINATION OF COMPACTED STRIP

After preparation of the rolled samples as above, a strip about 1.5cms. wide was cut from the samples at the point where the rolls had been stopped (Figure 25). These strips were set in araldite and then cut at five places along their length with a hacksaw. (It was found to be necessary to evacuate the strips before setting in araldite so that all the pores would be filled. When this was not done it was sometimes found that the coating would separate from the substrate.) One face of each segment was polished and examined under the microscope so that the initial and final thickness of the steel could be measured together with the thickness of the compacted powder layer. Figure 26 shows cross-sections of the rolled strip as seen under the microscope.

To calculate the density of the coating it was necessary to measure the weight of the aluminium compact per unit area. Radiation techniques were examined but found to be unusable in a system where the lowest density material formed the thinnest layer. X-ray Fluorescence would have been a possible technique but it was very complex and time consuming.

Therefore an attempt was made to measure the weight of aluminium directly by dissolving the coating in 10% Caustic soda solution. A strip 1 cm. wide was cut directly behind the strip for thickness
measurement (Figure 25) and this was cut into five 1 cm. square segments. It was found that a circular diamond saw was the best tool to use to prevent the coating peeling off. Each segment was then measured for area of coating on a piece of transparent graph paper, weighed, dissolved in 10% Caustic soda, and weighed again. Uncoated mild steel was found to be completely unaffected by exposure to the alkaline solution.

Reproducability of this technique was tested by analysing several squares from a sample of coated strip and it was found that the difference between any two adjacent squares along the length of the sample was never more than 5%. However, width-wise the difference was much greater. This is probably due to the method used for powder coating and shows that the dry electrophoresis technique gives a fairly even powder distribution along the sample length but is not as good over the sample width. This result is quite satisfactory for the technique finally adopted. The complete set of results from these experiments is to be found in Table 2.

It can be seen that for several of the sample strips, results are only quoted for one end. This is because it was found to be difficult to stop the rolls at the desired place and sometimes the compacted region was taken too close to one of the ends. In these cases a microscopic examination of the strip cross-section showed the coating thickness to be decreasing towards the end. The technique for determining the powder weight per unit area depends upon an even powder coating so these samples were invalidated.

The initial powder weight per unit area was calculated from the measured final weight by a mass balance. Thus the total weight of aluminium on the strip could not change so the reduction in weight per unit area is proportional to the increase in area.
CHAPTER 3

Theory.
FIGURE 27  Diagrams showing the dimensions and forces on a bimetallic strip in a roll nip.
3.01 Determination of Relative Strains

MASS BALANCE

The conclusion reached from the literature survey was that a relationship between powder compaction and strain in the substrate would be useful. This problem may first be approached via a mass balance over the rolling stage.

Rate in - Rate out = Rate of accumulation.

It is to be hoped that there is no accumulation of material in the nip. Thus:

\[ \text{Rate in} \approx \text{Rate out} \]

For the steel this takes the form:

\[ D_s \cdot V_{s1} \cdot y_{s1} = D_s \cdot V_{s2} \cdot y_{s2} \quad \text{for unit width} \] (1)

For the powder:

\[ D_{c1} \cdot V_{c1} \cdot y_{c1} = D_{c2} \cdot V_{c2} \cdot y_{c2} \quad \text{for unit width} \] (2)

It is to be expected that powder and substrate will travel at the same speed outside of the compaction zone.

\[ V_{c1} = V_{s1} = V_1 \] (3)

\[ V_{c2} = V_{s2} = V_2 \] (4)

Therefore

\[ \frac{y_{s2}}{y_{s1}} = \frac{y_{c2}}{y_{c1}} \cdot \frac{D_{c2}}{D_{c1}} \] (5)

It can be seen from the above equation that there is no direct relationship between compaction of powder and deformation of substrate unless a separate correlation is found for determination of compact density. Now the density of a compact is related to its yield strength, and the pressure distribution in a roll nip is a function of material yield strength and roll friction forces. The distribution of pressure in the nip must therefore be determined.
PRESSURE DISTRIBUTION

The steel strip being used in this work is 0.035 inches thick and 3 inches wide. It therefore falls within the conditions stipulated by Hill (43) for application of the von Karman analysis (Page 18). With a two-material system the technique must be modified as below.

A two-component strip undergoing roll compaction is shown in Figure 27. Resolving these forces horizontally produces the exact equation below:

\[- d(q_e \cdot y_e) - d(q_s \cdot y_s) + P \cdot dy + 2 \mu P \cdot dx = 0\]  

(6)

It will be remembered that the friction term is negative for the entry side of the neutral place and positive for the exit side.

The above equation contains seven unknowns but by definition:

\[y = y_s + y_C\]  

(7)

Also the profile of the roll itself provides a boundary condition which, for thin strips can be approximated:

\[y = h + \frac{x^2}{R'}\]  

(8)

Where \(R'\) is the radius of the flattened roll as calculated from Hitchcock's formula (Page 20). A complete solution of equation 6 however, requires a knowledge of the deformation characteristics of the materials involved.

PLASTIC DEFORMATION OF STEEL

Previous work has shown (43) that the von Mises criterion of plastic yielding is generally the most accurate whilst the Tresca criterion is easier to apply.

According to von Mises, yielding occurs in a continuum when the following condition is obeyed.

\[(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2 = 2k^2\]  

(9)
For thin strip Hill has shown that there is virtually no lateral strain in the strip so the rolling process is a plane strain situation. Now the Tresca yield criterion (Page 17) takes no account of stress in the third direction ($\sigma_3$) but with the von Mises criterion it must be given a value. This value is usually assumed to be the mean pressure on the element.

$$\sigma_3 = \frac{(\sigma_1 + \sigma_2 + \sigma_3)}{3} = H$$

(10)

For plane strain:

$$\sigma_3 = H$$

(11)

$$\sigma_3 = \frac{(\sigma_1 + \sigma_2)}{2}$$

(12)

According to Hill this assumption is only strictly true when Poisson's Ratio is 0.5. This in fact, must always be the case for plastic deformation with no volume change.

Substitution of equation 12 into equation 9 gives:

$$\sigma_1 - \sigma_2 = 1.155.k$$

(13)

For the tensile tests on the steel:

$$\sigma_1 = T_s$$

(14)

$$\sigma_2 = 0$$

(15)

$$\sigma_3 = 0$$

(16)

This is not a plane strain situation so the results must be substituted into equation 9.

Then

$$2.(T_s)^2 = 2.k^2$$

$$k = T_s$$

(17)

Substitution into equation 13 gives:

$$\sigma_1 - \sigma_2 = 1.155. T_s$$

(18)

Now the assumption of von Karman is that the forces $P$ and $q_s$ on the element of rolled strip are the principal stresses $\sigma_1$ and $\sigma_2$ in the plastically deforming continuum.
\begin{align*}
\sigma_1 &= P \\
\sigma_2 &= q_s \\
P - q_s &= 1.155.T_s
\end{align*}

This relationship can now be used to eliminate \(q_s\) from equation 6.

From equation 17:

\[-q_s = 1.155. T_s - P\]

\[-d(q_s,y_s) = 1.155. T_s \cdot dy_s - P \cdot dy_s + 1.155. y_s \cdot dT_s - y_s \cdot dP\]

Remembering that \(T_s\) is a function of \(y_s\) due to work hardening (Fig. 15)

PLASTIC DEFORMATION OF COMPACT

For the compact the yield criterion must be modified to allow for the variation of yield strength with hydrostatic pressure.

\[(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2 = 2 \left[ f_N (H) \right]^2\]  \hspace{1cm} (23)

For the die compaction tests Long (120) calls the horizontal stress on the die halves 'radial' stress. However, this is not strictly true because strain in the die is limited to just two directions.

The split die therefore represents a plane-strain situation and the measured transverse stress is simply \(\sigma_2\).

From equation 13, for plane strain.

\[\sigma_1 - \sigma_2 = 1.155.k\]  \hspace{1cm} (13)

or in this case

\[\sigma_1 - \sigma_2 = 1.55. f_N (H)\]  \hspace{1cm} (24)

It can be seen that the stress relationship in the die is identical to the stress relationship in the roll nip.

Therefore:

\[\sigma_1 = P = \sigma_a\]  \hspace{1cm} (25)

and

\[\sigma_2 = q_c = \sigma_t\]  \hspace{1cm} (26)

By experiment this relationship falls into three linear regions of
general form:

\[ \sigma_t = K_1 \sigma_a + K_2 \] (27)

Therefore

\[ q_c = K_1 \cdot P + K_2 \] (28)

where \( K_1 \) and \( K_2 \) change depending on the pressure range.

Equation 28 can be used to eliminate \( q_c \) from equation 6.

**DENSIFICATION OF THE COMPACT**

Equations 7, 21 and 28 are used to eliminate variables from equation 6. This leaves just three variables and means that one more equation is needed for a solution. This is given by the mass balance. If equation 5 is continuous (which is only true if there is no slip between compact and substrate at any point in the nip) then it can be differentiated to give:

\[ \frac{dy_s}{y_s} = \frac{dy_c}{y_c} + \frac{dD_c}{D_c} \] (29)

\( D_c \) can be divided by the density of pure aluminium \( (D_{co}) \) to give a dimensionless term \( (s) \) which is the volume of solids in the compact divided by the total volume of compact.

\[ s = \frac{D_c}{D_{co}} \times 100\% \] (30)

It can also be seen that:

\[ S = 100 - E' \] (31)

where \( E' \) is the voidage of the compact expressed as a percentage. \( s \) is therefore called the 'Solidity' or the 'Theoretical Density'.

Then the mass balance becomes:

\[ \frac{dy_s}{y_s} = \frac{dy_c}{y_c} + \frac{ds}{s} \] (32)

The unfortunate thing about this equation is that it introduces a new variable, \( s \). However, the die compaction results in Figure show a relationship between \( s \) and the axial stress \( \sigma_a \).
Using the relationship proposed by Balshin (121) it was found that the equation below could be used to relate \( s \) and \( O \).

\[
\frac{100}{s} = K_3 \ln (\sigma_a) + K_4 \quad (33)
\]

where \( K_3 \) and \( K_4 \) are constants which change in three distinct regions (Fig. 20). From equation 25 this becomes:

\[
\frac{100}{s} = K_3 \ln (P) + K_4 \quad (34)
\]

Equation 6 can now be solved by a numerical calculation using a computer.

**DEFORMATION OF POWDER LAYER ONLY**

As the yield strength of the powder is very much less than that of the steel, there must be an initial stage where only powder is being deformed. Now, one of the requirements of von Karman's analysis is that plastic deformation must be continuously taking place in all part of the nip or the internal stress term in equation 6 cannot be evaluated. However, if this equation is rearranged the stress in the steel can be calculated from the plastic behaviour of the deforming powder.

\[
d(q_s, y_s) = -2\mu P dx + P dy - d(q_c, y_c) \quad (35)
\]

It can be seen from Figure 15 that the elastic deformation of the steel is very small (0.1%) and will be insignificant even in comparison with the powder thickness. Therefore:

\[
dy_s = 0
\]

\[
d(q_s, y_s) = y_s dq_s
\]

and

\[
dY = dy_c
\]

From equation 35

\[
y_s dq_s = 2\mu P dx - P dy_c - d(q_c, y_c) \quad (36)
\]

This can be solved for \( q_s \) which can then be substituted into equation
CHECKING FOR SLIP

On page 57 it was pointed out that the theory can only be valid when there is no slip between powder compact layer and steel substrate. Experimental results (Fig. 16) have shown that the coefficient of friction between an aluminium compact and the steel strip is 0.58 before bonding occurs (when it would obviously be greater). Thus the shearing force at the aluminium-steel interface must always be less than 0.58 times the normal force at the interface for the theory to be valid. The shearing force on any incremental length of interface can be given by:

$$dF = -d (y_c q_c) + \frac{1}{2} P\, dy - \mu P\, dx$$

The normal force on the interface cannot be exactly determined but it will be something less than $P\, dx$. It is then possible to say that:

if $dF > 0.58 P\, dx$

slip WILL occur.

However, it cannot be guaranteed that slip will not occur if $dF$ is less than this.
INTRODUCTION

The pressure profile analysis in the last section is greatly dependent on the compaction results obtained with the split-die apparatus. It has been pointed out in the Literature Survey however, that die compaction is not as straight-forward as it looks. Unckel (re.115) for example, points out the effect of die wall friction on the pressure transmission through the die whilst Train (118) et. al. have shown that compact density is seldom the same at all points within the die. To estimate the extent of these effects a theory is suggested by which the magnitude of the wall friction could be estimated.

ESTIMATION OF DIE WALL FRICTION

With the split-die apparatus, compression takes place from both ends so that the friction force at the wall effectively increases the recorded axial pressure. It must be assumed that friction forces at the punch faces do not have a significant effect upon the magnitude of the mean radial pressure (Fig. 17). The recorded axial pressure is then given by the equation below.

\[ \sigma_a = \sigma_a' + \frac{4L}{D} f' \sigma_r \]  (37)

This shows that the magnitude of recorded axial pressure is a function of the compact height so that for runs at heights \( L_1 \) and \( L_2 \) there are two equations:

\[ \sigma_{a1} = \sigma_{a1}' + \frac{4L_1}{D} f' \sigma_{r1} \]  (38)

and

\[ \sigma_{a2} = \sigma_{a2}' + \frac{4L_2}{D} f' \sigma_{r2} \]  (39)

Now it can be assumed that the effective axial pressure is a unique
function of radial pressure so that if:

\[ \sigma_{r1} = \sigma_{r2} = \sigma_r \]

then

\[ \sigma_{a1}' = \sigma_{a2}' = \sigma_a' \]

Substitution into equations 38 and 39 and subtraction of the two resulting equations gives:

\[ (\sigma_{a1} - \sigma_{a2}) = \frac{4.f'.\sigma_r}{D} (L_1 - L_2) \]  \hspace{1cm} (40)

So by doing runs at different initial heights it should be possible to estimate \( f' \) and hence the effective axial pressure, which is the true value that should be used in the pressure profile analysis.
CHAPTER 4

Results
VALIDITY OF TENSILE STRAIN TESTS ON STEEL

It will be noted that the steel strength and work-hardening characteristics are established by a tensile test but the results are being used for a compression problem. According to Hill (Ref. 43) this is quite acceptable so long as the tensile strain is defined as:

\[
\frac{l - l_0}{l}
\]

rather than in the conventional way:

\[
\frac{1}{l_0} - 1
\]

This must then be compared with the conventional compressive strain:

\[
1 - \frac{y_s}{y_{sl}}
\]

VALIDITY OF SPLIT-DIE RESULTS

The published work on die compaction as discussed on page 32 points out that, in normal compaction, a distribution of densities always exists in the die. The validity of the split-die results, which are of primary importance to this thesis, must therefore be questioned.

Results from a paper by Kamm, Steinberg and Wulff (Ref.116) show that 45 micron iron powder in a cylindrical die of diameter 0.56 inches can give a variation of 76 - 98% in a compact of average solidity 91.4%. This is for an initial bed depth of 0.55 inches in a simple-acting die. For a double-acting die the same results would be achieved for an initial bed depth of 1.1 inches. The workers point out that this density distribution would be drastically reduced by a decrease in the initial bed depth or by an increase in the die diameter.
for the same length/diameter ratio.

The results used in this thesis were obtained from a double-acting, split die of diameter 1.5 inches and initial bed depth about 1.0 inches. This is a length/diameter ratio of 0.67 whereas the Kamm, Steinberg, Wulff results were for a ratio of 1.97. Thus both the die diameter and length/diameter ratio tend to reduce the density variation.

Another important factor emerging from the literature survey (page 33) is the effect of die wall friction on the density variation within a compact. The theory postulated in the previous section (page 60) shows how the magnitude of the friction coefficient may be calculated from compaction tests in the split-die equipment at two different initial bed depths. Figure 18 shows experimental results for two such compacts and it can be seen that there is no consistent distinction between the results. It therefore seems that the mechanism of the split die (in which the transverse pressure is reduced after each increment of strain) operates so as to effectively eliminate all wall friction.

EFFECT OF PARTICLE SIZE

Figure 19 compares the stress results for the aluminium powder fractions with those for the -36 mesh powder (Fig.18). It can be seen that, in the low stress region, there is a distinct tendency for the finer powder to give a lower transverse stress for the same axial stress. In the intermediate region there is a wider scattering of results but the trend seems to be maintained.

For the stress-strain results (Fig.20) there is a very marked distinction between powder fractions, with the larger powder giving a greater density for the same pressure. (Density of solid aluminium
is taken as 2.7 grms./cc.). It is interesting to note that, although the intermediate size of the -36 mesh powder lies between that of the largest and smallest size fractions, the density is always greater than either. This could be due to a particle packing effect as discussed on page 31.

THE CRUSHING STRENGTH TESTS

Figure 21 shows quite clearly that a larger particle will give a stronger compact for the same pressure. This effect is to be expected because, for larger particles, the number of particles per unit area of surface will be less and therefore each must carry a greater load. This means that the area of contact between particles must be greater and so must the bond strength.

This result is not central to the argument of this thesis but it is relevant to the general consideration of powder roll-compaction.
FIGURE 28  Theoretical Pressure Profiles for variation of powder coating thickness and roll gap

Friction coefficient constant at 0.1
Roll radius constant at 7.62 cms.

1. Roll Gap = 0.09 cms.
   Powder coating = 0.04 grms./sq. cm.

2. Roll Gap = 0.09 cms.
   Powder coating = 0.03 grms./sq. cm.

3. Roll Gap = 0.09 cms.
   Powder coating = 0.02 grms./sq. cm.

4. Roll Gap = 0.94 cms.
   Powder coating = 0.02 grms./sq. cm.

5. Roll Gap = 0.98 cms.
   Powder coating = 0.02 grms./sq. cm.
FIGURE 29 Theoretical Pressure Profiles for variation of roll radius and surface friction

Powder coating constant at 0.02 grms/sq.cm.
Roll gap constant at 0.09 cms.

1. Roll Radius = 22.86 cms
   Friction coefficient = 0.1
2. Roll Radius = 15.24 cms
   Friction coefficient = 0.1
3. Roll Radius = 7.62 cms
   Friction coefficient = 0.15
4. Roll Radius = 7.62 cms
   Friction coefficient = 0.1
5. Roll Radius = 7.62 cms
   Friction coefficient = 0.5
The equations presented in the theoretical section (page 52) are solved in a numerical manner by computer. It was found that increments of \( dx \) of 0.0005 cms. were best for optimisation of time and accuracy. In the initial stages of the compaction, before the steel begins to deform, the pressure is calculated directly from equations 32 and 34 whilst equation 36 is used to determine the point at which the steel begins to deform.

During the initial stages of steel deformation a value of \( P \) has to be selected and substituted into equation 34. The calculated value of \( s \) is then used to calculate \( y_e \) and \( y_s \) which are used to find a value of \( P \) from equation 6. The two values of \( P \) are compared and a new value of \( P \) selected if they do not agree. \( P \) is increased in increments of 0.5 so the maximum pressure theoretically predicted could be up to 3% greater than the true pressure. Once \( P \) has been calculated the program moves on to a new value of \( x \).

The final stage of the deformation is quite straightforward because the pressure has reached a maximum and the density of the coating remains constant. Once the program has got more than half-way to the Line of Roll Centres (the maximum pressure never occurs before the half-way stage) various endings are calculated until one is found in which \( P \) falls to the yield strength of the steel at \( x = 0 \). During this final stage the friction force is in the opposite direction.

After the calculation of one complete pressure profile the average pressure is calculated and substituted into Hitchcock's equation (page 20) for the calculation of the lattened roll radius. A new
### Table 2: Experimental Results

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## Experimental Results Continued - 2

### INITIAL CONDITIONS

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**Powder used:**
- Sheet 1 - 36 mesh
- Sheet 2 - 36 mesh
- Sheet 3 - 36 mesh
- Sheet 4 - 18 micron
- Sheet 5 - 35 micron
- Sheet 6 - 55 micron

**Initial steel thickness 890 microns for all samples.**

**L2** means extreme left hand side.

**R2** means extreme right hand side. (See Figure 25)
FINAL COATING THICKNESS (microns)

Locus of Initial Coating Thickness (45% solidity)

Roll Gap (microns)
1. 980
2. 940
3. 900
4. 820

Effect of Air Entrainment at 5.5 cm./sec.

INITIAL COATING WEIGHT (grms./sq.cm.)

FIGURE 30 Presentation of Theoretical Results for Comparison with the Experimental Results shown in Table 2
profile is then calculated using this corrected radius.

**RESULTS**

The calculated shape of the pressure profile is shown for various conditions in Figures 28 and 29. Figure 28 shows how the maximum pressure is increased by increasing the amount of powder coating but decreased by increasing the roll gap. Figure 29 shows that larger rolls and a larger coefficient of surface friction between roll and specimen both tend to increase the maximum pressure. Since the coating density is a function of pressure only, (equation 34) it can be seen that conditions which give an increased maximum pressure will give a denser coating for the same reduction of steel. The effect of roll flattening is also shown for high pressure situations.

It will be noted that the theoretical pressure profiles are not rounded near the peak pressure as is the experimental example shown in Figure 4. This is because no allowance is made for the sticking region that must occur at this point (Figure 6). It might be thought that this is an important error because the final compact density is a function of the maximum roll pressure. However, the theory showed that the maximum compact density of 99% theoretical was generally reached well before the peak pressure so any further pressure increase makes no difference. Thus although example 5 in Figure 28 has a final solidity of 82%, all other examples in both Figures 28 and 29 have final values of 99%.
Comparison of Experimental and Theoretical Results

PRESENTATION OF THEORETICAL RESULTS

In order to compare the theoretical technique with the experimental results of Table 2 the theoretically predicted final coating thickness was plotted against initial powder coating for various roll gaps (Fig. 30).

The experimental evidence of previous workers (page 21) indicates that the roll surface friction should be about 0.08 for the steel and 0.095 for the aluminium. Now equation 6 is formulated by a horizontal force balance so it is acceptable to use an average value for the friction coefficient. 0.09 was used but it was found that the same results were obtained for a coefficient of 0.085.

Although the experimental results shown in Table 2 indicate a wide variation in the initial powder coating density, theoretical manipulation of this variable had no effect on the results. The normal bulk solidity of the aluminium powder was 45% so this was the value finally adopted. This initial condition gives a linear relationship between powder weight per unit area and coating thickness as is shown in Figure 30.

At any point on the graph (Fig. 30) the final steel thickness can be calculated as the difference between the final coating thickness and the roll gap.

i.e.) \[ y_{s2} = h - y_{c2} \] (41)

If the compaction of the coating is not sufficiently great for the steel to deform, then:

\[ y_{s2} = y_{s1} = 0.089 \]

and \[ y_{c2} = h - 0.089 \] (42)
FIGURE 31  Cross-section of a coated sample at point where rolls were stopped
Theoretical Relationship between Total Roll Force and Initial Powder Coating Thickness

Initial Coating: -
- 0.015 grms/sq.cm.
- 0.025 grms/sq.cm.
- 0.035 grms/sq.cm.
Whis is a straight horizontal line for a given roll gap as shown in Figure 30.

**COMPARISON WITH EXPERIMENTAL RESULTS**

The technique of measuring steel reduction and final coating thickness has an accuracy of ± 5 microns while the determination of coating weight per unit area has a reproducability of 5%. Within these accuracies the agreement between experimental and theoretical results is as shown in Table 2. Each size fraction was compared with the theoretical result for its own size although the difference between results for the various fractions was virtually negligible.

A careful study of these results reveals that, apart from samples 6BR2, 5AR2 and 6AR2, all results where the final roll gap is greater than or equal to 834 microns agree with the theory and all those where the gap is less than or equal to 833 microns do not. Consideration of the reduction in thickness of steel does not give such a clear separation of results.

**POSSIBLE REASONS FOR DISCREPANCIES**

The result for sample 6BR2 does not agree with the theory although it is for a roll gap of 912 microns. Inspection of Table 2 however shows that this sample has a much smaller coating of powder than the adjacent sample 6BR1. With such a difference it is possible that the adjacent results interfere with one another and the steel substrate to 6BR2 is deformed by shearing action from sample 6BR1 and the outer edge of the strip, rather than by pressure transmitted through the powder coating.

The results for samples 6AR2 and 5AR2 are not very far from the supposed threshold of 834 microns and therefore may be explainable
when the reason for the threshold is understood.

The threshold between those results which fit the theory and those that do not is not as distinct as it may look, because results for large gaps generally tend to fit the theory better than those for gaps just over 833 microns. In other words there is a gradual trend towards lower compact densities than predicted as the roll gap decreases. This may be due to a rate of strain effect because it was found that for smaller roll gaps the rolls tended to stop much more quickly once the power had been cut off. For large gaps the momentum of the motor tended to make them run on a little. Also, when the reduction was very great, there was a tendency for creep to occur in the time between stopping the rolls and releasing the pressure. A particularly bad example of this is shown in Figure 31 and it can be seen that, for meaningful results, measurements have to be taken well away from the point where the rolls stopped. In other words, for very small roll gaps the results are representative of roll speeds of up to 7 r.p.m., whereas for large gaps the roll speed could be virtually zero. The effect of roll speed is therefore discussed in Chapter 5.

COMPARISON WITH RESULTS OF PREVIOUS WORKERS

It has been mentioned that in coating steel strip with zinc, Lund and Armstrong (ref.12) found a linear relationship between total roll force and percentage reduction of steel substrate for any particular initial powder coating. Figure 32 shows this relationship as predicted by the present theory for negligible roll speed and it can be seen that the relationship is not quite linear and is not dependant on the powder coating thickness. This is probably because aluminium is much softer than zinc and will play a smaller part in
the compaction; especially at low strain rates.

THE OCCURRENCE OF SLIP

During the computation of the results shown in Figure 30 it was found that slip messages were given at the point where the sample first entered the nip and just before the steel began to plastically deform. (The calculation is not adjusted to take account of slip but only indicates where it could occur.)

The slip message for the first increment of nip is to be expected and, in practice, it probably represents a slight rotation of the loose powder. The small stresses in this region make it of very little significance.

However, in the region just before the steel begins to deform, the stresses are relatively large. But this occurrence does not seem to effect the validity of the final results so it may be that at these high pressures the coefficient becomes much greater than the 0.58 measured (Fig. 16) due to a moulding together of the layers.
CHAPTER 5

The Effects of Roll Speed
Chapter 5

5.01 Discussion of Possible Effects

INTRODUCTION

The first half of this thesis has shown that the experimental results for a roll gap of greater than 834 microns can be successfully predicted by a theory which takes no account of roll speed. The discussion in section 4.03 indicates that the reason for the discrepancies may be that the experimental results for smaller roll gaps are representative of higher roll speeds. This chapter therefore considers the possible effects of roll speed.

EFFECT OF STRAIN RATE

A very approximate idea of the order of magnitude of strain rates involved in this process can be obtained by dividing the total strain in either the steel or aluminium layer by the residence time in the roll nip. An examination of the theoretical results for the range of conditions under consideration showed that for a roll speed of 7 r.p.m. (peripheral speed 5.5 cm/sec.) the maximum strain rate in the compact is about 10 cm/cm/sec. and in the steel is about 2 cm/cm.sec. Strain rates of this magnitude could have several possible effects.

(1) Plastic flow of a solid is similar to viscous flow in a liquid so that the shear strength increases with flow rate. This effect could increase the strength of the steel or the aluminium metal.

(2) For powder, Whitman and Healy (re. 125) showed that at high loading rates lateral inertia causes an increase in yield.
strength. This could mean that the relationship between axial and transverse stresses (equation 27) would change and also that the powder particles would begin to plastically deform without the normal preliminary of particle rearrangement (ref.118). Therefore the relationship between axial stress and solidity (equation 34) could also be affected.

(3) The pores of any compact contain air which must be expelled as its density increases. Therefore, as Long and Alderton have shown (ref.122), the pore pressure due to trapped air can be increasingly important at higher strain rates. Quantification of these effects would be very difficult and it would certainly be impossible to run the split-die equipment (page 43) at high strain rates. The tensile testing equipment used for characterising the steel could be run at a maximum speed of 40 cm/min. but only if the extensometer is not used. An experimental examination of the effect of strain rate on the material strength must therefore be put beyond the scope of this thesis. However, by a simple experimental technique it is possible to examine the effect of the entrained air as shown in the following sections.
To Vacuum

From Atmos.

Tap A

Sample

Graduated Glass Tube

Oil Level

Low Vapour Pressure Oil

FIGURE 33  Apparatus used to Measure the Permeability of Powder Compacts
Relationship between Compact Density and Permeability

\[ K_a \times 10^4 = 6.05 - 0.074s \]

\[ K_a \times 10^4 = 2.33 - 0.0247s \]
POSTULATION OF THEORY

The velocity at which air can pass through a powder compact of incremental length $dx$ is given by:

$$v_a = \frac{K_a}{n} \cdot \frac{dP_a}{dx}$$  \hspace{1cm} (43)

(Ref. 126)

where $K_a$ is a constant depending on the compact voidage and specific surface. It can be seen from this equation that the air being expelled from the roll nip as the compact density increases will cause an increase in the compact pore pressure which may have a significant effect on the pressure profile.

If there is a pressure difference of $dP_a$ across the increment shown in Figure 27 then equation 6 must be modified to:

$$d(P_a \cdot y_c) - d(q_c \cdot y_c) - d(q_s \cdot y_s) - P \cdot dY + 2uPdx = 0$$  \hspace{1cm} (44)

If the pore pressure in the increment of compact is $P_c$ then the pressure acting on the powders in the compact is given by:

$$P_c = P - P_a$$  \hspace{1cm} (45)

Equation 28 must then be modified to:

$$q_c = K_1 \cdot P_c + K_2$$  \hspace{1cm} (46)

and equation 34 becomes:

$$\frac{100}{s} = K_3 \cdot \ln(P_c) + K_4$$  \hspace{1cm} (47)

If it is assumed that the quantity of air passing right through the nip is negligible then the velocity relative to stationary co-ordinates must be zero. Therefore, relative to the compact:

$$v_a = \frac{V_{a1}}{y_{s1}} \cdot \frac{y_{s1}}{y_s}$$  \hspace{1cm} (48)

which is the velocity of the strip at a given point in the nip.

The relationship between $K_a$ and the solidity $s$ can be found
experimentally and the equations solved numerically using a computer.

**EXPERIMENTAL DETERMINATION OF COMPACT PERMEABILITY RELATIONSHIP**

Compacts of -36 mesh aluminium powder were produced by compaction in a steel tube of known diameter and weight. By measuring the compact length and weighing the tube the compact density was established. The tube was then inserted into the apparatus as shown in Figure 33 and the vacuum pump used to raise the oil level in the graduated tube. Appendix A shows how the constant $K_a$ was calculated by timing the rate of fall of the oil level after tap A was opened to the atmosphere. (The apparatus was tested for leaks by raising the oil level and leaving the tap A closed for several minutes). The results of this experiment are shown in Figure 34.

**PRACTICAL LIMITATIONS**

The results in Figure 34 show that the permeability of the experimental compacts fell to zero at about 93% theoretical density. If this were to happen in the roll-compacted compact then the air which is in the compact at 93% theoretical density would be trapped and carried right through the nip, building up a tremendous pressure in the pores.

Now the experimental technique used to obtain the results in Figure 34 involves forming a cylindrical compact and passing the air through it in a direction parallel to its axis. Therefore air has to pass through the faces of the compact which have been in contact with the die punches. The coordination number of particles at a powder surface must always be less than in the powder bulk so the surface particles will deform more and may well seal the compact
FIGURE 35 Theoretical Density Profiles showing Effect of Air Entrainment

1 Standard Result
2 Modified for Air

Gap = 820 microns
Powder
Weight = 0.015 gms/sq.cm.
FIGURE 36  Photograph showing the disruption of coating for 18 micron powder
to any air flow. Long and Alderton (ref.122) have shown that compacts formed in a die often contain high pressure air pockets. In the rolled compact however, there is always a direct path through the compact bulk between any point in the compact and the loose powder. For this reason it is impossible to say when the compact will become unpermeable but it may be at a density somewhat greater than the 93% indicated by experiment.

**COMPUTATION AND RESULTS**

The experimental limitations discussed above are very unfortunate but they do not completely invalidate the results of this analysis since it is only intended to get an idea of the order of magnitude of errors due to pore pressure. For the purposes of this calculation it was therefore decided to assume that the relative velocity of air to compact is given by equation 48 at all points in the nip and that the pores are only completely sealed at 100% theoretical density. (Experimentation with the calculation showed this to be the worst case). $K_a$, (Fig.34) was therefore extrapolated linearly from $s = 90\%$ to a value of zero at $s = 100\%$.

The viscosity of air is 0.0175 cp. at 20°C and 0.0225 cp. at 135°C which was the highest temperature in the roll nip measured by Tundermann (ref.26). A value of 0.02 cp. was therefore used in these calculations.

Theoretical results are shown in Figure 30 so that they may be directly compared with results where the air effect is ignored. It can be seen that the final coating thickness is greater for the same initial powder coating due to it being less dense. (Generally about 97% rather than 99%). Comparative theoretical density profiles are shown in Figure 35. However, comparison with the experimental results in Table 2 shows that the difference is not sufficient to
account for the discrepancies between experimental and theoretical results at very small roll gaps.

**POWDER FLUIDISATION**

Before passing on to draw final conclusions from this work it may be interesting to make a note on the fluidisation of powder in the nip of the roll. A sample of steel strip was thickly coated with the fine (18 micron) fraction of powder and rolled at 5.5 cm/sec. The result is shown in Figure 36 where the feathery pattern can be clearly seen. The fact that some of the powder in the pattern has fallen away shows that it is caused by low density regions in the coating.

It is almost certain that this effect is caused by the expelled air and not by roll vibration as was thought by Lund and Armstrong (ref.12) Any region with a slightly lower loose-powder density than its surroundings will act as a preferential channel for the expelled air which will tend to reduce the density in that channel still further.

According to Leva (Ref.33) the velocity of incipient fluidisation of a 50 micron particle is 0.135 cm/sec. which is far smaller than the experimental roll speed of 5.5 cm/sec. However, the elutriation velocity of a 50 micron particle is 18.3 cm/sec. For a 50 micron particle one would therefore expect fluidisation but not full elutriation and no pattern was produced in the -36 mesh powder. The elutriation velocity of an 18 micron particle is 6.1 cm/sec. so it looks as though it is the elutriation velocity which is of greatest importance in predicting the occurrence of the feathery pattern.
Chapter 6

6.01 Aims

THE POWDER COMPACTION COATING PROCESS

Steel, coated to prevent corrosion, has always been a saleable commodity. This process is intended as a cheap method of coating steel with aluminium.

The survey of existing literature in Chapter 1 concluded that one of the main problems with the powder compaction coating process is the expelled air which tends to fluidise the powder in the roll nip. In order to eliminate this problem a tremendous amount of work had been done with chemical binders and different deposition techniques. Virtually nothing was known about the actual mechanical behaviour of the compact and substrate in the roll nip. The intention of this thesis was to examine this mechanical behaviour in more detail with particular reference to the air problem.

MULTI-STAGE COMPACTION

General physical principles suggested several methods by which the powder fluidisation effect could be reduced. Addition of chemical binders was obviously one solution but also it should have been possible to reduce the fluidisation velocity of the powder by increasing the particle size or by reducing the expelled gas viscosity. These techniques have the disadvantage of being expensive to apply. Another possibility however, is to roll in stages so that not all the air is expelled at once. The idea would be to provide conditions that would enable the particles to bond together without expelling all the air in the first rolling stage, so that they would not be
fluidised when the air is almost completely expelled in the second stage.

Another problem with the process is that the substrate must be deformed to a certain extent or the powder compact will not bond to it. Therefore, the design of such a system depends upon knowledge of the relative strains in the compact and the substrate.

THE PRESSURE PROFILE

When two layers of incompressible material are rolled the strain in the two layers must be equal. (Provided there is no slip between the layers). It is therefore possible to predict the final relative thickness of the two layers from their initial thickness. The first part of the theory (page 53) shows that this is not possible if one of the layers is a powder compact and therefore compressible. The magnitude of the relative strains can only be calculated from a knowledge of the pressure profile in the roll nip. The bulk of this thesis was therefore concerned with the theoretical prediction of pressure profiles and hence the relative strains in the steel and compact layers.
APPLICATION OF THE SPLIT-DIE

The nature of the process (i.e. thin strip with large width/thickness ratio) made it possible to apply the simple von Karman-type analysis (ref. 69) but for compressible materials it had to be drastically modified. It was not possible to sufficiently characterise the powder using a normal die but it was found that a split-die, built by the U.K.A.E.A., gave all the necessary information and had the added advantage of apparently friction-free compaction.

RESULTS

Pressure profiles were calculated for various size fractions of aluminium powder and the relative strains calculated from these results. The theoretical results were checked by experiment and the following conclusions reached:-

1) With all the factors constant:-
   Increasing the roll gap tends to reduce the maximum roll pressure.
   Increasing the powder coating tends to increase the maximum roll pressure.
   Increasing the roll size tends to increase the maximum roll pressure.
   Increasing the roll roughness tends to increase the maximum roll pressure.

2) Aluminium is so much softer than steel that for any significant reduction of the substrate the powder layer rapidly compacts to its maximum density of 99% theoretical.

3) Particle size variation, within the range examined, and
slight friction coefficient variation (0.085 - 0.095) made very little difference to the relative strains in the layers.

4) For the range of conditions tested there is a unique relationship between reduction of substrate and total roll force which is independent of the initial thickness of powder coating.

5) Theory and experiment are in agreement for most of the results but discrepancies tend to increase as the roll gap is decreased. This was thought to be due to a roll speed effect. The possibility that agreement was dependent on reduction of substrate thickness or initial powder coating density was investigated and found to be unlikely.

POSSIBLE EFFECTS OF ROLL SPEED

The main concern from the above results was that, for large thickness reductions of the coated strip, the experimentally determined coating density was not only less than was predicted by the theory, but was also less than the density for much larger roll gaps. The only reason for this seemed to be that experimental limitations caused results for the smaller roll gaps to be representative of higher roll speeds.

The discussion in Chapter 5 concludes that this effect could be explained by roll speed. However, a detailed theoretical analysis of the effect of air entrainment at high roll speeds showed that this factor alone could not be responsible for the observed results.

It was noted that the feathery pattern formed at higher roll speeds seemed to be related to elutriation of the particles rather than simple fluidisation.
GENERAL CONCLUSIONS

Generally, when rolling bimetallic strips, the stronger layer tends to restrict the lateral strain of the weaker layer and so increase its yield strength, while the weaker layer reduces the yield strength of the strong layer. This work shows however, that at negligible roll speeds, aluminium is so much softer than steel that the powder causes negligible lateral stress in the substrate. Thus the compact density must be about 90% theoretical before the steel begins to plastically deform and will reach 99% theoretical before the strain in the steel is more than about 0.5%. It is not likely that a commercial process could operate with smaller or smoother rolls than the ones used in this experimental work, so it seems that the multi-stage idea is not possible. It would be interesting however, to test the theory for a stronger powder such as iron.

INVESTIGATION OF ROLL SPEED EFFECTS

This work shows that at negligible roll speed the multistage idea is not feasible. However, experimental work indicates that at higher roll speeds it may be possible to obtain adequate deformation of the substrate at fairly low compact densities. It is hoped that this would enable more air to pass through the rolls and so increase the speed at which fluidisation becomes a problem.

Solution of the pressure profile for such a system would require the design of apparatus for dynamic determination of axial to radial stress ratios in a compact. Obviously the split-die apparatus cannot be speeded-up but a hydraulic system based on the triaxial test
may be feasible although it would involve very complex mechanical and electrical design. Such equipment may show that at high rates of strain metal powders have no time to rearrange and so bond together at very low densities. In this situation the initial bulk powder density may be important.

**PREVENTION OF 'PICK-UP'**

Experimental work has shown that if the degree of compaction is not sufficiently great the powder emerging from the nip will stick to the roll surface rather than to the steel substrate. It is not clear whether this is due to insufficient bonding between individual particles in the compact or between the compact and the substrate. However, it has been shown that large particles bond together at lower compacting stresses than small ones. It would be interesting to carry out a controlled scientific test to see whether the extra stiffness obtainable with larger particles is of any use in the prevention of 'pick-up'.

6.04 Summary of Conclusions

1) Roll speed in the coating process is restricted by fluidisation of powder entering the roll nip and the need to deform the substrate sufficiently to enable the coating to bond to it.

2) Fluidisation seems to become a problem when the strip speed is approximately equal to the elutriation velocity of the powder.

3) Fluidisation can be reduced by several methods, including mixing a binder with the powder, using larger particles, reducing the viscosity of the expelled gas and trying to increase the proportion of air which passes right through the rolls.

4) Where the roll speed is very small the mechanical behaviour in the roll nip can be predicted by a von Karman - type analysis using results from split-die equipment.

5) The deviation from theory which occurs at higher roll speeds cannot entirely be explained by excess pressure due to entrained air.

6) With more quantitative information about the behaviour of powders at high rates of strain it may be possible to design a system in which the problem of fluidisation is drastically reduced.
**APPENDIX A**

Calculation of compact permeability-density relationship from experimental results

For a compact of given voidage the pore velocity is given by:

\[ V_a = \frac{K_a}{n} \cdot \frac{d}{dx} \frac{dP_a}{dx} \]  

(49)

Air is compressible so its density will be a function of pressure. Therefore its velocity will also be a function of pressure. The equation above can therefore only be applied to a compact of finite length if \( dP_a \) is very small compared with \( P_a \). For the apparatus shown in Figure 33 the pressure drop across the compact can be no more than 20 cm of water so the equation becomes:

\[ V_a = \frac{K_a}{n} \cdot \frac{\Delta P_a}{L} \]  

(50)

If the cross section area of the compact is \( A_c \) then:

area for air flow \[ = A_c \cdot (1 - s/100) \]

The velocity of air is then proportional to the rate of fall of the oil level in the graduated tube.

\[ V_a = \frac{A_o}{A_c \cdot (1 - s/100)} \cdot \frac{dh_o}{dt} \]  

(51)

The pressure drop across the compact is proportional to the height of oil level.

\[ \Delta P_a = D_o \cdot h_o \]  

(52)

Substitution into equation 50 gives:

\[ \frac{A_o}{A_c \cdot (1 - s/100)} \cdot \frac{dh_o}{dt} = \frac{K_a}{nL} \cdot D_o \cdot h_o \]  

(53)

Integration gives:

\[ \frac{A_o}{A_c \cdot (1 - s/100)} \cdot \ln\left(\frac{h_o}{20}\right) = \frac{K_a}{nL} \cdot D_o \cdot t \]  

(54)

where the initial height of oil level is 20 cm. It can therefore be seen that the value of \( K_a \) for any compact of known density can
be found by plotting $h_0$ against $t$ on log-linear paper and measuring the gradient. The results of this experiment are shown in Figure 34.
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