Utilisation of silicon rubber to characterise tool surface quality during die compaction

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A new methodology was developed to observe and measure tool wear and tool surface quality during the die compaction process. The newly developed method is a non-destructive test that relies on silicon rubber to transcribe the inner surface profile of the compaction die. After verification of the method, aluminium and iron alloy powders were compacted to quantify tool wear and tool surface quality with two die materials, tungsten carbide and tool steel. The tool surface quality was quantified by recording surface roughness of the die replicas on a surface profiler.

Keywords: Tool wear, Compaction, Silicon rubber, Surface roughness, Aluminium alloy powder, Iron alloy powder

Introduction

1 Compaction of powders is a common method of making components by shaping and densifying powders through mechanical pressing in the field of powder metallurgy (PM). Although there may be alternate manufacturing methods such as casting and forging, many times the cost and properties provided by PM components are more attractive. During die compaction, pressure is applied through upper and lower punches to press powders into the desired shape. Compaction tools are designed for the given powder, press and desired shape.[1]

A common problem in compaction is the phenomenon of tool wear. Wear is the gradual removal of material from one surface due to exposure to another material surface. There are numerous factors that affect the wear rate in powder compaction. The punches and dies are in contact with each other as well as other metals, many times with hard particles, so the normal force, velocity, distance, and lubrication, and in combination with the large production quantities lead to tool wear.[2] Wear with aluminium and iron alloy powder compaction is no different. Because of aluminium's high oxidation rate, the surface oxide makes it especially prone to cause wear when forced onto another metal surface.[3] The oxide content increases even more as the particle size become smaller, which increases the hardness of the particle. Aluminium alloys can cause even more wear problems when they contain higher volume percentages of a constituent or a hardening phase, such as silicon or a ceramic.[4–6] For iron alloy powder, smaller particles harden at lower pressures during pressing since there are more contact points per unit volume. This leads to lower density and causes more wear on the die than with the larger particles.[7]

It is difficult to quantify tool surface quality in a production environment, largely because the equipment must be diverted from production to perform tests. In this paper, the authors demonstrated a way to measure and quantify tool surface quality in compaction of aluminium and iron alloy powders with two die materials. Silicon rubber replicas of the die cavity, taken at set intervals, were measured using a surface profilometer.

Experimental

Powder and tool materials

Two powders and two tool materials were used in this study.

As for the powders, one is the premixed and air atomised aluminium alloy powder (Ampalloy AMB 2712, Ampal, Inc., Maryville, TN) and the other is a water atomised iron alloy powder (HVC + 0·7% lubricant, North American Hoganas, Hollsopple, PA). Table 1 gives characteristics of the powders and Fig. 1 shows the scanning electron microscopy (SEM) images of the powder particles using Stereoscan 360 (Car Zeiss SMT Inc., Oberkochen, Germany).

As for the tool materials, one is a standard cemented tungsten carbide (BC-14S, Basic Carbide, Lowber, PA) and the other is the tool steel (CPM T15, Crucible Materials Corp., Syracuse, NY). The hardness of the die material was measured using a diamond indenter and 150 kg load on the Rockwell C scale (LR 100, LECO, Joseph, MI) and the results are nearly identical to the expected values as given in Table 2.[8] Note that all punches are made from M4 tool steel (M4, Crucible Materials Corp., Syracuse, NY).

Sample preparation by compaction

The Al alloy powder and Fe alloy powder were compacted using a Carver manual press (Carver Inc., Wabash, IN) and an industrial Stokes tablet press

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(Advanced Machinery Inc., Clinton Township, MI). The hand press was used to make rectangular compacts for compressibility measurements and the tablet press was used for the tool wear studies.

Compressibility experiments

Transverse rupture strength bars of the Al alloy and Fe alloy powders were compacted using the hand press with die wall lubricant (Zinc Stearate Dry Powder Mold Release, Aervoe Industrial Inc., Gardnerville, NV). The green densities of the compacts were calculated using mass and volume measurements and Archimedes method. The mass scale was calibrated using multiple measurements of a 10 g mass standard and its accuracy was $\pm 0{\cdot}0008$ g. The vernier caliper accuracy was $\pm 0{\cdot}01$ mm.

Compaction cycles

To observe the effect of wear of the Al alloy and Fe alloy powders on tools in an industrial environment, the industrial Stokes tablet press was used to compact the powders with the two die materials. Cylindrical compacts with diameters of 6·35 mm and heights of $\sim 5{\cdot}30$ mm were compacted at a rate of ~ 70 compacts per minute.

The carbide die was used initially to benchmark the press features by calibrating the upper punch stroke length to achieve compact relative densities of 95% theoretically. The force exerted on the powder during compaction can be calculated using the relation between compact density and pressure from the compressibility curve obtained in the previous experimental part.

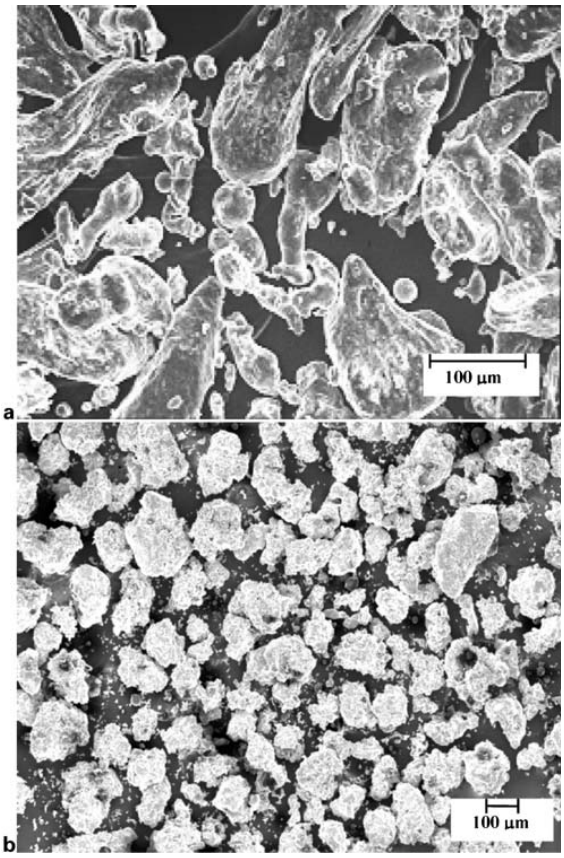
The Al alloy powder was compacted in both tungsten carbide die and CPM T15 die and Fe alloy powder was only compacted using the CPM T15 die.

Transcription of die surface using silicon rubber

Silicon rubber

The silicon rubber has negligible shrinkage as it sets. It could be used to produce a replica of the die cavity with resolution of 0·1 μm, strength of 15 to 20 kPa, and its application temperature ranges from -10 to 180°C.[9]

Die cavity replicas were created using RepliSet (Struers Ltd, West Midlands, United Kingdom) which is a two part silicone rubber compound that is squeezed into the die via a dispensing gun through a static mixing nozzle as shown in Fig. 2. Replicas were taken from the die installed on the press and compaction was started immediately upon removal of replica. The filled die cavity was then covered with backing paper, provided by Struers, to indicate the orientation on the press at 0, 90, 180 and 270° counterclockwise. These marks gave an orientation that was referenced during measurements of the replica. Replicas of the die cavity were made at intervals from 1000 to 10 000 compactions.



1 Scanning electron microscopy images for *a* Al alloy powder and *b* Fe alloy powder

Surface quality characterisation

Three dimensional surface data of the die cavity replicas were obtained using a surface profilometer (Talysurf CLI 2000, Taylor Hobson Ltd, Leicester, United Kingdom). The conventional roughness parameter S_a, which is the arithmetic average of absolute value in the height or z direction, was calculated as follows[10–12]

$$S_a = \frac{1}{N}\sum_{i}^{N} |z| \tag{1}$$

where i is the dummy variable for summation and N is the total number of measurement points.[11]

Verification experiments using standard roughness block and the replica material were performed to verify

Table 2 Measured hardness values of die materials

Die material	Hardness, HRC
Tungsten carbide	78·1±0·3
CPM T15	65·8±0·3

Table 1 Powder characterisation of aluminium and iron alloy powders

Powder	Al alloy	Fe alloy
Vendor	Ampal	North American Hoganas
Composition	Al–3·8Cu–0·75Si–1·0Mg	Fe–0·9Mo–2Ni–0·2C
Lubricant, wt-%	0·0	0·7
Mesh	−100/+325	−60
Pycnometer density, g cm^{-3}	2·71	7·64
Apparent density, g cm^{-3}	1·22	3·16
Tap density, g cm^{-3}	1·51	3·87

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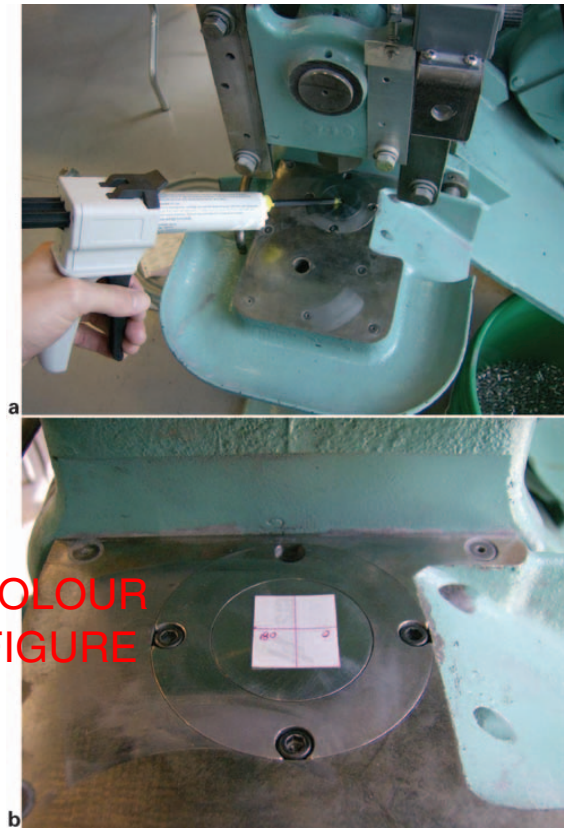


2 Process of creating die cavity replica using Struers RepliSet system by *a* injecting two part mixture via dispensing gun, *b* applying backing paper to remove die replica from die cavity after curing: paper orientation is labelled for later use on surface profilometer

the accuracy of the proposed method through the surface profilometer measurements.

Microstructure of wear surface

The replicas and punches were prepared for SEM. The SEM used for viewing the replicas and punches was a JOEL field emission gun scanning electron microscope (field emission FE-SEM; 6500F, JOEL Ltd, Tokyo, Japan) at 5 and 10 kV accelerating voltage respectively.

Results and discussion

Calibration of replica

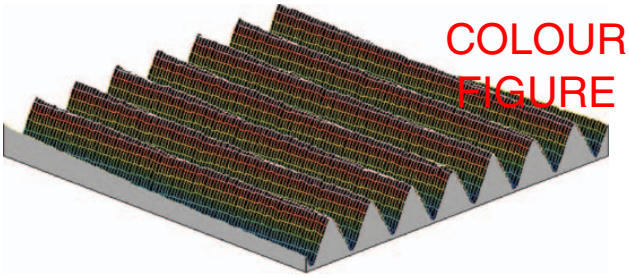
Three-dimensional surface data of the die cavity replicas were obtained using a surface profilometer with a manufacturer specified resolution of 0·1 μm. Calibration experiments were performed to investigate the accuracy of the surface profilometer measurements using a flat standard roughness block and the replica material.

Flat surface

First, scanning was performed on a standard block with 6·0 μm roughness using different combinations of measurement speed, spacing distance, resolution, and scanning area with consideration of the total time to perform each reading.

Table 3 gives results from the resolution study comparing five different speeds combined with two different measurement areas. The resolution chosen criteria was based on the fastest time to complete the measurement without sacrificing accuracy. The authors found that scanning speed of 1 mm s^{-1} with scanning area of 1 mm^2, gave the closest S_a value with positive deviation of 0·23 μm, while scanning speed of 2 mm s^{-1} with scanning area of 2 mm^2 produced the most close S_a value with negative deviation of −0·14 μm.

To further improve measuring efficiency and accuracy, extra replicas were then taken for the flat roughness standard (Fig. 3). Those replicas were tried measuring using the scanning speed of 2 mm s^{-1} and scanning area of 1 mm^2. The relation between measured S_a value and scanning speed with the effect of scanning area size could be seen in Fig. 4*a*. Roughness parameters were read at three zoomed areas within each measurement. Five measurements were made, and the standard deviation of the total 15 readings is 0·08 μm. As indicated in Fig. 4*b*, it is the optimum scanning set-up. The resolution for replicating flat surface is ±0·08 μm.



3 Three-dimensional surface profile of roughness standard

Curved surface

However, validation data were also required of a curved surface since the die cavity is cylindrical. This was performed by replicating a hole in an aluminium plate and then sectioning the hole and comparing both surfaces on the profilometer. Five measurements were made for both the surfaces of aluminium plate sectioning hole and replica, and the average S_a values are 7·40 and 6·92 μm respectively, while the standard deviations for two groups of data are 0·16 and 0·13 μm respectively. The average difference of S_a between real curve surface and replica surface is 0·48 μm, the error is 6%. Therefore, accurate and repeatable results could be obtained by averaging small scan areas. The resolution for replicating curved surface is ±0·48 μm.

Table 3 S_a calibration results of flat 6·0 μm standard

	Input parameters				Output
		Spacing, μm			
Condition	Speed, mm s^{-1}	*x*	*y*	Area, mm^2	S_a, μm
1	9·00	10	5	1	5·59
2	6·00	10	5	1	5·64
3	3·00	10	5	1	5·86
4	1·00	10	5	1	6·23
5	0·25	10	5	1	6·55
6	0·10	10	5	1	6·68
7	0·05	10	5	1	6·79
8	9·00	10	5	16	5·66
9	2·00	10	5	16	6·28
10	1·00	10	5	16	6·32

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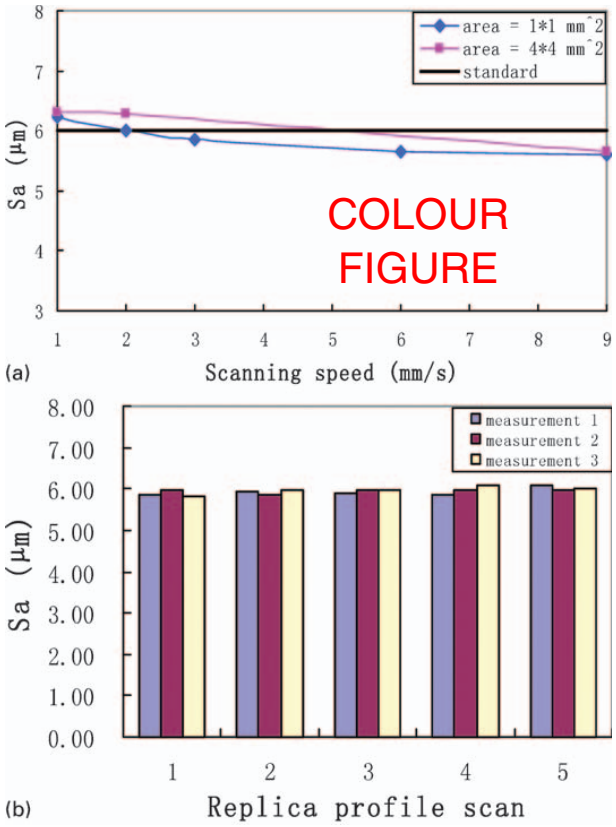
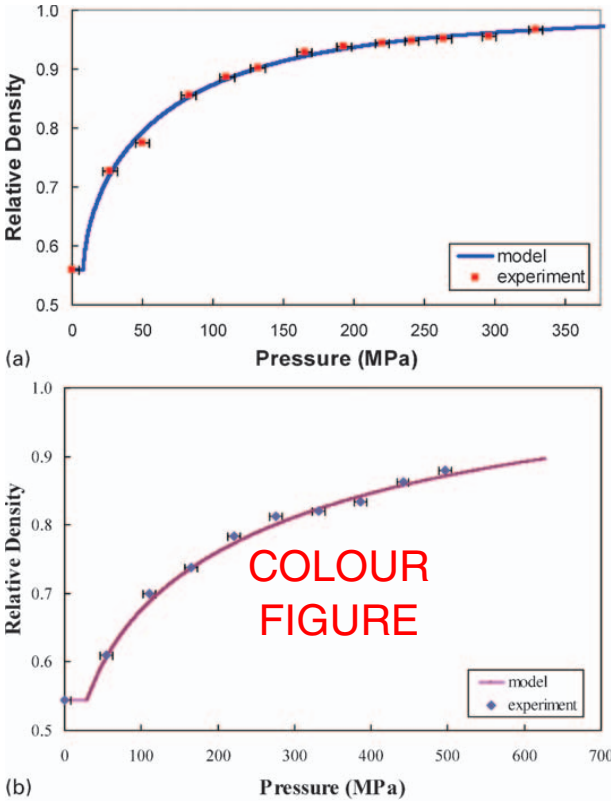


a S_a value versus scanning speed with effect of scanning area; *b* S_a value distribution for five measurements with optimum scanning speed and scanning area

4 Replica resolution calibration results



5 *a* green density versus compaction pressure of Al alloy powder including curve fit using Shima and Oyane model and *b* density versus compaction pressure of Fe alloy powder

Diameter measurement

Lastly, the replica diameters were measured on the surface profilometer using the single scan surface profile feature. Measurements recorded using this feature give values to the nearest 10 μm interval.

Die compaction behaviour

Compressibility experiment

The rectangular Al and Fe alloy powders compact green densities versus compaction pressure are given in Fig. 5. The results show the trend in that it becomes asymptotically harder to increase relative density as the compaction pressure increases. Error bars in Fig. 4*a* are included to show the accuracy of pressure and density measurements. The density variance is too small to observe on plot.

The constitutive model proposed by Shima and Oyane is used to fit the green density data, as determined by PMSolver. The yield surface F of the Shima and Oyane model is expressed as follows[13]

$$F=\left(\frac{q}{\sigma_m}\right)^2+\alpha(1-\rho)^{\beta}\left(\frac{p}{\sigma_m}\right)^2-\rho^{\delta} \quad (2)$$

where q and p are the effective stress and hydrostatic pressure, ρ is the relative density, σ_m is the flow stress of the matrix material, α, β and δ are the material parameters. The matrix material is assumed to be work hardening in form of following equation

$$\sigma_m=a_m+b_m\varepsilon_m^{n_m} \quad (3)$$

where a_m, b_m and n_m are the material parameters and ε_m is the effective strain of matrix material.

Table 4 shows all material parameters of Al and Fe alloy powders by curve fitting based on the Shima and Oyane model. The relative density curve fits the experimental data points with 0·5% average difference. From these data, the goal is to predict the pressure applied to Al and Fe alloy powders in compaction.

Industrial compaction cycles

Cylindrical compact diameters were measured after ejection. The diameter changed from nominal dimension of 6·35 to 6·36 mm, which results in 0·2% radial springback.

Seven compacts were measured for each material condition. The average density of aluminium alloy compacts is 2·59 g cm^{-3}, while average density of iron alloy compacts is 6·19 g cm^{-3}. As referred to Fig. 4*a* and *b*, the pressure could be predicted. For Al alloy powder, the pressure applied was ~340 MPa, while for Fe alloy powder, the pressure applied was ~130 MPa.

Table 4 Material parameters of Al and Fe alloy powders based on Shima and Oyane model

Material parameters	Al alloy powder	Fe alloy powder
α	6·2	6·2
β	1·028	1·028
δ	1·84	9·98
a_m, MPa	15·1	514·9
b_m, MPa	292	254
n_m	1·92	0·05

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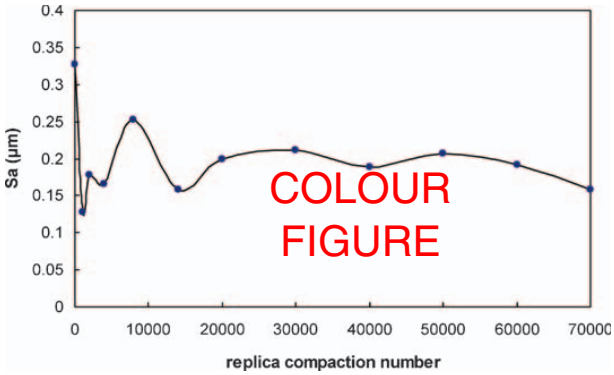


6 **Surface roughness measurements for carbide die replicas measured by surface profilometer**

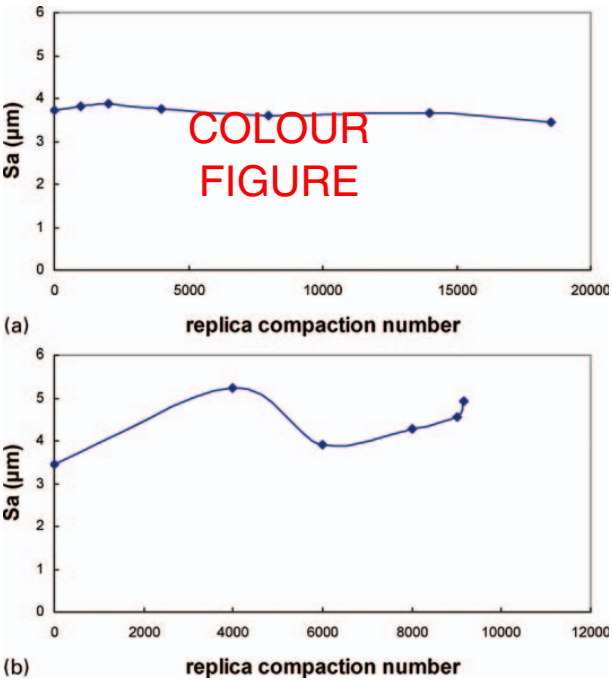
Surface property measurement

Surface quality characterisation

The die replicas were created at intervals as previously described. The surface roughness and profile measurements for carbide and T15 dies are given in Figs. 6 and 7 respectively. As indicated previously, the resolution of the profilometer for curved surface is $\pm 0{\cdot}48$ μm.

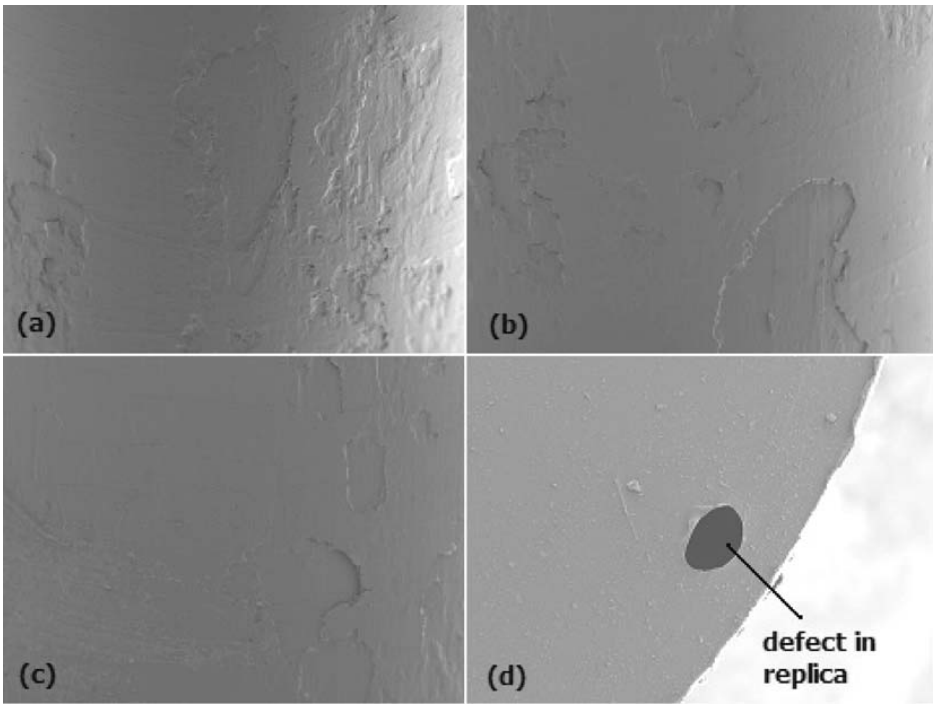
Carbide die

For carbide die replicas, all values fall within $\pm 0{\cdot}48$ μm, the calibrated resolution of the profilometer for curved surface, thus no surface roughness change was observed. The carbide die did not show any wear throughout the first 70 000 compaction cycles based on the surface profilometer data. The lack of a trend in the surface roughness measurement data show that the number of compaction cycles did not cause significant wear in the carbide die. It is common for a die material such as carbide to produce one million compacts before needing replacement. However, the die replicas only described



7 **Surface roughness measurements for T15 die replicas measured by surface profilometer**

the die cavity from the top face of the bottom punch up to the opening on the die table. Punch seizure started below the bottom punch face due to a build-up of powder between the bottom punch and die wall. This is proven by the large amount of flashing resided on the compact seized between the punches after failure. The lower punch also had a significant amount of Al adherence and the die cavity had a thin layer of Al alloy powder in the area where the lower punch was positioned. Because the bottom punch was forced out of



a–c surface around radius of replica; *d* bottom surface where bottom punch comes in contact with powder

8 **Scanning electron microscopy images magnified from 30 to 75 times at 5 kV of T15 die replica after sputter coating for 40 s**

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the die, there is no way to quantify how much Al alloy powder was 'trapped' between the lower punch and die.

Therefore, compacting soft material such as aluminium within a relatively small compaction cycle number, 30 000 or less, the surface roughness change occurred on WC die is out of range for the present technique.

T15 die

For the T15 die replicas, no obvious surface roughness change was observed during the Al powders compaction. After shifting to Fe powder, the surface roughness change was obvious. Unlike the Al powder, the Fe compacts would have an effect on the surface roughness of die cavity wall as they were very hot upon ejection due to adiabatic heating. The replicas reproduced the die cavities exceptionally well. This methodology proved to be excellent for observing galling on the die wall.

Surface microstructure

A coated die replica as observed in the SEM after sputter coating is shown in Fig. 8. This replica is the final condition of the T15 die after punch failure due to high compaction pressure using the Fe powder. The defect in this replica was caused by entrapped air during the curing process. The lines around the curvature of the die (left to right) are tooling marks from the manufacturing of the die. The marks were visible upon visual inspection of the die cavity immediately after receiving from the manufacturer. The large recessed areas show the amount of build-up along the die wall. The lower right image shows the top view of the replica which is the area that replicates the lower punch face. The hole pictured is a defect in the replica where air was trapped during the replication process.

Conclusion

Measured flat surface roughness values were most accurate ($\pm 0{\cdot}08$ μm) when taken with small area scan of 1 mm^2, at a 2 mm s^{-1} measurement speed, at a consistent scanning resolution. With the same scanning set-up, a surface roughness value as accurate as $\pm 0{\cdot}48$ μm can be achieved for curved surface. This RepliSet technique has its limitation when applied to aluminium powder compaction in WC die, while it reproduced the CPM T15 die cavity very well, which is used to compact iron powder. Scanning electron microscopy images proved to be useful in observing sputter coated replicas at low acceleration voltages.

The data from the experiments can now be verified computationally. Unlike dimensional measurements on the green compact, which are influenced by ejection through the die, this replication approach gives tool surface roughness data without requiring tool removal from the press, and the influence of dimensional change is minimised.

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