Adhesion of tin droplets impinging on a stainless steel plate: effect of substrate temperature and roughness

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Abstract

We photographed impact of small tin droplets on stainless steel surfaces of varying temperature and roughness. To achieve high impact velocities the test surfaces were mounted on the rim of a rotating flywheel. Substrate temperature ($T_s$) was varied from 120 to 220 °C and surface roughness ($R_a$) kept at either 0.05 or 2 μm. We kept constant the impact velocity (30 m/s) and droplet diameter (0.6 mm). To form a coating 60 droplets were deposited randomly on each stainless steel test coupon. Deposition efficiency was evaluated by dividing the mass adhering to the coupon by the mass of sixty droplets prior to impact. The maximum deposition efficiency was achieved at a substrate temperature of 160 °C. For $T_s < 160 °C$ the deposition efficiency was higher on a rough surface ($R_a = 2 \text{ μm}$) than on a smooth surface ($R_a = 0.05 \text{ μm}$), since splats did not adhere well to the smooth surface. For $T_s \geq 160 °C$ the deposition efficiency was higher on a smooth surface ($R_a = 0.05 \text{ μm}$) than on a rough surface ($R_a = 2 \text{ μm}$), since splats splashed less on the smooth surface.

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1. Introduction

The surface properties of a component can be modified by depositing a thin layer of a different material on it. Coatings are often employed to protect exposed surfaces from heat, corrosion and wear, or they can be used to alter substrate electrical, magnetic and optical properties. One widely used technique of applying such surface layers is thermal spray coating, in which particles of the coating material are melted and sprayed onto the substrate. Depending on the method used to heat and accelerate particles, their velocities may range from a few meters per second to supersonic speeds, and diameters from tens of microns to a few millimeters. Molten droplets flatten as they impact, coalesce with each other and freeze, forming a dense layer. Metal, ceramic and polymer coatings have all been applied by this method.

When applying a thermal spray coating it is important to ensure that it adheres well to the substrate and does not peel off during use. Careful surface preparation before spraying is important to ensure good coating adhesion. Usually the part to be coated is grit-blasted to clean it and increase surface roughness. During spraying molten droplets flow into microscopic cavities in the rough substrate as they spread and solidify, so that mechanical interlocking provides strong bonds.

Roughening the surface enhances coating adhesion, but it also promotes splashing of impinging drops. Molten particles fragment into smaller pieces during impact, which bounce off the surface. Since the deposition efficiency (the fraction of coating material sprayed that adheres to the substrate) of thermal spray coating processes is often less than 50%, and coating materials are expensive, wastage is a significant problem.

When molten particles in a thermal spray impinge on a solid surface and freeze, the shapes of the flattened splats formed are a function of both the roughness and temperature of the substrate. If the substrate temperature is above the so-called ‘transition temperature’, splats are disk-shaped, without any evidence of splashing [1–3]. Computer simulations [4] showed that splashing is produced by solidification. If there is sufficient heat transfer between the droplet and substrate, the edges of the droplet freeze while the droplet is still spreading. This obstructs the flow of liquid and produces an irregular splat. Alternately, if heat
transfer from the droplet is low (as when the substrate is hot, or if there is large contact resistance between the splat and substrate), it spreads completely before solidifying and produces a circular splat. Large surface roughness also provides an obstacle to flow and promotes splashing. In coating applications it is preferable to have disk splats, since they adhere well to the substrate, increasing adhesion strength \([5]\).

The interactions between droplet impact parameters and surface properties that trigger splashing are very complex. Stow and Hadfield [6] studied the effects of surface roughness on spreading and splashing of water droplets and established that splashing was promoted by increasing drop diameter \((D)\), impact velocity \((V)\), and surface roughness \((R_a)\). These drop impact parameters were combined with the density \((\rho)\), viscosity \((\mu)\), and surface tension \((\sigma)\) of the liquid to give two non-dimensional groupings: the Reynolds number \((Re = \rho V D / \mu)\) and Weber number \((We = \rho V^2 D / \sigma)\). They found that droplets splashed only if the so-called ‘splash parameter’ \(K = We^{0.25} Re^{-0.25}\) exceeds a critical value \((K_c)\), which depends on the surface roughness. Fukumoto et al. [7] argued that since the deformation of a molten droplet is greatly affected by solidification, provided that the impact and solidification times are of the same order of magnitude, a new splash parameter should be defined to account for the substrate temperature. Li et al. [8] suggested that droplet splashing could also be caused by surface contamination. When molten metal droplets were sprayed onto a surface coated with volatile organic materials they splashed, which was attributed to vapor escaping from below droplets. When the substrate was preheated sufficiently to evaporate all impurities there was no splashing and splats were disk shaped.

Though a large literature exists on thermal spray coating, there have been few attempts to directly view molten droplets as they land on a solid substrate, freeze and coalesce to build up a coating. Our objective in this study was to observe how surface roughness and temperature affect splat shape and deposition efficiency. We photographed molten tin droplets (0.6 mm diameter) as they landed with uniform velocity (30 m/s) on stainless steel coupons to form a coating. We designed and built a molten metal droplet generator to produce tin droplets on demand. In order to achieve the required impact velocity the substrate was mounted on a rotating flywheel. By synchronizing the time at which a droplet was ejected with the rotation of the flywheel we ensured that the moving substrate hit airborne droplets with high velocity and photographed the ensuing impacts. We varied substrate temperature \((T_s = 120–240^\circ\text{C})\) and roughness \((R_a = 0.05\text{ or } 2\text{ \mu m})\) and measured deposition efficiency for coatings built up by depositing 60 droplets.

### 2. Experimental method

#### 2.1. Tin droplet generator

A molten metal droplet generator was used to produce uniform-sized molten tin droplets. Fig. 1 shows a schematic diagram of the droplet generator. The main body of the generator was a 50.8 mm diameter by 50.8 mm long stainless steel cylinder. The chamber was filled with tin pellets (99.8\% pure, Aldrich Chemical Company, Milwaukee, WI). A 400 W band heater (Model HBA-202040, Omega Engineering Co, Stamford, CT) regulated by a temperature controller (Model CN9000A, Omega Engineering Co, Stamford, CT) was used to maintain the chamber temperature above the melting point of tin \((232^\circ\text{C})\). The temperature of the droplet generator was measured with a K-type (Chromel–Alumel) thermocouple inserted into a hole drilled into the steel cylinder.

A commercially available synthetic sapphire nozzle (Model??, Swiss Jewel Co, Philadelphia, PA) with a 178 \(\mu\text{m}\) orifice was inserted into a hole drilled through the other wall of the chamber and sealed in place with Teflon tape. Teflon o-rings sealed both bottom and top of the chamber. A T-fitting was connected to the top plate of the droplet generator, so that one outlet acted as a vent while the other was connected to a nitrogen tank whose outlet pressure was varied from 125 to 200 kPa. A solenoid valve (Model 8262G202, Convalve Company, Toronto, ON) was placed between the nitrogen tank and chamber. When the solenoid valve opened briefly \((8–40\text{ ms})\) a pressure pulse was sent to the chamber forcing a molten tin droplet out through the nozzle. The pressure in the chamber was then relieved by gas escaping through the vent hole, preventing more droplets from being ejected. By this method single droplets could be produced on demand by sending a signal to

| Nomenclature | \(T_s\) | substrate temperature |
|--------------|-------|----------------------|
| \(D\)        | droplet diameter |
| \(K\)        | splash parameter |
| \(K_c\)      | critical splash parameter |
| \(P_s\)      | stagnation pressure |
| \(P_{\sigma}\) | capillary pressure |
| \(R_a\)      | average surface roughness |
| \(V\)        | droplet impact velocity |
| \(V_f\)      | maximum flattening velocity |
| \(\sigma\)   | surface tension of droplet |
| \(\mu\)      | viscosity of droplet |
| \(\rho\)     | density of droplet |
| \(Re\)       | Reynolds number \((= \rho V D / \mu)\) |
| \(We\)       | Weber number \((= \rho V^2 D / \sigma)\) |
the circuit controlling the solenoid valve. Experiments were performed using pressure pulses with an amplitude of 33 kPa and width of 12.5 ms which produced droplets with a diameter of 0.6 mm.

Molten tin oxidizes promptly once it is exposed to air; to prevent oxidation of tin droplets emerging from the droplet generator, an aluminum pipe (nitrogen co-flow pipe in Fig. 1) with an inner diameter of 10 mm was attached to the bottom of the chamber. Nitrogen was injected into this pipe to shield droplets from atmospheric oxygen. The volume flow rate of nitrogen was adjusted until it was just enough to prevent oxidation. The effect of oxidation on droplet formation was immediately obvious since it produced non-spherical, tear-drop shaped droplets.

2.2. High-speed impact mechanism and photography technique

To achieve high impact velocities, it is easier to accelerate the substrate rather than the droplet. One way of doing this is to mount the substrate on the end of a rotating arm. Fig. 2 shows a schematic diagram of the apparatus we built to capture images of droplet impact by synchronizing the ejection of a droplet from the generator with the position of the moving substrate.

A rectangular aluminum plate (38.1 mm long × 25.4 mm wide × 9.5 mm thick) was mounted on the rim of an aluminum flywheel with an outer diameter of 406.4 mm. Another identical plate was fixed at the opposite side of the flywheel to act as a counterbalance. Replaceable stainless steel coupons (with the same linear dimensions as the plate and 0.51 mm thick) were bolted to the aluminum plate and used as test surfaces. Experiments were done with two sets of surfaces. One set was polished on a metallurgical wheel to a mirror finish. Its average surface roughness ($R_a$) was measured to be 0.05 μm, using a PDI surfometer (Milan, MI) that records the surface profile of a component by running a stylus over it. The second set of coupons was sand blasted to have a surface roughness of $R_a = 2 \mu m$.

The center of the flywheel was mounted on a vertical stainless steel shaft whose diameter and length were 19 and 190 mm, respectively. The shaft was connected to a variable speed DC motor (Model MS3130-04/T, Dynetic Systems, Elk River, MN) through a flexible coupling. By adjusting the voltage applied to the motor a rotational speed of 1363 rpm was obtained, giving the test surface a linear speed of 30 m/s. The droplet velocity as it exited the generator was less than 1 m/s, small enough to assume that droplet impact was normal to the surface. In order to prevent unwanted vibrations and wobbling of the flywheel the upper end of the shaft was supported by a stainless steel plate (565 mm × 50.8 mm square and 19 mm thick), which was secured on two threaded posts (11 mm in diameter and 457 mm long). The whole system was mounted on a vibration isolation table. A Lexan enclosure surrounded the whole apparatus for safety.

A slip ring (Model S4, Michigan Scientific, Charlevoix, MI) was mounted on the upper end of the flywheel shaft and
used to carry electric power to the test surface and thermocouple signals from it. The stationary part of the slip ring was fixed to a support frame. To heat the substrate two 120 W cartridge heaters (Omega Engineering Co., Stamford, CT) were inserted into holes in the aluminum plate. A Chromel–Alumel thermocouple was inserted into the center of the plate with its tip touching the stainless steel coupon. The rear surface and sides of the aluminum plate were insulated to minimize heat losses due to convection to the air and conduction to the flywheel. By varying the voltage applied to the heaters the temperature of the test surface could be controlled. Spatial temperature variations from one side of the test surface to the other when it was moving were less than 5 °C.

A CCD video camera (Sensicam, Optikon Corporation Ltd., Kitchener, ON) was used to photograph droplet dynamics during impact. It had an intensified CCD chip capable of recording 30 frames per second with a resolution of 1280 × 1024 pixels. The camera could also superimpose up to ten images in every frame, each with an exposure time as short as 0.1 μs, separated by delays that varied from 0 to 1 ms (selectable in 0.1 μs time steps). A 0.1 μs exposure time was short enough to capture the deformation of the droplet during the impact, without any blurring caused by the extremely fast motion of the substrate.

To hit a falling droplet with the moving substrate, and to photograph its impact, three events had to be synchronized with the position of the arm: ejection of a droplet, triggering of the camera, and triggering of a flash to provide illumination. An optical sensor (Model HOAO880, Honeywell Optical Switch, Toronto, Ont.) was used to pick up the signal caused by the flywheel rotation. This signal was then used to trigger the camera, flash, and droplet generator. Since the frequency of this signal was too high to directly drive the droplet generator, it first passed through a frequency divider, which reduced the frequency by a factor that varied from 2 to 32, depending on the rotational speed of the arm. The low frequency signal formed one input of an AND gate (see Fig. 2).

When we were ready to take a photograph we pressed a switch which activated the second input of the AND gate, so that the pulses at the other input were transmitted to a time delay unit. The rising edge of each pulse provided a reference we used to time all other events. The digital time delay generator (Model DG 535, Stanford Research Systems, Sunnyvale, CA) controlled the timing of three subsequent actions with pico-second resolution. We made droplets collide with the substrate by varying the delay between the reference pulse and triggering of the droplet generator. Each droplet was ejected from the generator and fell to a position coincident with the center of the test surface just as the substrate approached the droplet.

The flash (Model MVS 7000, EG and G Corp., Salem, MA) timing was adjusted so that droplet impact was illuminated by a 10 μs long burst of light. While the flash was on, the camera was activated to take a single 0.1 μs
exposure of an impacting droplet. By varying the time at which the camera was triggered, different stages of droplet impact were recorded, and the entire process of droplet impact was reconstructed from a sequence of such pictures.

A software package (SensiControl 4.03, Kitchener, ON) was used to transfer the images from the camera to a computer. Droplet dimensions were measured using an image analysis software. The resolution of these measurements, corresponding to one pixel of the digital image viewed on a computer monitor, was 5 µm.

To measure the deposition efficiency, for any given substrate temperature and roughness, 60 droplets were deposited on a coupon. We deliberately introduced some randomness in the time the droplets were produced, so that they landed on different points on the test surface. The coupon was then removed and weighed. The increase in mass of the coupon divided by the calculated mass of 60 droplets gave the deposition efficiency. Each experiment was repeated eight times for each set of impact conditions, and the average deposition efficiency calculated.

3. Results and discussion

Fig. 3 shows the spreading of a 0.6 mm molten tin droplet impacting with a velocity of 30 m/s on a stainless steel surface with $R_a = 0.05$ µm. The images in Fig. 3 are labeled a–f, indicating successive stages of spreading. Before impact the droplet was at a temperature of approximately 233°C, just above the melting point of tin (232°C). The stainless steel substrate was initially at room temperature, approximately 20°C. The Reynolds and Weber numbers had values of $Re = 69,700$ and $We = 7102$, respectively. Following impact a circular sheet of liquid jetted out from under the drop (Fig. 3(a)). The leading edge of this sheet detached (Fig. 3(b)) and disintegrated into fine particles (Fig. 3(c)–(e)). The remaining metal formed a solidified splat (Fig. 3(f)). The pattern of splashing was quite different from that observed previously by Aziz and Chandra [9] for larger (2–3 mm diameter) droplets impacting at low (1–4 m/s) speed, where finger shaped perturbations formed and detached producing satellite droplets around the edge.

Increasing the substrate temperature produced significant changes in droplet impact dynamics. Fig. 4 shows a sequence of photographs of the impact of a 0.6 mm tin droplet under the same conditions as those in Fig. 3 (impact velocity 30 m/s, substrate roughness $R_a = 0.05$ µm), except that the surface temperature was raised to 240°C, above the melting point of tin (232°C). In this case the droplet did not freeze, but remained liquid after spreading. It spread to a much larger diameter (Fig. 4(e)) than it did on a cold surface (Fig. 4(f0)), and we no longer observed the rim of the droplet detach immediately after impact as in Fig. 3(b). Computer simulations [4] have shown that when a drop impacts on a cold surface, freezing around its circumference obstructs flow; the remaining liquid jets out over the solidified periphery and splashes. Heating the surface retards solidification, allowing the droplet to spread out further and avoid splashing.

Fig. 5 shows splats formed following impact on surfaces at three different temperatures: 80, 150, and 260°C. At the lowest temperature, 80°C, the droplet splashed, and both the surface and the rim of splat were irregular. At the highest temperature, 260°C, the splat was smooth and quite circular in shape. The transition occurred at a surface temperature of approximately 160°C; droplets landing on surfaces at temperatures above this did not splash. We also observed that adhesion between the droplets and substrate improved when the substrate temperature was in the range 160–180°C. This was deduced from the fact that fewer splats detached off the rotating surface due to centrifugal forces.

Fig. 6 shows successive stages during the formation of a tin coating by deposition of droplets on two stainless steel coupons at the same temperature (120°C) but with different roughness. The left picture in each frame of Fig. 6 shows the surface with $R_a = 0.05$ µm while the right one has $R_a = 2$ µm. The number under each frame denotes the number of droplets deposited on the substrate. On the smooth surface there was much less splashing, and more of
the mass of the first droplet remained attached than on the rough surface (see the frame labeled $n = 1$). However, the splats were not strongly attached to the smooth surface, and frequently large portions of the deposit detached and flew off the surface (see $n = 5$). Consequently the deposition efficiency on coupons with $R_a = 0.05 \text{ mm}$ showed very large variation from one sample to the next. On average, the deposition efficiency was slightly lower on the smooth surface than on the rough surface.

Deposition efficiency on the smooth surface increased when its temperature was increased to 180 °C. At this temperature droplets impinging on coupons with $R_a = 0.05 \text{ mm}$ did not splash as much as on those with $R_a = 2 \text{ mm}$, and formed smooth, rounded splats (see Fig. 7). They adhered strongly after impact and did not detach.

On the rough surface ($R_a = 2 \text{ mm}$) droplets exhibited a large degree of splashing upon impact, and only a small portion of the splat remained attached to the substrate.

On a substrate at a temperature of 220 °C (Fig. 8), there was little splashing on the smooth surface. However, the substrate was almost at the melting point of the droplet and solidification was delayed, allowing the splats to rupture at several places. When a droplet landed on another previously deposited splat it splashed and broke up (see $n = 9$). Adhesion of the splats was not as strong as that on a surface at 180 °C, and several droplets failed to stick to the surface. For example, of the first nine drops deposited only a few are visible on the test surface. On the rough surface both splashing and detachment of splats led to the deposition efficiency being even lower than on a smooth surface.

Our measurements of deposition efficiency as a function of surface temperature are summarised in Fig. 9. Each symbol represents the average of eight measurements; error bars mark the largest and smallest values recorded. For $T_s < 160 \text{ °C}$ the deposition efficiency was higher on rough
coupons ($R_a = 2 \mu m$), with which splats bonded well. The error bars are very large for smooth coupons ($R_a = 0.05 \mu m$) because coating adhesion was weak and sometimes large portions of the coating fell off, producing great variability in deposition efficiency measurements. For $T_s \approx 160 ^\circ C$ the deposition efficiency was higher on a smooth surface, because there was less splashing and loss of mass upon impact.

Deposition efficiency increased with temperature for $T_s < 160 ^\circ C$, because droplet solidification was delayed as the substrate became hotter, allowing liquid more time to flow into surface cavities and form strong bonds. When $T_s \geq 160 ^\circ C$ surface temperature had little effect on coating adhesion to a smooth coupon. For rough coupons deposition efficiency decreased with surface temperature because splashing was enhanced on hotter surfaces.

A droplet adheres to a rough substrate because of mechanical interlocking between the surface and the bottom of the splat. To estimate how rough the surface has to be to allow good adhesion we can do a simple order-of-magnitude comparison between the stagnation pressure in an impacting droplet, which drives liquid into the substrate roughness, and surface tension forces that restrain the liquid.
The stagnation pressure in the liquid \( (P_s) \) is approximately:

\[
P_s = \frac{1}{2} \rho V^2
\]  

where \( \rho \) is liquid density and \( V \) the impact velocity. The capillary pressure \( (P_\sigma) \) resisting the entry of liquid into a surface pore of radius \( r \) is

\[
P_\sigma = 2\sigma/r
\]

where \( \sigma \) is the surface tension. If \( P_s > P_\sigma \), liquid will be forced into the pore. Assuming \( r \sim R_a \), the condition for a pore to be filled with liquid is:

\[
R_a > \frac{4\sigma}{\rho V^2}
\]

In our experiments for tin we have: \( \sigma = 0.53 \text{ N/m} \), \( \rho = 6970 \text{ Kg/m}^3 \), and \( V = 30 \text{ m/s} \). If we substitute these values in Eq. (3) we obtain \( R_a > 0.33 \) \( \mu \text{m} \) for good coating adhesion. For the polished coupons \( R_a = 0.05 \) \( \mu \text{m} \) and hence we expect poor bonding between the splat and the substrate. For the grit-blasted coupons \( R_a = 2 \) \( \mu \text{m} \) and surface pores will be easily filled with liquid. This increases the mechanical bonding considerably and therefore no splat detachment was observed on the rough surface. However, at high temperatures splashing became the dominant effect on a rough surface, decreasing deposition efficiency (see Fig. 9).

4. Conclusions

We have designed and built an apparatus to simulate high-speed impact of molten metal droplets on stainless steel plates. We photographed impact of droplets to build up a coating on the surface. We measured the deposition efficiency for 0.6 mm tin droplets impinging on surfaces of varying temperature and roughness. We arrived at the following conclusions:

- Below a substrate temperature of approximately 160 °C tin droplets splash during impact. At higher temperatures they form smooth, round splats.
- Adhesion strength of splats to the substrate increases above this transition temperature.
- The maximum deposition efficiency for a coating formed by deposition of 60 droplets was achieved at a substrate temperature of 160 °C.
For $T_s < 160 \, ^{\circ}C$ the deposition efficiency was higher on a rough surface ($R_a = 2 \, \mu m$) than on a smooth surface ($R_a = 0.05 \, \mu m$), since splats did not adhere well to the smooth surface. The deposition efficiency increased with surface temperature.

For $T_s \geq 160 \, ^{\circ}C$ the deposition efficiency was higher on a smooth surface ($R_a = 0.05 \, \mu m$) than on a rough surface ($R_a = 2 \, \mu m$), since splats splashed less on the smooth surface. Deposition efficiency did not change with surface temperature for the smooth surface, but decrease with increasing temperature on the rough surface.

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