Compression Tooling Surface Treatments—Options for Resolving Corrosion, Wear, Filming, and Tablet Defects

Armin H. Gerhardt

“Pharmaceutical Processes” discusses scientific and technical principles associated with pharmaceutical unit operations useful to practitioners in compliance and validation. We intend this column to be a useful resource for daily work applications. The primary objective for this feature: Useful information.

Reader comments, questions, and suggestions are needed to help us fulfill our objective for this column. Manuscripts submitted by readers are most welcome. Please send your suggestions to column coordinator Armin Gerhardt at arminhg@comcast.net or to coordinating editor Susan Haigney at shaigned@advanstar.com.

KEY POINTS DISCUSSED
The following key points are discussed in this article:

• Selection and maintenance of appropriate compression tooling is an important consideration in tablet manufacturing
• Tooling problems during commercial manufacturing are difficult to predict
• Tooling problems may be caused by moisture content, granulation particle size, abrasiveness of formulation, and other factors
• Careful evaluation of punch appearance including location and appearance of abrasion and wear may suggest causes for tablet defects
• Compression tooling punch tip treatments such as plating and vapor deposition may be recommended to correct tablet defects
• Tooling vendors are a valuable technical resource for correction of tooling problems
• The regulatory impact of tooling including punch tip treatments must be considered
• Compliance professionals should be aware of potential tablet defects and associated concerns that may be indicative of compressing tooling problems. These include tablet defects such as picking and sticking. Punch tip treatments may be useful in solving these problems.
• Other compliance concerns associated with tooling include proper use and care of tooling and proper evaluation of new tooling.
INTRODUCTION
There are myriad factors to consider and balance when a new compressed tablet product is introduced to commercial scale manufacturing. Among them is the selection of robust compression tooling. Tablet mass and shape, the choice of a film coat application, and printing or debossing characters onto the tablet faces frequently garner the most attention. The choice of steel for the compression tooling often defaults to a standard grade of tooling steel. Based on the limited experience from pilot-plant or small-scale lots that fulfill clinical study and stability study needs, it may not be possible to predict if future challenges due to excessive wear or corrosion of contact areas between the tooling and drug product will appear. Sporadic film formation on the punch faces may eventually force a halt to production. Interim cleaning of the punch tips that may include partial disassembly and reassembly of the turret causes downtime; production can then be restarted after quality control tests are acceptable. Prediction of which product will cause these types of challenges is very difficult. Often they are noticed only after tens of lots have been completed. At this point there are no options for reformulation or equipment changes, and the research and development team has moved to other efforts. Then the compressing operators and manufacturing supervisor’s skills are challenged to maintain productivity. The erratic pattern of film formation or picking can be particularly troublesome. Factors such as formulation moisture content, relative humidity as impacted by weather conditions, granulation particle size, uneven wear of particular punches within a set (perhaps due to uneven buffing or polishing) and others are causes for tooling problems (see Table I). These sporadic problems may defy prediction and diminish productivity.

TABLE I: Primary causes for tooling problems.

<table>
<thead>
<tr>
<th>Cause</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture content</td>
</tr>
<tr>
<td>Relative humidity granulation particle size</td>
</tr>
<tr>
<td>Uneven wear of particular punches within a set</td>
</tr>
<tr>
<td>Abrasive particles in formulation</td>
</tr>
<tr>
<td>Corrosion on punch cups and faces during production or storage</td>
</tr>
<tr>
<td>Uneven tooling buffing or polishing</td>
</tr>
<tr>
<td>Excessive compressing forces</td>
</tr>
</tbody>
</table>

NEW COMPRESSION TOOLING
New compression tools are delivered with highly polished punch faces. These contact surfaces arrive with a very smooth finish that is readily apparent by their reflective shine. The punch cup surface forms the surface of the compressed tablet. It is imperative that the original punch cup finish be retained for as long as possible. With use, the punch faces gradually lose their smooth surface and shine due to the abrasive and/or corrosive effects of the powder blend, along with the scratches induced by abrasive cleaning techniques. These microscopic imperfections in

**TABLET TOOLS BASICS AND TERMINOLOGY**
Fundamental information about tablet tooling, including technical terminology, should be discussed before addressing compression tooling surface treatment (1, 2). Figures 1 and 2 illustrate typical tablet tooling and a corresponding compressed tablet. Terminology indicated on the figures will be used in later discussion. Important terms to note include the following:

- **Punch face.** The portion of the tablet punch that contacts the formulation to form the tablet. Includes the punch cup and punch land.
- **Punch cup.** The rounded portion of the tablet punch that contacts the formulation to form the rounded portion of the tablet.
- **Punch cup depth.** The perpendicular distance from the highest point of the cup (apex) to the punch edge.
- **Punch cup apex.** The deepest point of the punch cup.
- **Punch land.** The area between the punch cup and the edge of the punch.
- **Debossed tooling and embossed tablets.** Tablet identification by means of engraving into the punch face. This results in raised “embossed” identification on the tablet surface.
- **Embossed tooling and debossed tablets.** Tablet identification by means of raised engraving on the punch face. This results in engraved “debossed” identification on the tablet surface.
the punch face provide increased surface area into which the powder blend is compressed. As the punch retracts, powder within the scratches or grooves of the punch may stick to the punch. This produces a less smooth tablet surface and provides a location for subsequent compression cycles to accumulate increasing amounts of powder on the punch surface. This eventually yields the gross defects called sticking and picking (see Table II). Buffing serves to restore the smooth punch face, but it does this by removing thin layers of punch surface material from the punch face. Eventually this may diminish the metal of the punch land area (i.e., the region between the outside of the punch tip and the punch face) and increase the likelihood of gross cracks in this zone of thin metal on the punch tip. Buffing may also have limitations in reaching certain regions of a punch face. For instance, the embossed characters may have corners that are relatively more difficult to access during buffing.

**TOOLING SURFACE EFFECTS**

What surface treatment options for compression tooling are available for these situations of excessive wear, corrosion of contact areas, film formation, or picking? The first step is a thorough evaluation of the location and appearance of the punch tip.

**Punch Land Effects**

With deep cup tools, it is possible for abrasive products to cause a band of wear (i.e., visible as loss of the original mirror sheen) near the punch land or for film formation to appear consistently at the punch cup apex.
In both cases, there are significant regional differences in the compression force delivered to a tablet. The band near the punch land is under relatively greater compression force, thus causing the higher density scratches and matte (dull) finish.

**Cup Apex Effects**

On the other hand, film formation at the cup apex has relatively little compression force that is insufficient to form strong bonds within the powder bed. A first option here is to modify the cup depth to a shallower configuration that makes for a more uniform distribution of compression force to the tablets.

**Cup Surface Effects**

When punch wear is seen uniformly across the entire cup surface, the compression force is consistent. In this situation, improvements are possible by selecting a different grade of steel for the tooling, or by application of a surface hardening treatment to the tip area of the tooling.

**STANDARD TOOLING AND PREMIUM TOOLING**

The type of steel used in the manufacture of tooling provides the basic strength of the tooling. Considerations for choice of steel include the number of tablets to be produced, the design of the tablets to be manufactured, the abrasiveness of the material (i.e., formulation components) to be compressed into tablets, and the amount of force needed for compression. Standard tooling steel is selected on the basis of demands to which it will be subjected. For pharmaceutical materials, the typical choices are steel grades S1, S5, or S7. These grades provide an appropriate surface contact material for cleanliness and a relatively high degree of toughness to withstand millions of compression cycles with reasonable wear resistance and to withstand the abrasion from formulation components. Once a tablet has been compressed between the upper and lower punches, there is relatively larger potential for abrasion to cause wear on the interior of the die cavity as the compressed tablet is forced out. For this reason, premium steel grades D2 or D3 steel are used to make the tablet dies. Dies made with premium steel grades have relatively larger wear resistance and have a service life comparable to the punches. Premium steel grades are harder than standard steel grades, but are also more brittle and subject to breakage. Tooling manufacturers can also recommend maximum levels of compression force for tablet punches based on the type of steel used, punch tip treatment, and punch shape.

While the excipients of a formulation rarely cause a chemical corrosion to occur, there have been cases where the drug substance does induce punch corrosion. This may occur because of either the reactive moieties within the drug or their salt formation. For these instances, changing to grade 440C stainless steel (i.e., premium grade steel) with higher chromium content makes the punch material less reactive.

**PUNCH TIP TREATMENTS**

Among other options available are various punch tip treatments. To be effective, these must not compromise the punch tip surface finish. Hardness has to be maintained or improved. There needs to be strong bonding between the punch tip and the coating to prevent flaking and product contamination. The treatment has to be of uniform thickness and chemically inert (3).

**Plating**

Plating of the punch tips is also an option. Hard chromium plating has been utilized to prevent film formation, minimize wear, prevent or minimize corrosion, and/or reduce friction. Chromium plating is applied in an electrodeposition process from a chromic acid solution to a thickness of approximately 30 µm (4). Key process controls are required for the chromic acid solution, its temperature, the electrical current density, and the anodes. Nickel may also be plated in an electrolytic process, either alone or in combination with boron or polytetrafluoroethylene (PTFE, Teflon)

**TABLE II: Tablet defect terminology.**

<table>
<thead>
<tr>
<th>Term</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Picking</td>
<td>Removal of a small portion of tablet due to sticking to the tablet punch or die</td>
</tr>
<tr>
<td>Sticking</td>
<td>Adherence of granulation particles to the tablet punch or die</td>
</tr>
<tr>
<td>Filming</td>
<td>Formation of a very thin film of formulation on the tablet face resulting in a dull matte appearance</td>
</tr>
</tbody>
</table>
to improve hardness or lubricity, respectively. This technique is based on a chemical oxidation-reduction reaction. A reducing agent reacts with metal ions in solution to cause deposition of the metal. It has the advantages of not being dependent on electrical flux differences at the surface and thus yields an even deposit on irregularly shaped tools. It also permits the incorporation of various suspended additives that then become integrated in the plating. A subsequent heat treatment step is then required to achieve full hardness (Ni) or prevent hydrogen embrittlement (Cr). Adhesion of the coating to the punch tip is based on mechanical interlocking of the precipitate to the surface. There may need to be an abrasive step included before the plating is applied, and the coated punches must be re-polished after plating.

**Physical Vapor Deposition**

A second type of punch coating process is physical vapor deposition (PVD). In this approach, a vaporized form of the coating material condenses onto the surface being treated, much like frost formation on glass at cold temperatures. This process requires temperatures of 900°F or more to ensure adhesion. These high temperatures may soften the entire tool or produce fluctuation in tooling dimensions; this most commonly occurs in total tool length. It is important to use a matched set of tooling that ensures uniform tablet hardness, disintegration/dissolution performance, and ultimate drug bioavailability. As the coating grains grow, there may be variation in their orientation and growth rate thereby degrading the original surface finish on the cup face. Among the materials that may be applied are titanium nitride (TiN), chromium nitride (CrN), and diamond-like carbon (DLC). The DLC coatings are amorphous carbon-based material with high hardness, low friction properties, chemical inertness, and the potential to reduce sticking.

**Ion Beam Enhanced Deposition**

A third type of punch coating process is ion beam enhanced deposition (IBED), which employs two independent beams directed onto the surface being coated. One beam contains coating material; the second contains an inert or reactive ion. The IBED coating initially permeates the punch tip surface and alloys with the base material. As more coating material is applied, a coating layer that is 2 to 4 microns thick is formed. This process is performed under vacuum at temperatures below 200°F, which reduces the potential for tool distortion. And unlike chrome and nickel plating, there is no need to abrade or roughen the punch surface to provide coating adhesion. The original highly polished cup face surface finish is thus maintained. Coating materials that may be applied include chrome, chromium nitride, and titanium nitride. The titanium nitride material is relatively better than the others at prolonging tool life with high wear rate products. See Table III for a comparison of wear resistance for punch tip treatments.

The machine tool industry serves the needs of a wide range of products, with pharmaceutical tooling comprising a small portion. As technical innovations are developed within the machine tool industry from metallurgists and materials scientists, they seem likely to be applied first outside drug manufacturing because of their larger size and commercial impact. The pharmaceutical industry benefits when it remains open to adapting the tool industry’s progress to pharmaceutical products. Thorough testing and careful evaluation of these advancements need to be done so that the pharmaceutical industry may yield improved efficiency and economic benefits.

**TOOLING VENDORS**

Establishing a strong relationship with the vendor(s) of your tooling provides a valuable technical resource

---

**TABLE III: Relative wear resistance for punch tip treatments (adapted from reference 3).**

<table>
<thead>
<tr>
<th>Punch Tip Treatment</th>
<th>Relative Wear Resistance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electroless nickel plating with PTFE</td>
<td>0.4</td>
</tr>
<tr>
<td>D2 and S7 steel hardened to Rockwell C of 56</td>
<td>1.0</td>
</tr>
<tr>
<td>High-speed steel hardened to Rockwell C of 65</td>
<td>3.3</td>
</tr>
<tr>
<td>Hard chrome plating</td>
<td>4.3</td>
</tr>
<tr>
<td>IBED chromium nitride</td>
<td>8.7</td>
</tr>
<tr>
<td>Diamond-like carbon by PVD</td>
<td>16.3</td>
</tr>
<tr>
<td>IBED titanium nitride</td>
<td>86.7</td>
</tr>
</tbody>
</table>
that may be of considerable assistance when challenged with tablet tooling problems. There are a wide variety of punch tip treatments that may work for specific product(s) with specific problems. It is important to recognize that experimental data needs to be generated supporting each application. It is entirely feasible that a particular tooling treatment may not resolve the issues despite the best theoretical justification. It is still necessary to build a solid foundation of experience before initiating commercial production with new tooling. Perhaps two or more treatment options appear logical. Evaluating both types with three to five stations of tooling under identical conditions for a minimum of 200,000 compression cycles per punch to as many as 25 million compression cycles may be predictive. Altering the design of the tablet shape or the type of steel to a premium quality may be a first option. There are punch treatments available that cost more than the punch itself; however, this may be justified by the increased productivity and/or life span of these treatments.

**Regulatory Considerations**

Verification of the acceptability of a particular material for use on human or veterinary pharmaceutical products is required. While many of the materials used for punch treatments have been utilized successfully for many years, requiring documentation of this from the tooling vendor and performing a legitimate review internally is desirable. Regulatory agency reviewers prefer to base their review on a documented evaluation that found the material(s) inert or non-reactive, not carcinogenic, and without toxicity from either acute or chronic exposure. A regulatory reviewer’s conclusion that there is no objection to the use of a particular material in food or drug applications is likely the strongest statement they will provide. Very rarely, if ever, does an agency grant approval for use. It is the responsibility of the manufacturer to ensure a product’s safety.

**IMPLICATIONS FOR COMPLIANCE**

Compliance professionals should be aware of potential tablet defects and associated concerns that may be indicative of compressing tooling problems. These include tablet defects such as picking and sticking, film ing on punches, and other problems. These problems often may be corrected by minor formulation changes (e.g., increased tablet lubricant). However, the inherent abrasive properties of the formulation may require special treatment to compression tooling for extended commercial manufacturing without problems or tablet defects. These treatments include use of premium steel and various surface treatments such as plating, physical vapor deposition, and ion beam enhanced deposition.

**Use And Care Of Tablet Tooling**

Tablet compression tooling punches and dies are carefully manufactured to defined tolerances. Punches and dies must be treated with care, protected from damage, prevented from scratching, prevented from bumping other punches, well lubricated, and carefully packaged when shipped or transferred between manufacturing sites. Manufacturing operators must know and not exceed maximum compression forces recommended by manufacturers. Using the lowest compressing force necessary to produce acceptable tablets is desired.

**Evaluation Of New Tooling**

When tablet tooling needs to be upgraded to include surface treatments, there should be adequate evaluation of the new treatment before use in production. This may be accomplished by obtaining several punch and die combinations with the new surface treatment (or multiple treatments) and running simulated production materials for an extended time period. After evaluation, full sets of tooling may be purchased and introduced into commercial scale production. Tablets produced with the new tooling should be carefully monitored for presence of defects by means of additional sampling.

**Tooling Replacements**

Replacement punches must use the same steel grade and tooling finish as originally purchased or modified. Tooling vendors may supply standard steel unless specifically requested to provide special coating. Purchasing departments should be cautioned to not change or omit notations on purchase orders.

**Tooling Vendor Expertise**

Tooling vendors are extremely knowledgeable regarding diagnosis of tableting problems and corrective actions.
They are readily accessible and can offer expert recommendations when tableting problems occur. Special tooling treatment such as plating and other techniques described previously are made only when less rigorous approaches to correct the problem have failed.

**CONCLUSIONS**

Compliance professionals should be aware that compression tooling treatments may be very effective in solving tablet defect problems. Careful tooling evaluation should identify the cause of tablet defects. Vendors of tablet tooling may be helpful in recommending solutions to problems caused by excessive tooling wear. Potential surface treatments should be evaluated before implementing in full-scale commercial manufacturing. Compliance professionals should be aware of factors that may contribute to the potential for tablet defects including the proper use and care of tablet tooling.

**REFERENCES**


**ARTICLE ACRONYM LISTING**

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>CrN</td>
<td>Chromium Nitride</td>
</tr>
<tr>
<td>DLC</td>
<td>Diamond-Like Carbon</td>
</tr>
<tr>
<td>IBED</td>
<td>Ion Beam Enhanced Deposition</td>
</tr>
<tr>
<td>PTFE</td>
<td>Polytetrafluoroethylene</td>
</tr>
<tr>
<td>PVD</td>
<td>Physical Vapor Deposition</td>
</tr>
<tr>
<td>TiN</td>
<td>Titanium Nitride</td>
</tr>
</tbody>
</table>

**ABOUT THE AUTHOR**

Armin H. Gerhardt, Ph.D., is an industry consultant who spent more than 16 years at Abbott split between formulation services in R&D and project management for new drug product development teams. Armin retired from Abbott in 2007. He has taught various courses in pharmaceutical processing for many years. Armin has also authored book chapters on pharmaceutical unit operations. He can be reached at arminhg@comcast.net.