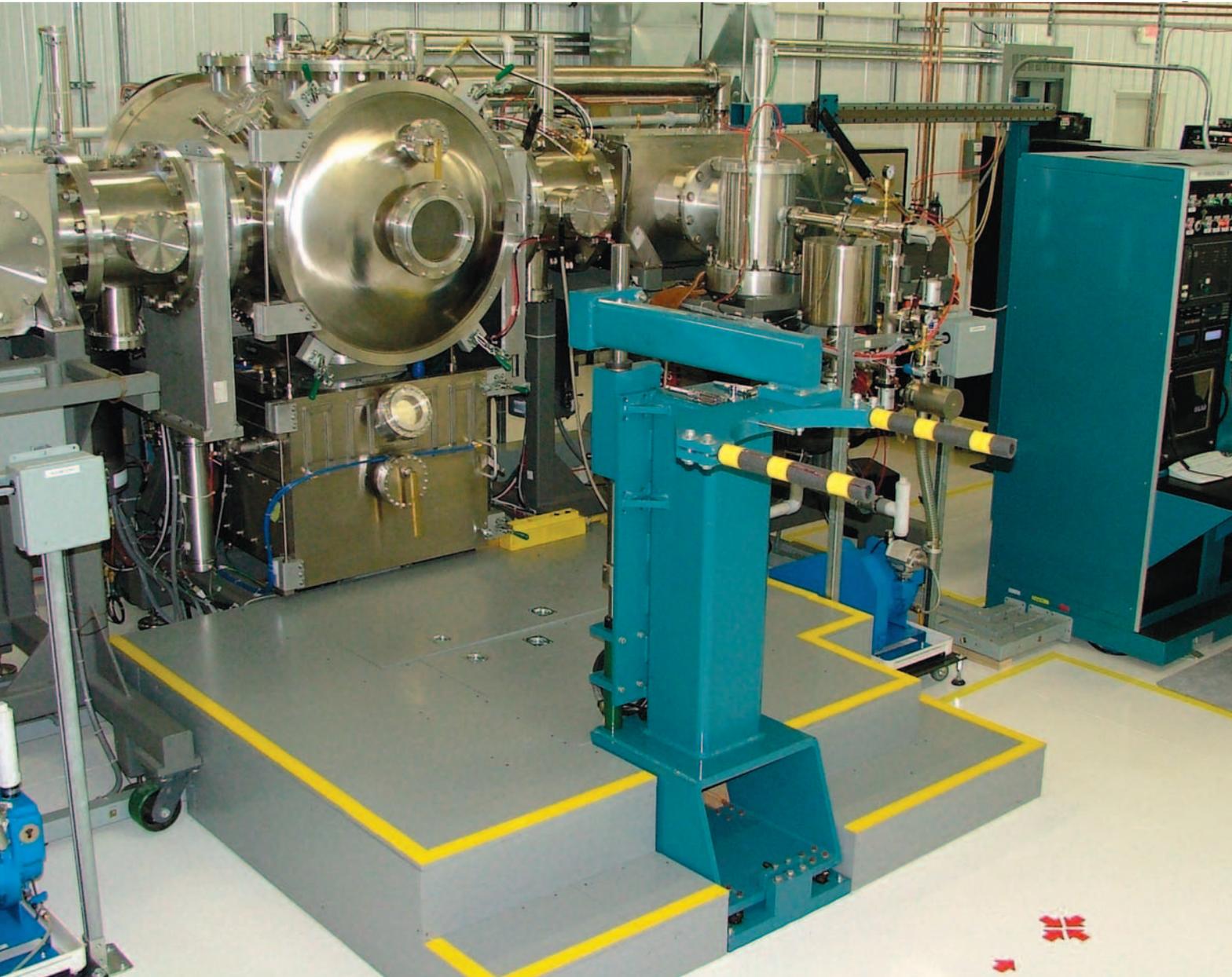


coatings for tooling

CHOOSING COATINGS TO IMPROVE
THE PERFORMANCE OF TABLET
TOOLING

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If tableting equipment is the engine driving your manufacturing operation, you want to keep it running as smoothly and efficiently as possible. This article describes how specialized coatings preserve and improve your tooling. It examines several coatings and coating methods and assesses the advantages and disadvantages of each.

Industrial processes, such as plastic molding and metal stamping, use engineered coatings to improve the performance of manufacturing tooling. These same coatings can help tableting tools resist corrosion and slow the rate at which tool surfaces degrade, thus improving product quality and manufacturing productivity. The coatings can also improve the performance of tablet press tooling.

To take full advantage of these coatings, it's important to understand the surface interactions on the tablet tool face. With this knowledge, you can choose a coating and coating process that best meet your needs.

Surface finish and tool performance

When you buy new tableting punches from the manufacturer, they're free of wear and corrosion, and the punch-cup surfaces are highly polished. As these punches compress powders into tablets, the punch cup replicates its surface finish on each tablet. Therefore, to ensure tablet quality, you must preserve the surface finish on the punch cup. In addition, if the punch cup surface becomes abraded, scratched, or corroded, the roughened face can trap small powder particles, which can lead to sticking and picking defects.

Figure 1 shows the surface profiles of the polished areas on the face of new and used tablet punch cups. The profile of the polished face of the new punch with a spectral (highly reflective) finish is very smooth and shows no features deeper or wider than 0.05 micron. To see features this small requires very high magnification, 4,000X or more.

As you use the punch, the powder you're tableting will abrade the surface, and the spectral surface will eventually appear matte. This is caused by microscopic scratches and digs in the surface that are 0.25 to 1.00 micron wide and deep. To see these requires magnification of 750X or more. Large, hard granules in the powder or contaminants from buffing wheels (from punch manufacture or routine maintenance) can produce even deeper scratches. To see them requires a magnification of 500X

or more. Surface corrosion can leave very deep pits, some of them hundreds of microns in depth. It can also build up oxide or sulfide scales on the surface. These you can see with the naked eye.

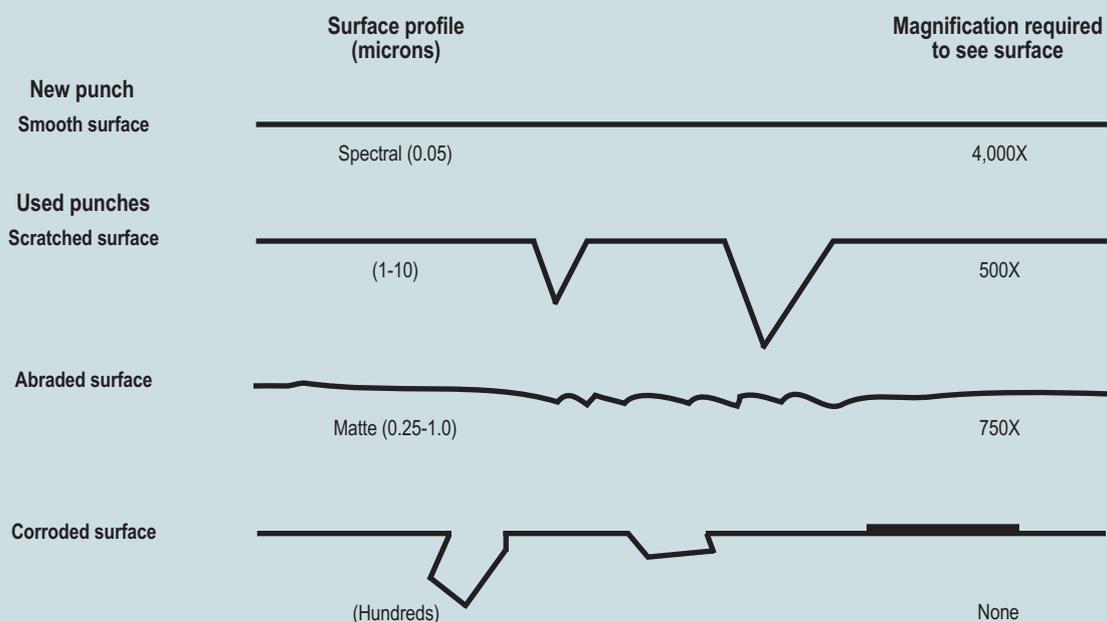
Increased mechanical interlocking. As the polished surfaces of the punches roughen, powders can become trapped in mechanical surface imperfections. See Figure 2. Over time, more powders will stick to these trapped particles and will build up on the surface. Eventually, filming and sticking will appear, and you'll have to halt tableting to clean the punches.

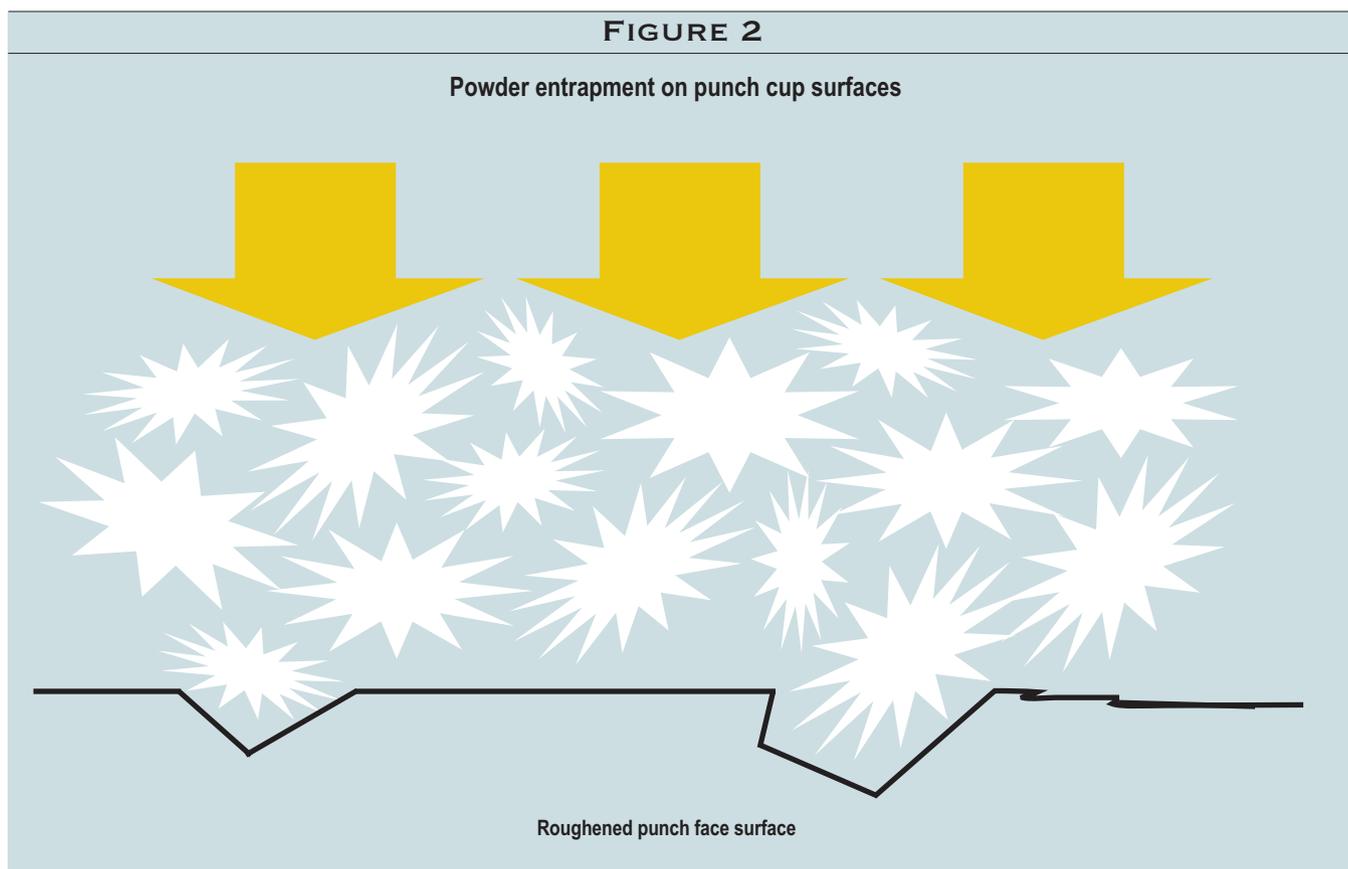
Likewise, if the surfaces corrode and rust layers build up on the punch surface, powders will stick to the roughened rust layers. While detergents and solvents will remove the adhered powder, they won't restore the original finish to the punch surfaces. Instead, you may have to buff out the scratches, pits, and corrosion layers to restore the surface finish in the cup.

Reduced tool life. If you have to rely on buffing and polishing to restore worn and corroded punch faces, your efforts will become counterproductive. Buffing compounds contain abrasives that actually remove thin layers of the metal surface to polish out scratches and pits. Repeated buffing can reduce the critical punch dimensions in the land area—the area between the edge of the punch cup and the outside diameter of the punch tip—thus degrading the fit between the punch and die. Frequent buffing also rapidly degrades the aspect ratios of the embossing on the punches. In short, buffing and polishing dramatically reduce the service life of the punch.

FIGURE 1

Surface profiles across the faces of new and used tableting punches





Wear and corrosion directly decrease tablet quality, reduce tableting productivity, and increase manufacturing costs. While the base metals of tableting tools are ideal for the mechanical demands of tableting, their surfaces can be improved. Engineered coatings, if chosen and applied properly, are an excellent means of improving resistance to wear and corrosion.

TABLE 1

Engineering requirements for tablet punch coatings	
Surface-related properties	Benefits
Non-degraded surface finish in cup	No mechanical interlocking of powder
Increased surface hardness	Improved wear resistance
Good coating adhesion	No flaking or chipping
No pits or voids in the coating	Continuous corrosion barrier
Chemically inert coating	Meets FDA and cGMP guidelines
Bulk-related properties	
Hardness of barrel	No tempering or softening
Overall dimensions	No distortion
Land area	No asymmetric buildup

metal used to make the punch to bolster wear resistance. They must adhere well to the cup surface so that they don't chip or flake. The coatings must be free of pinholes and voids to offer a barrier to corrosion. Finally, the coatings must be chemically inert to satisfy Food and Drug Administration (FDA) and current Good Manufacturing

General requirements for tablet tool coatings

Tableting punches are precision tools, and no coating or coating process can be allowed to compromise their original properties. Table 1 lists engineering requirements for tablet punch coatings. To apply a coating successfully, you must be aware of these requirements.

Surface-related properties. First, coatings must not degrade the original spectral finish on the cup surface. Coatings that roughen the surface, even though they may be hard, will promote mechanical adhesion of powders to the punch. Coatings must be harder than the

Practice (cGMP) requirements.

Properties related to punch construction. The coating process must not reduce the hardness of the punch. High processing temperatures can soften the punch, thus weakening such key surfaces as the land area and domed head. You must also maintain the physical dimensions of the punch during coating. Excess heat can easily distort key dimensions, such as overall length, so that they exceed critical tolerances. Certain plating processes apply coatings non-uniformly, leaving too thick a coating on sharp land edges and on the embossing.

Coating processes

Of the many coating processes in the industrial marketplace, three are commonly used on tablet tooling:

1. Chemical plating
2. Physical vapor deposition (PVD)
3. Ion-beam-enhanced deposition (IBED)

All the available coatings are deposited by one of these three basic processes. Metallic coatings, such as nickel (Ni) and chromium (Cr), are deposited either by electroless electroplating (Ni) or electroplating (Cr). Materials such as boron (B) and Teflon (polytetrafluoroethylene, or PTFE) can be added to Ni to enhance plated coating hardness and coating lubricity, respectively. Processing temperatures during plating are below 200°F, but the coated tool must be baked after plating at higher temperatures (375°F) to prevent hydrogen embrittlement (Cr), or at temperatures between 650°F and 900°F to achieve full hardness (Ni).

Hard refractory nitride coatings include titanium nitride (TiN), chromium nitride (CrN), and diamond-like carbon (DLC). These are usually applied by PVD. Double-layer PVD coatings, consisting of a layer of TiN with CrN overcoat, are also possible. They are known as *superlattice* coatings. To ensure good adhesion and optimum coating properties, the PVD processing temperature must be 900°F or more. Those temperatures will soften the tooling material and distort the precision dimensions of the punch. Fortunately, you can also apply these nitride coatings with a low-temperature IBED process, which coats the tooling at temperatures below 200°F. This eliminates the danger of softening and dimensional distortion of the punch. An IBED coating system is pictured on page 17. Table 2 summarizes the major features of the three coating processes.

Coating structure. The nature of the coating process you select determines the physical structure of the coating, and the physical structure determines such key properties as adhesion and smoothness. Figure 3 shows diagrams of the coating structures obtained with plating,

PVD, and IBED. Electroplated and electroless-plated coatings are precipitates from aqueous solutions and thus grow as individual grains on the surface. The grains nucleate randomly on the surface and grow together, eventually forming a continuous coating, although voids and pits may remain in the coating as it grows. Adhesion is mechanical, thus the punch requires mechanical or chemical surface roughening before plating.

PVD coatings are grown in a vacuum, using a high-temperature, chemically reactive vapor. They grow epitaxially—that is, the grains grow at slightly different orientations and at different rates, depending on the metallurgical composition of the area to be coated. Adhesion is chemical and relies on high-temperature application to form a bond between the coating and the substrate. IBED coatings are also deposited in a vacuum, but the growth is atomistic, meaning that the coating doesn't grow as individual grains, and is nearly amorphous and free of voids. Adhesion occurs by injecting the coating into the surface (the case layer). The coating then grows from the case layer so that there is no distinct interface between the coating and the substrate. Because kinetic energy generates the case layer and forms the coating, the processing temperature remains below 200°F.

Coating surface appearance. Figure 4 shows the appearance of surfaces coated by plating, PVD, and IBED. The coatings were applied to discs of M-series high-speed steel (Rockwell C hardness of 62) that were polished to a spectral finish before being coated. The images are taken from an electron microscope at a magnification of 2,500X. The electroplated Cr was buff-polished after plating, but still shows the pits and voids that form between the individual grains of Cr. This is characteristic of the plating process. The diameter of the pits is 1 micron or less, so after polishing, this Cr-plated surface will not be prone to mechanical interlocking of powders.

The electroless Ni coating shows grains 10 to 15 microns in diameter. After deposition, the coating was infused with PTFE and baked. As you can see in Figure 4, there are small grains of PTFE on the Ni surface. The

TABLE 2

Coating process comparisons

Feature	Plating	Physical vapor deposition (PVD)	Ion-beam-enhanced deposition (IBED)
Pretreatment	Mechanical or chemical roughening	Degreasing and cleaning	Degreasing and cleaning
Temperature	Plating: <300°F Baking: 300°F to 900°F	900°F to 1,200°F	<200°F
Adhesion mechanism	Mechanical	Chemical	Metallurgical
Available coatings	Nickel (Ni), chromium (Cr), nickel boron (NiB), nickel polytetrafluoroethylene (NiPTFE)	Titanium nitride (TiN), chromium nitride (CrN), diamond-like carbon (DLC)	Ni, Cr, TiN, CrN, aluminum oxide (Al ₂ O ₃)
Post-coating polishing	Required	May be required	Not required

FIGURE 3

Growth mechanisms of various coating processes

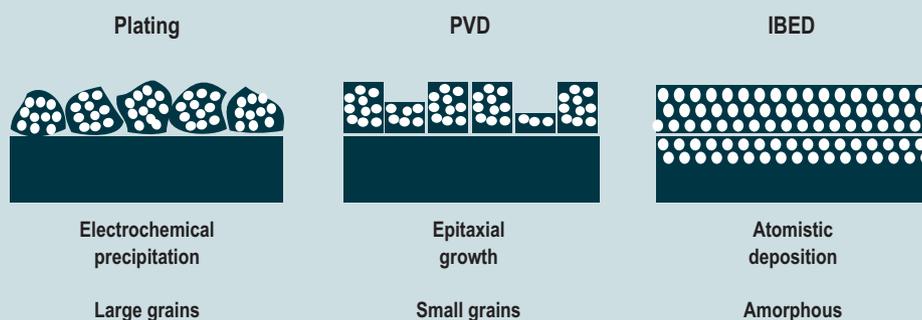
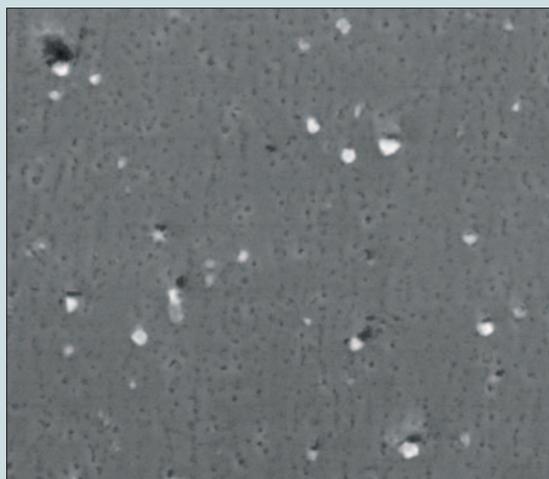


FIGURE 4

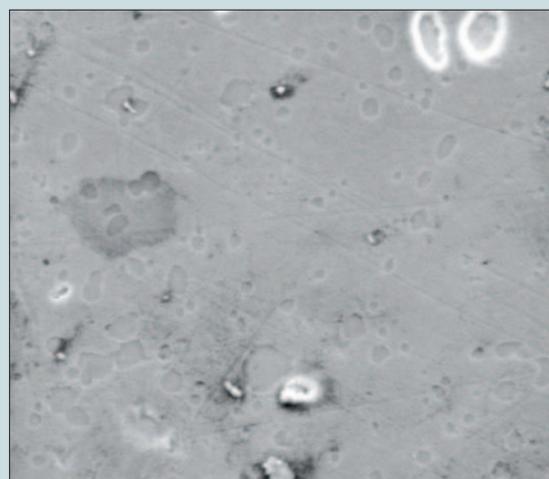
Surface appearances of various coatings magnified 2,500X



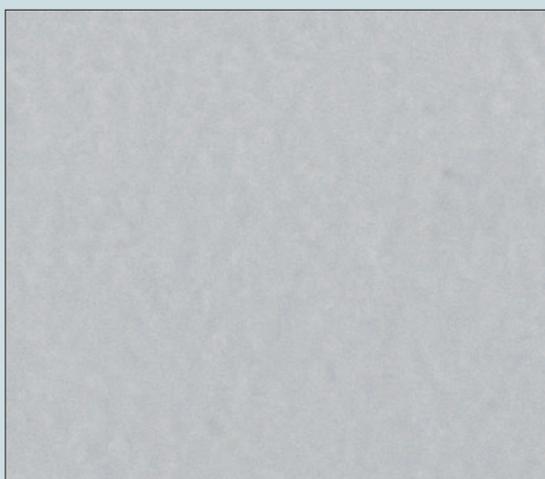
Electroplated chrome



Electroless plated nickel



PVD chromium nitride



IBED chromium nitride

PTFE will act as a lubricant on the surface, but will wear away rapidly, leaving a surface that is significantly rougher than before plating.

The surface of the CrN coating applied by PVD shows the physical appearance of an epitaxially grown coating. The metallurgical composition of the M-series

steel substrate includes alloying constituents of Cr, tungsten (W), molybdenum (Mo), and vanadium (V), along with hard carbide precipitates of these alloying constituents. The CrN coating grows at variable rates on surface regions rich in each of these constituents and results in a coating with an uneven surface. Crater-like structures, ranging from 2 to 8 microns in diameter, appear on the PVD coating surface and are large enough to trap powder particles. The Cr₂N coating applied by IBED appears very uniform, with no grain structure, as is seen with the plated coatings. The surface is very smooth, free of pits, voids, and crater-like regions derived from regional differences in coating growth rate. Here, coating adhesion is optimized, because the interface between the substrate and the coating is indistinguishable.

Coating properties

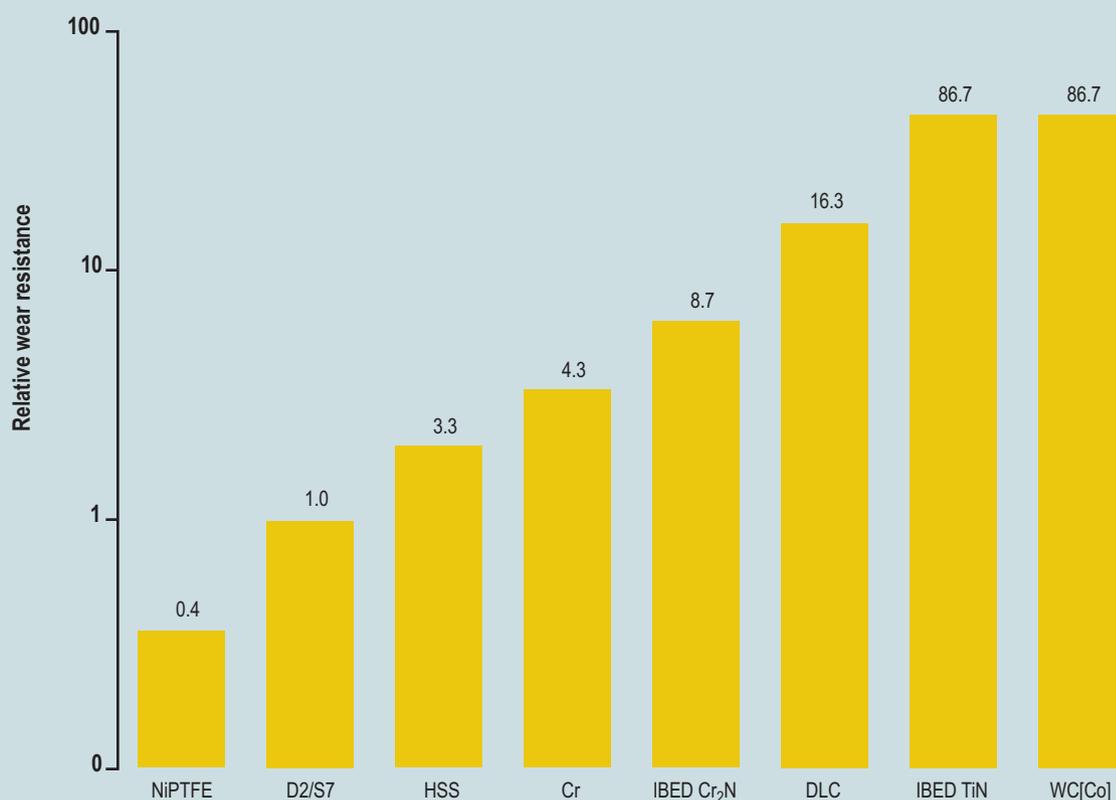
Resistance to abrasive wear is one of the key features required of coatings applied to the working surfaces of

tableting punches. You can get a comparative index of the wear resistance that coatings provide by using a device called the Taber Abraser. The standard procedure (Society of Automotive Engineers/AMS 2438A) is to deposit the coatings on 4-inch circular discs and run the discs against resilient rollers impregnated with 50-micron-diameter, alpha-phase aluminum oxide grit. By weighing the discs before and after running them a fixed number of cycles, you can calculate the thickness of the coating material that is worn away. The inverse of that result is the index of wear resistance. Because test parameters are standard (grit sizes, wheel revolutions per minute, and surface loading), the abrasive wear index numbers that you calculate are directly comparable as measures of abrasive wear resistance.

Figure 5 compares the abrasive wear resistance of electroplated and refractory nitride coatings with that of uncoated, hardened steels typically used for tablet punches (D2 and S7). PTFE-impregnated Ni plating actually wears twice as fast as uncoated, hardened D2 or S7 steels and has

FIGURE 5

Abrasive-wear resistance of various coatings compared with that of uncoated hardened steels used for tablet punches



NiPTFE: Electroless nickel plating with polytetrafluoroethylene impregnation

D2/S7: D2 and S7 tool steels hardened to a Rockwell C scale hardness of approximately 56

HSS: High-speed tool steel hardened to a Rockwell C scale hardness of approximately 65

Cr: Hard chrome plating

IBED Cr₂N: Chromium nitride applied with the IBED process

DLC: Diamond-like carbon applied with the PVD process

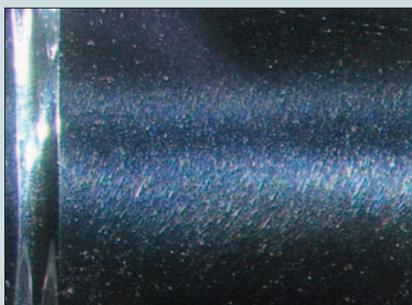
IBED TiN: Titanium nitride applied with the IBED process

WC[Co]: Carbide (also called cobalt-cemented tungsten carbide)

FIGURE 6

Appearances of coated and uncoated punch faces (15X magnification)

Uncoated punch face



Before production cycle

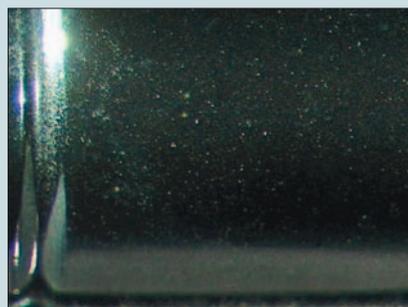


After production cycle

Punch face with CrN coating applied by IBED



Before production cycle



After production cycle

less than half the wear resistance. Cr plating and hardened high-speed steels (Rockwell C 65) show wear resistance approximately two to four times greater than that of D2 or S7 steels. The wear resistance of Cr_2N applied by IBED is approximately eight times that of D2 or S7 steels. DLC coatings applied by PVD show wear resistance 16 times greater than that of uncoated D2 or S7 steels. TiN applied by IBED shows the most resistance to abrasive wear. In this case, wear resistance is equivalent to that measured for cobalt-cemented tungsten carbide ($\text{WC}[\text{Co}]$).

Coated tablet punch example

A Cr_2N coating was applied by IBED to a set of punches used to tablet calcium gluconate, a particularly abrasive and corrosive product. The coating is a chemically inactive ceramic material that resists abrasive wear better than electroplated Ni and Cr. As seen in Figure 6, there is a great difference between the coated and uncoated punch tips before and after a production run. The Cr_2N coating dramatically reduced the abrasive and corrosive attack on the polished punch tip surface. In addition, the improved abrasion and corrosion resistance of the Cr_2N will allow repeated cleaning of the tool without sacrificing the enhanced performance properties.

Conclusion

You can significantly reduce wear and corrosion on tablet tooling by choosing the correct coating and application method. Benefits include better tablet quality and higher manufacturing productivity, both of which fall right to the bottom line as improved profitability. T&C

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