Section 4: Tooling Coatings

Specialized coatings and treatments can be used to enhance the properties and performance of tool steels or to solve specific problems. Coatings normally are applied to tool steels to (1) improve wear resistance to combat abrasion from aggressive granules, (2) improve corrosion resistance, and (3) improve lubricity for sticky products.

Today, a wide range of specialized coatings is available, and new solutions are being developed constantly. Some of the coatings are suitable for the tablet tooling application; however, tooling users must take great care in selecting coatings and seek advice from their tooling manufacturer regarding the suitability of a coating and its interface with the base material of the tooling. Table 14 lists the most common treatments and their function(s).

TABLE 14. FUNCTION(S) OF COMMON STEEL COATINGS AND TREATMENTS

COATING/TREATMENT	IMPROVED WEAR RESISTANCE	IMPROVED CORROSION RESISTANCE	IMPROVED LUBRICITY
HARD CHROMIUM PLATING	√	√	√
PVD TITANIUM NITRIDE (TiN)	√	√	
PVD CHROMIUM NITRIDE (CrN)	√	√	√
ELECTROLESS NICKEL/P.T.F.E.		√	√
IBED (TiN, Cr_2N, Cr, Al_2O_3)	√	√	√

NOTES:
1. ALL TREATMENTS HAVE BEEN TRIED IN CERTAIN APPLICATIONS AND HAVE BEEN SUCCESSFUL IN SOME AREAS. 2. THE PERFORMANCE OF THESE TREATMENTS MAY NOT ALWAYS JUSTIFY THEIR COST. INITIALLY, THE TREATMENTS SHOULD BE APPLIED ON AN EXPERIMENTAL BASIS.
3. CONSULT YOUR TOOLING MANUFACTURER FOR SPECIFIC DATA FOR THESE TREATMENTS.
P.T.F.E. = teflon; IBED = ion beam enhanced deposition.

Coating Processes

Of the many coating processes in the industrial marketplace three have been used for tableting tooling. The processes are chemical plating, physical vapor deposition (PVD), and ion beam enhanced deposition (IBED). All of the various coatings available are deposited by one of these three basic processes. Metallic coatings such as nickel (Ni) and chromium (Cr) are deposited either by electroplating (Cr), or electroless plating (Ni). Materials such as Boron (B), and Teflon (PTFE), can be added to nickel (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 post-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 such as titanium nitride (TiN) and chromium nitride (CrN), and diamond-like carbon (DLC) are usually deposited by physical vapor deposition (PVD) processes. Double layer PVD coatings consisting of a layer of TiN with a CrN overcoating are also possible, and are termed “superlattice” coatings. The PVD processing temperature must be 900°F or above to ensure good adhesion and optimum coating properties. At these temperatures the bulk tool material will soften and precision dimensions will distort. These nitride hardcoatings can also be deposited by the low temperature ion beam enhanced deposition (IBED) process. Coating temperatures do not exceed 200°F, which is low enough to eliminate the danger of dimensional distortion or bulk softening of the punch. Coating adhesion is guaranteed by first forming a layer (termed a case layer) in the surface of the punch, and then growing the coating out from the case layer. The major features of all three coating processes are summarized in Table 15.

TABLE 15. COATING PROCESS COMPARISONS

FEATURE	PLATING	PVD	IBED
PRETREATMENT	MECHANICAL OR CHEMICAL ROUGHENING	DEGREASING & CLEANING	DEGREASING & CLEANING
TEMPERATURE	PLATING: <300°F BAKING: 300-900°F	900-1200°F	<200°F
ADHESION MECHANISM	MECHANICAL	CHEMICAL	METALLURGICAL
COATINGS AVAILABLE	Ni, Cr, Ni(B), Ni(PTFE)	TiN, CrN, DLC	Ni, Cr, TiN, Cr_2N, Al_2O_3
POST-COATING POLISHING	REQUIRED	MAY BE REQUIRED	NOT REQUIRED

General Requirements for Tablet Tool Coatings

Coatings, and coating processes used to apply them, must not compromise the original properties of the punches. Key properties of precision punches include surface-related features and bulk-related features, and are listed in Table 16. All of these features must be maintained and/or optimized if the coating applied is to be successful. .

Surface-Related Properties

First of all, coatings must not degrade the original spectral (shiny, highly reflective**)** finish on the cup surface. Coatings that roughen the surface, even though they may be hard, will act to enhance mechanical adhesion of powders. The coatings must be harder that the bulk material in order to provide enhanced wear-resistance, and they must adhere well to the cup surface so that they do not chip or flake away. The coatings must be free of pinholes or voids in order to act as barriers to corrosion. And the coating material must be chemically inert in order to satisfy FDA current Good Manufacturing Processes (cGMP) requirements.[2]

Bulk-Related Properties

The bulk hardness of the punch must not be affected by the coating process. High processing temperatures can soften the punch, thus weakening key areas such as the land and punch head. The physical dimensions of the punch must also be maintained during coating. Again excess heat can easily distort key dimensions such as overall length so that they exceed critical tolerances. And certain plating processes apply coatings non-uniformly producing excess coating buildup predominantly on sharp land edges and embosses.

TABLE 16. ENGINEERING REQUIREMENTS FOR TABLET PUNCH COATINGS	
FEATURE	**BENEFITS**
SURFACE-RELATED PROPERTIES	
NON-DEGRADED SURFACE FINISH IN CUP	NO MECHANICAL INTERLOCKING OF POWDER
INCREASED SURFACE HARDNESS	IMPROVED WEAR-RESISTANCE
COATING ADHESION	NO FLAKING OR CHIPPING
NO PITS/VOIDS IN THE COATING	CONTINUOUS CORROSION BARRIER
COATING CHEMICALLY INERT	MEETS FDA cGMP GUIDELINES
BULK-RELATED PROPERTIES	
BARREL HARDNESS	NO TEMPERING/SOFTENING
OVERALL DIMENSIONS	NO DISTORTION
LAND	NO ASYMMETRIC BUILDUP

Relative Wear Resistance of Tooling and Coating Materials

Resistance to abrasive wear is one of the key features required of coatings applied to the working surfaces of tableting punches. The wear rates of tool materials and various coatings can be obtained using a Taber Abraser Test. Performed according to a standard procedure (Society of Automotive Engineers/AMS 2438A), coatings are deposited on circular disks that are run against resilient rollers impregnated with fifty-micron diameter hard aluminum oxide grits. The coated disks are weighed, run for a fixed number of cycles, and then reweighed. The thickness of coating material worn away is calculated, and the actual wear rate is obtained. Since standard test parameters are used (grit sizes, wheel RPM, and surface loading), the wear rates measured for the various coatings are directly comparable.

Figure **37** shows the abrasive wear rates measured for electroplated and refractory nitride coatings compared with the abrasive wear rates of uncoated, hardened steels typically used for tablet punches (D2 and S7, Rockwell "C" 56-58). Nickel plating (either co-deposited or impregnated with teflon) actually wears 6× faster than uncoated, hardened D2 or S7 steels. Chromium plating and hardened high speed steels (Rockwell "C" 65) show wear rates approximately 10% to 40% less than that of D2 or S7 steels. The wear rate of Cr_2N deposited by IBED is approximately 3× less than that of S7 steels. DLC coatings deposited by PVD show a wear rates approximately 5× less than that of uncoated D2 or S7 steels. The lowest abrasive wear rate was found with TiN deposited by IBED. In this case the wear rate was less than that measured for cobalt cemented tungsten carbide (WC[Co]).

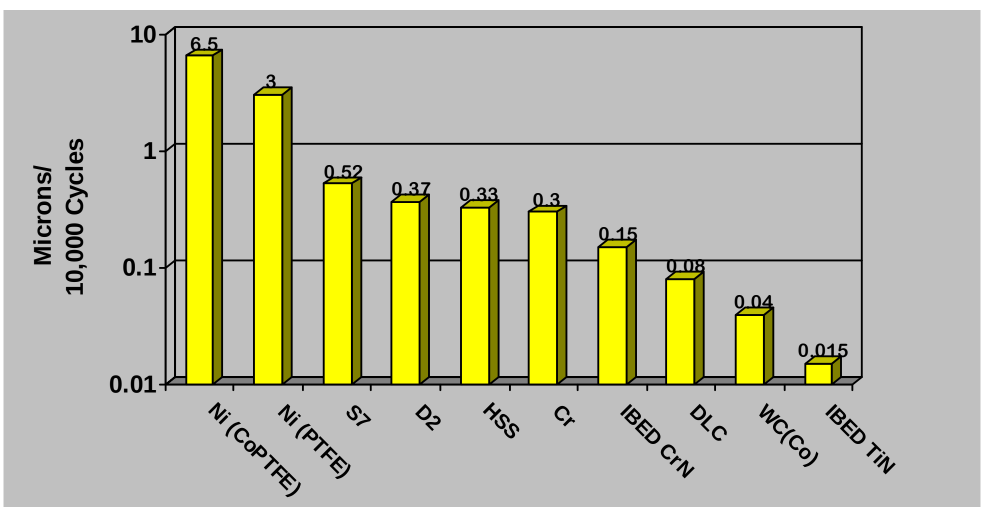

Figure 37. Abrasive Wear Rates of Tooling Materials and Coatings

Ni (coPTPE)	Ni plating with co-deposited PTFE particles
Ni (PTFE)	Ni plating, surface impregnated with PTFE
HSS	Fully hard high-speed tool steel (M-series)
DLC	Diamond-like carbon
WC(Co)	Cobalt cemented tungsten carbide (carbide)

FDA Regulatory Issues

The Food and Drug Administration (FDA) regulates the pharmaceutical and nutritional products manufacturing industry. Regulations governing the manufacture of pharmaceuticals in solid dosage form are found in the "Code of Federal Regulations: Title 21 – Food and Drugs." The regulations specific to equipment used to manufacture solid dosage tablets is found in Part 211 "Current Good Manufacturing Practice for Finished Pharmaceuticals;" Subpart D "Equipment," Section 211.65 "Equipment Construction." [2] As stated in 21CFR211.65:

"(a) Equipment shall be constructed so that surfaces that contact components, in-process materials, or drug products shall not be reactive, additive, or absorptive so as to alter the safety, identity, strength, quality, or purity of the drug product beyond the official or other established requirements."

Thus coatings can be applied to tableting tooling and used in solid dose tablet manufacture as long as the chemical nature of the coating is non-reactive, non-additive, and non-absorptive relative to the formulation being tableted.

Because a coating that solves all problems for all products is not available, new applications normally are used under trial conditions. Your tooling manufacturer can advise and assist in these matters.