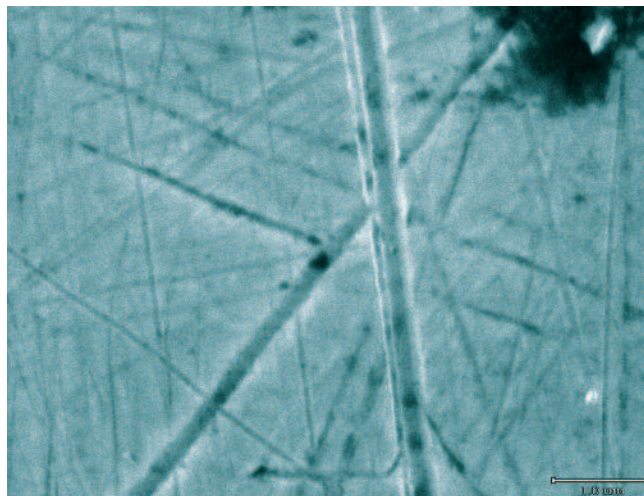


tablet tooling

HOW ROUGH PUNCH-CUP SURFACES PROMOTE STICKING

ARNOLD H. DEUTCHMAN
BEAMALLOY TECHNOLOGIES



Because the cup surfaces of new tablet punches are polished smooth, they don't entrap granules during compression. As the punches are used, however, the cup surfaces exhibit scratches, cracks, pits, or other imperfections, and granules become entrapped and adhere there. This adhesion is a key contributing factor to sticking during tablet compression. This article presents a brief case study that illustrates the problem.

When tableting punches are new, the punch-cup surfaces are highly polished and free of scratches, pits, wear, and corrosion. As the punch cup surfaces wear and corrode during production, cleaning, and polishing, they roughen and those imperfections can entrap powders during compression (Figure 1). As tableting progresses, the cup faces begin to film and, eventually, the entrapped particles become a major contributor to sticking [1-4]. At that point, production must stop so that the punches can be cleaned and polished.

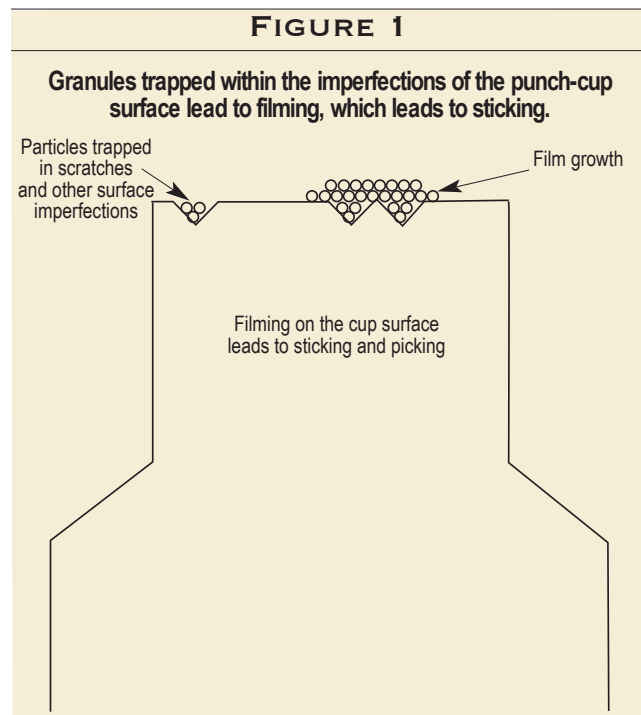
By examining the roughened surface of the punch cups and the powder entrapped there, it's possible to gain insight into the relationship between the punch cup's surface condition and sticking. When granulated powders get trapped in the imperfections of punch-cup surfaces during compression, they become a major cause of sticking [1-4].

Wear resistance, toughness, and surface finish

Many of the active ingredients and excipients in pharmaceutical, nutritional, chemical, and candy granulations are hard and abrasive, as are some of the media used to clean and polish the punch-cup surfaces. These abrasives degrade the original finish on the working surfaces of tablet tools, and the amount of degradation depends on several factors, including the ability of a material to withstand abrasive damage, called wear resistance. In general, the harder the tool material, the more wear resistant it is and thus the less prone it is to scratching, pitting, and surface roughness in general.

The composition and structure of the tool material determines its hardness, which establishes a limit on how wear resistant it can be. Material structure, in turn, is determined by how the material is processed. In the case of a metallic material, hardness can be improved (within certain limits) by how it is heated and cooled during tool manufacture. Hardened tool materials, however, are brittle and therefore subject to fatigue failure (cracking) under cyclic loading, such as during tablet compression. Metals that are not heat-treated to attain maximum hardness are less brittle and

FIGURE 1



therefore resist fatigue. How well a material resists fatigue is called its toughness. Thus the wear-resistant and fatigue-resistant properties of metals are inversely related, and the trade-off between the two is a major consideration when specifying and fabricating tools for specific granulations.

Hardness and wear rates of materials and coatings

Tabletting punches that are manufactured with the correct balance of wear resistance and toughness minimize wear on the cup surfaces while eliminating the danger of fatigue failure during cyclic compression. Since punch fatigue is a more serious failure, punches are most often heat-treated such that they are not as hard as possible, which limits the wear resistance of the cup surface. In those cases, the only way to improve wear resistance of the cup surface is to use coatings, including chromium (chrome) plating and other harder nitride coatings. Table 1 lists the hardness of various steels used to manufacture tabletting tools and the hardness of chrome, a popular tool coating [5].

The wear rates of tool materials and coatings can be measured and compared using the Taber Abraser test, which is a better predictor of wear performance than simply comparing material hardnesses. The test entails rubbing abrasives against the material surface in a mode similar to the abrasive action of tablet compression. Performed according to a standard procedure (Society of Automotive Engineers/AMS 2438A), the test uses resilient rollers impregnated with 50-micron-diameter,

aluminium oxide grits that are run against circular disks of the materials being measured. The disks are weighed, allowed to run for 10,000 cycles, and then re-weighed. The mass of material lost can then be used to calculate the thickness of material worn off, which is expressed as microns per 10,000 cycles.

Because the test parameters—grit size, wheel speed, and surface loading—are standardized, the wear rates of various materials are directly comparable. Figure 2 shows the abrasive wear rates measured for the hardened steels (408, S7 and D2) typically used in tablet punches, as well as for electroplated coatings of nickel and chrome. Nickel plating, it should be noted, actually wears at least five times faster than hardened 408, S7, or D2 steels. Of the hardened steels, D2 wears at the slowest rate. Chrome plating wears at a measurably slower rate than hardened D2 steel and can be used to augment the wear resistance of the working surfaces of tablet punches. Hard coatings like chromium nitride and titanium nitride wear much more slowly than hardened tool steels and even chrome plating.

TABLE 1

Hardness ranges of punch steels and chrome plate (Rockwell C hardness scale)

408 steel	S7 steel	D2 steel	Hard-chrome plate
48 - 56	54 - 58	58 - 60	70 - 72

Case study: Sticking of powders to chrome-plated punches

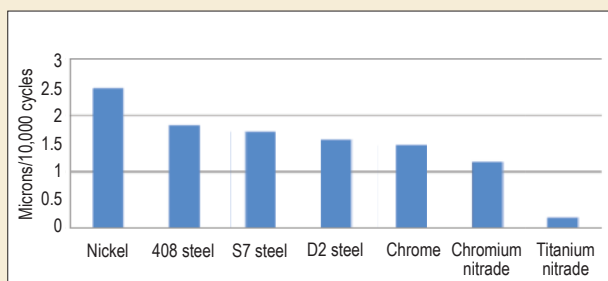
A set of 66 chrome-plated punches (Elizabeth Carbide Die, McKeesport, PA) were obtained, and 33 of the punches were installed in a customer's tablet press. As the press manufactured tablets, the granulation began to stick to the flat cup surface between the bottom of the embossed "500" and the land. Since the customer had a second set of 33 new punches identical to the ones installed, these were installed to replace the sticking punches. The sticking

stopped thereafter and the tableting run was completed. A visual inspection of the cup surfaces of both sets of punches revealed no differences; they appeared the same. Thus an upper punch (#43) that was sticking and an upper punch (#22) that was used lightly and not sticking were sent out for examination and analysis.

Both punches were cleaned in acetone and methanol and dried. The faces of both punches were then imaged using a CamScan Series II scanning electron microscope (Obducat CamScan, Waterbeach, UK) equipped with a Kevex energy-dispersive x-ray spectroscopic (EDS) analyzer (ThermoScientific, Waltham, MA) and running appropriate software (IXRF Systems, Houston, TX). Initial images were taken at a magnification of 30 \times , followed by high-resolution images at a magnification of 4,000 \times . Also subject to EDS analysis were the chrome plating on both punch faces and a particle of material adhered to the face of Punch #43.

FIGURE 2

Abrasive wear rates of punch steels and nickel and chrome plating as measured by Taber Abraser test (SAE/AMS 2438A)



Results

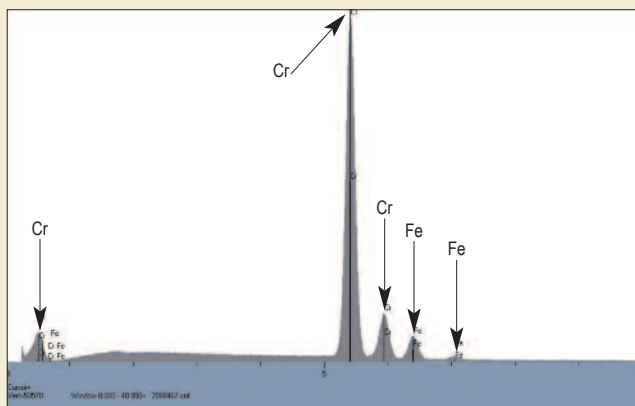
Images of the punch faces, taken in the secondary electron mode at a magnification of 30 \times , appear in figures 3a and 3b. The flat cup surface between the embossed "500" and the land on both punches was imaged. At this magnification, no structure is resolved in

FIGURE 3

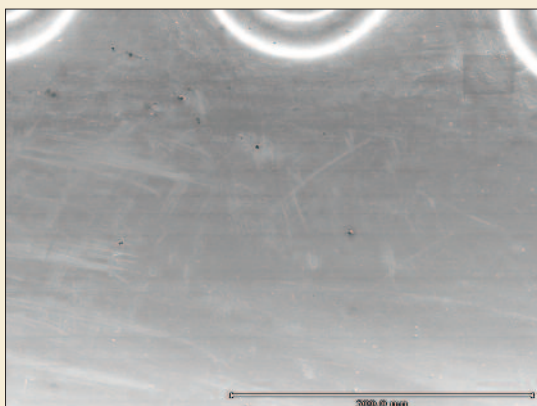
Images from a scanning electron microscope show the plated surfaces of Punch #22 and Punch #43 (30 \times magnification). Also shown are the energy-dispersive x-ray spectra (EDS) of each that confirm they're chrome-plated.



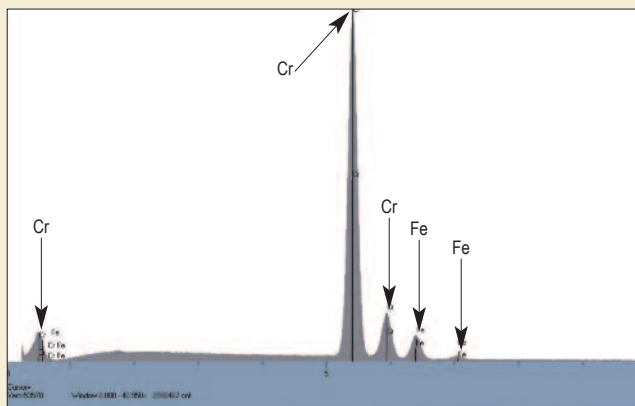
a. Punch #22 (new, chrome-plated)



c. EDS of Punch #22's chrome plating.



b. Punch #43 (used, chrome-plated)



d. EDS of Punch #43's chrome plating.

the chrome-plated layer, and the surface structure and smoothness of both punch faces appear equivalent. The raised dimples seen on the surface of both punch faces are large grains of chrome that were not polished down completely. The light smudges seen on the surface of Punch #43 are due to scratch fields on the surface of the chrome plating.

An EDS analysis was also conducted on the chrome plating of both punches and the results appear in figures 3c and 3d. The x-ray spectra from the chrome plating on both punch faces are equivalent except for

the presence of a higher magnitude iron (Fe) signal on Punch #43. The appearance of the iron signal from the punch steel is the result of thinning chrome plating on its surface, possibly due to polishing.

Higher-resolution images of the punch faces are seen at a magnification of 4,000× in figures 4a and 4b. There is a background of randomly oriented wide but shallow sub-micron scratches on the surfaces of both punches #22

and #43. The individual scratches are on the order of 0.05 micron wide and are normal features of a highly polished metallic surface. On Punch #43, however, there is a field of 0.33-micron-wide, deeper scratches randomly superimposed on the field of extremely fine scratches. These scratches are six times wider and deeper than the shallow polishing scratches and are very jagged at the top surface.

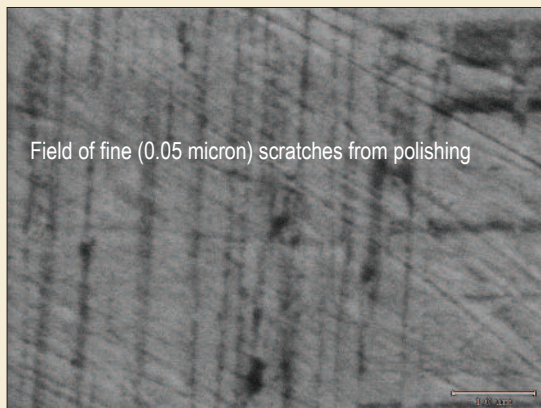
Particles of material were found trapped at various locations in the deep scratches on Punch #43, and an EDS spectrum of one of the particles (which includes

the surface below it) was taken and is presented in Figure 4d. Many elements were detected in the particle, including sodium (Na), calcium (Ca), potassium (K), sulfur (S), chlorine (Cl), phosphorous (P), carbon (C), and silicon (Si). These elements were not deemed contaminants in the chrome plating. Rather, they emanated from a particle of the granulation that lodged in the wide scratches in the chrome plating.

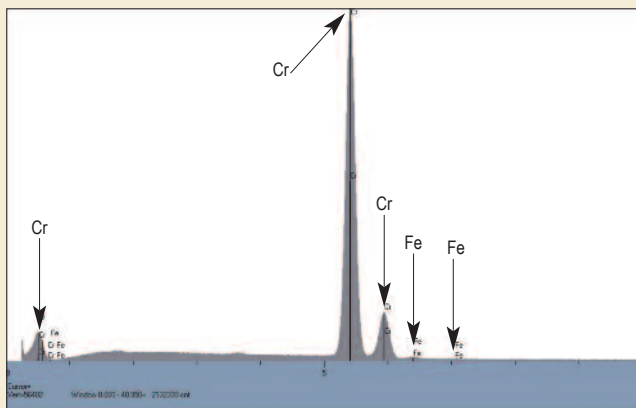
In many cases, the only way to improve wear resistance of the cup surface is to coat it.

FIGURE 4

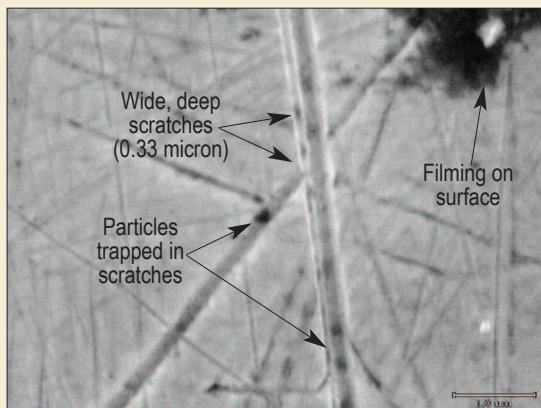
SEM images of the plated surface of punches #22 and #43 (4,000× magnification), as well EDS confirming chrome plating. The EDS of the contaminant particle (which includes the surface below it) shows the presence of chrome, iron, sodium, calcium, potassium, sulfur, chlorine, phosphorous, carbon, and silicon.



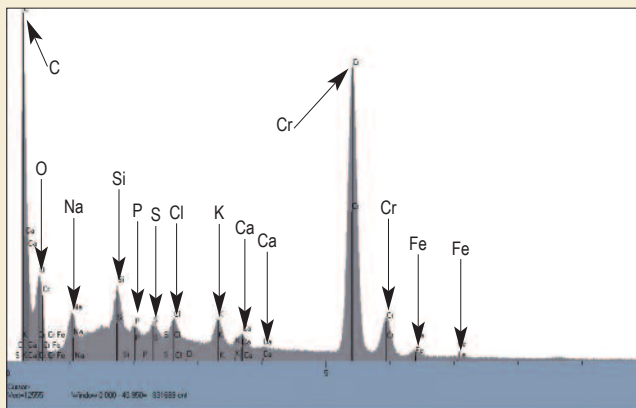
a. Punch #22 (new, chrome-plated)



c. EDS of Punch #22's chrome plating.



b. Punch #43 (used, chrome-plated)



d. EDS of Punch #43's chrome plating.

Conclusion

The surfaces of highly polished, unused chrome-plated tablet punches are shown to have a uniform background field of 0.05-micron-wide scratches, remnants of the initial polishing process. These scratches are narrow and shallow and do not promote entrapment of the granulation's particles, thus preventing sticking and picking. As the polished surfaces wear, wider, deeper scratches appear in the chrome-plated surface. As a result, particles of the granulation become trapped in the surface of the chrome plating, leading to filming and sticking.

Recommendations

In order to reduce (and possibly) eliminate punch filming and tablet sticking due to entrapment of granulation particles, the surface finish of the punch faces must start without—and remain free of—scratches that exceed 0.05 micron in width and depth. Although the addition of chrome plating—which has better wear resistance than the punch steel—slowed the appearance of deep scratches in the cup surface, it, too, eventually developed scratches due to abrasive wear, which led to the onset of filming and sticking. Other approaches include applying coatings that resist abrasive wear better, such as those containing chromium nitride and titanium nitride. T&C

References

1. A.H. Deutchman and D. Natoli, "Tableting punch performance can be improved with precision coatings," *Pharmaceutical Processing*, September 2002, p. 16.
2. M. Roberts et al., "Effects of surface roughness and chrome plating of punch tips on the sticking tendencies of model ibuprofen formulations," *Journal of Pharmacy and Pharmacology*, Vol. 55, 2003, pp. 1223-1228.
3. A.H. Deutchman, "Choosing coatings to improve the performance of tablet tooling," *Tablets & Capsules*, Vol. 2, No. 5, September 2004, p. 17.
4. A.H. Gerhardt, "Compression tooling surface treatments – Options for resolving corrosion, wear, filming, and tablet defects," *GXP Compliance*, Vol. 13, No. 3, summer 2009, p. 68.
5. "Tool steels, compression forces, and fatigue failure," *Tableting Specification Manual*, 7th Edition, L.I. Young, ed., American Pharmacists Association, Washington, DC, 2006, p. 81.

Arnold H. Deutchman is chairman and director of R&D at Beamalloy Technologies, 8270 Estates Parkway, Plain City, OH 43064. Tel. 614 873 4529, x114, fax 614 873 0167. E-mail: adeutchman@beamalloy.net. Deutchman received a BS in physics from the Ohio University in Athens and an MS and a PhD in nuclear/mechanical engineering from the Ohio State University in Columbus.