Sticking is often described as the Achilles’ heel of tablet production. It is one of the most common tablet tooling problems and can have a profound effect on production, leading to reduced output and increased costs. This article explains why sticking occurs and proposes several solutions.

Sticking is one of the most common problems tablet manufacturers encounter. Sticking describes the buildup of granules on the punch-tip face, which can be caused by several factors, including the formulation’s physicochemical properties and the surface characteristics of the punch face. Because sticking is such a universal problem for tablet manufacturers, tablet tooling experts have extensively studied its causes and how to prevent it.
Adhesive forces cause tablets to stick to the punch faces after compression. Adhesive force is affected by the surrounding environment, the chemistry at the interface between the formulation and the punch, the formulation’s spacial heterogeneity, the formulation’s deformation mechanics, and the tooling’s surface characteristics.

**Environment.** The environment around the tableting process and even around the formulation preparation process are critical. Temperature has a huge influence on some active pharmaceutical ingredients (APIs); ibuprofen, for example, has a very low melting point. Lowering the temperature in the tableting area can help reduce sticking during compaction.

Excess humidity in and around the tablet press or formulation processing equipment or in the area where the formulation is stored prior to tableting can also introduce capillary bridging between the granules and the tablet tooling, significantly increasing the chance of sticking.

During the TSAR project, researchers used a sessile droplet test to measure the influence of capillary forces for each individual surface. This test determines the static contact angle between a liquid (in this case, water) and a solid surface and should always be used when developing anti-stick coatings or surfaces for tablet tooling because it establishes the surface’s level of hydrophobicity or hydrophilicity. Hydrophobic surfaces will have a static contact angle greater than 90 degrees, while hydrophilic surfaces will have an angle less than 90 degrees.

Figure 2 shows the results of the sessile droplet test for different steels and coated surfaces. The yellow columns represent three pharmaceutical-grade steels (HPG-S, HPG-P, and HPG-SS), which have varying contact angles because of different compositions and polishes. The red columns represent tooling coated with two types of hard-chromium (HC and HC+). Hard-chromium-based coatings have been used in tableting for many years to decrease sticking by creating a less hydrophilic surface, but they are less common today due to the carcinogenic waste produced during manufacture. Also, when hard

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**Cohesive forces**

Cohesive forces hold a tablet together and are largely affected by three elements: Van der Waals forces, capillary action, and electrostatic force.

**Van der Waals forces.** Van der Waals forces are attractive forces between molecules. They act over a short range but, in mass, combine to increase the cohesive force within a tablet and can also cause sticking. They are relatively weak and, during tablet compression, are affected by the surface roughness and effective contact area of the punch.

**Capillary action.** Capillary action can be linked to tablet formulations with high moisture content and is generated when moisture condenses in the gap between particles or between a particle and a surface to create a liquid bridge. The strength of this cohesive force can depend on the relative humidity, gap geometry, and surface chemical condition. Capillary bridges increase the cohesive forces that help bind a tablet together. However, they can also increase the adhesion forces between the granules and the punch tip face, leading to sticking.

**Electrostatic forces.** Static electricity is a major cause of sticking and often occurs when tableting dry granules. Electrostatic forces can also arise when tribo-charging (a transfer of electrical charges) occurs between contacting materials. The resulting force can be relatively strong and long ranged, with the potential to create both cohesive and adhesive forces. Manufacturing equipment can also influence the presence and strength of the electrostatic force. To minimize electrostatic force generation and protect press operators from shock, tablet presses are typically connected to a grounding cable. Non-conductive tool coatings or treatments can also help to reduce electrostatic force generation.
chromium is applied to tooling, hydrogen penetrates the steel making it more brittle and prone to unpredictable fracture, decreasing the working load by 20 percent. The green columns represent more advanced coatings (ECxtra, CN, CN+, and CT), which have been developed in recent years to decrease sticking without causing hydrogen embrittlement and which are rapidly replacing the low tech hard-chromium-type coatings. The orange bar represents hard diamond-like carbon coating (DN).

Identifying its elements and also removing very small layers of the coating and the steel underneath, this method can examine any interaction between the tool and formulation in a localized area of the punch tip.

AFM is also used to analyze interface chemistry and spatial heterogeneity to develop anti-stick coatings. This technique can provide nanoscale imaging of surfaces and has been used to directly assess particle-to-particle and particle-to-surface interactions [1, 2].

Figure 4 illustrates the stages of the AFM experiment. The blue line represents the contact stage. The black line moving from right to left represents the movement of the stylus as it approaches the surface and presses the particle into place. The red line shows the force required to pull the particle away, indicating the interactive force between the granule and the surface. If the API is the root cause of the sticking, particularly if it is concentrated in one area of the punch tip, the problem can be resolved by selecting a coating with a specific chemistry to overcome the elemental changes.

Deformation mechanics. The deformation mechanics, or the physical properties of the granules being compressed, can be either plastic (stays deformed) or elastic (springs back to its original shape) in nature. For formulations with more elastic, time-dependent deformation behavior, a long dwell time is critical to creating strong bonds between the particles. When a manufacturing situation requires a greater dwell time, using punches with an extended head flat will enable a suitable compression dwell time for an elastic formulation without slowing the press through sticking.

Tablet tooling surface. The morphology (surface roughness) of the punch tip face is critical to stop sticking. A common misconception among many tooling suppliers is that polishing the punch tip faces to a mirror finish will prevent sticking. However, studies of the interaction between different surface textures have established that the standard specification for tablet punch tip faces can include a range of surfaces that interact with formulations in different ways.
The required standard surface for a punch tip is between 0.1 to 0.025 roughness average (Ra). Figure 5 shows two different surfaces within the Ra parameters. On the left is a surface coated with PharmaCote CN and on the right is a surface coated and processed using CN+, a proprietary method that modifies the coating surface to reduce formulation sticking to the punch face. The peak-to-valley height is the same on the CN surface as it is on the CN+ surface. However, when looking at the peak-to-peak value, the surface textures differ dramatically.

To eliminate sticking, manufacturers must analyze the effect of the interaction between the granule and the surface finish. There are several methods they can employ, including optical surface profilometry, which measures the surface roughness after polishing; scanning electron microscopy, which studies the structure of the steels and the coatings; and, as previously mentioned, AFM, which measures adhesion forces and can be used to create a nanoscale map of the surface.

Table 1 shows the results of a case study that tested different coatings on compression tooling used to tablet ibuprofen. The tooling was placed in a tablet press with 39 stations and run at three different speeds with a main compression force of 17 kilonewtons and a pre-compression force unchanged throughout the test. Five tooling samples were tested, including coated and uncoated stainless-steel tooling manufactured by a competitor and three PharmaCote-treated tooling samples. The competitor’s stainless-steel and coated tooling created severe sticking issues while the CN+ and ECxtra (E-Chrome) samples presented only partial sticking. The CT-textured chromium-nitrate coating, which does not have a mirror finish and would once not have been used due to its Ra value, resolved all the sticking issues during ibuprofen tabletting.

No single solution

Sticking is detrimental to tablet production and costly, as it inevitably results in downtime and reduced productivity. To mass produce quality tablets, you must first resolve any sticking issues. Because the physical properties of each sticky formulation are unique, there is no one-size-fits-all, anti-stick solution. Applying a predictive tool such as TSAR that analyzes several sticking causes and suggests a suitable punch or die coating solution can save time and money compared to slow and costly on- or off-site compression trials.

References