Lubricious Thin-Film Coatings for Forging Applications: A Literature Review

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Abstract

One of the industry-wide objectives identified by the Forging Industry Association (FIA) is the reduction of lubricant use during forging. Toward that goal, FIERF funded a research effort at the Colorado School of Mines (CSM) to test permanent PVD coatings that may minimize lubricant use during forging. The initial focus of the project is to develop a laboratory test to quantitatively measure the impact of permanent PVD coatings on the coefficient of friction between aluminum or steel workpieces and steel tooling.

The first stage of the project has been to perform a literature review to understand the state-of-the-art in lubricious PVD coatings. Table 1 gives an overview of the findings of this literature review, and lists the various types of liquid and solid lubricants available for forging dies, including lubricating mechanisms and temperature limits of various types of lubricants.

Table 1: Summary of current lubricants and potential coatings for forging dies.

<table>
<thead>
<tr>
<th>Lubricants and Mechanisms</th>
<th>Material Examples</th>
<th>Temperature Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Traditional liquid lubricants</td>
<td>Oils and greases</td>
<td>&lt;250°C</td>
</tr>
<tr>
<td>Atomic structures that can easily shear</td>
<td>MoS₂, graphite</td>
<td>&lt;300°C</td>
</tr>
<tr>
<td>PVD hard coatings (can contain softer lubricating phases)</td>
<td>CrN, AlCrN, TiAIN, TiCN, diamond-like-carbon, i-Kote</td>
<td>&lt;500-800°C depending upon coating</td>
</tr>
<tr>
<td>Soft metals within hard coatings that diffuse to contact surfaces</td>
<td>Ag, Au or Cu encapsulated in hard coatings such as TiN, CrN, VN, YSZ, CrAIN</td>
<td>300 - 500°C</td>
</tr>
<tr>
<td>Lubricious oxides</td>
<td>V₂O₅, MoO₃</td>
<td>500 - 1000°C</td>
</tr>
</tbody>
</table>
1. Introduction

FIERF has recently funded a research effort at the Colorado School of Mines (CSM) to perform laboratory testing to identify permanent PVD coatings to minimize lubricant use during forging. The initial focus of the project is to develop a laboratory test to quantitatively measure the impact of PVD permanent coatings on the coefficient of friction between steel tooling and the workpiece (aluminum or steel). The overall project goal is to reduce the amount of lubricant used during forging, an industry-wide objective identified in two documents published by the forging industry, the Forging Industry Technology Roadmap and FIA’s Vision of the Future(1,2).

The initial phase of this project is to perform a literature review to identify previous research on the use of PVD thin-film coatings on forging dies and the use of thin-film coatings for lubrication applications at elevated temperatures, such as would be experienced by the tool during the forging of aluminum or steel.

This project follows on from a successful effort at CSM to identify permanent PVD coatings to minimize lubricant use in aluminum die casting(3-6). Although the reasons are different for applying lubricants in die casting (where the “lubricant” essentially acts as a parting agent, to minimize soldering and alloying between the solidifying aluminum and the steel die), it is anticipated that this current forging project can leverage the previous project performed with the die casting industry.

This literature review initially briefly describes surface engineering technologies that could be used to meet the projected goal of reducing conventional lubrication during warm and hot forging, and then summarizes previous research on the use of thin-film technologies to reduce friction and wear in forging and other wear-sensitive applications. Some excerpts from literature reports are included.

2. Background

Forging is the working of metal into a useful shape by hammering or pressing(7). The metal is shaped using large compressive forces, so the forging tools are generally fabricated from high strength steels. The types of hot work tool steels listed in Table 2 are generally used for the warm and hot forging of aluminum-alloys, copper-alloys and steels. Forging dies are typically heat treated by austenitizing, quenching and double tempering, as shown in Figure 1, to provide a Rockwell C scale (HRC) hardness in the range of 44-60(9). Optimizing steel hardness for a given forging application may be important to provide a balance between wear resistance and toughness.
Table 2: Composition and typical hardness ranges of some commercially available hot work die steels (13)

<table>
<thead>
<tr>
<th>Supplier</th>
<th>Material</th>
<th>Composition (wt%)</th>
<th>Recommended Initial Hardness Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bohler</td>
<td>W360 0.5</td>
<td>0.2 0.25 4.5 3</td>
<td>52-57 in oil or salt bath</td>
</tr>
<tr>
<td></td>
<td>W302 0.39</td>
<td>1.1 0.4 5.2 1.4</td>
<td>50-54 in air</td>
</tr>
<tr>
<td></td>
<td>W303 0.38</td>
<td>0.4 0.4 5.0 2.8</td>
<td>52-56 in oil or salt bath</td>
</tr>
<tr>
<td>Uddeholm</td>
<td>QRO90 Supreme 0.38</td>
<td>0.3 0.75 2.6</td>
<td>48-50 in air</td>
</tr>
<tr>
<td></td>
<td>Unimax 0.5</td>
<td>0.2 0.5 5.0 2.3</td>
<td>50-54 in air</td>
</tr>
<tr>
<td></td>
<td>DIEVAR 0.35</td>
<td>0.2 0.6 5.0 2.3</td>
<td>44-46 in air</td>
</tr>
<tr>
<td></td>
<td>ORVAR 2M 0.39</td>
<td>1.0 0.4 5.3 1.3</td>
<td>44-50 in air</td>
</tr>
<tr>
<td>Daido</td>
<td>DRM1 0.6</td>
<td>NA NA 4.2 --</td>
<td>58 max</td>
</tr>
<tr>
<td>Nachi</td>
<td>DURO F1 NA NA NA NA NA</td>
<td>54-60</td>
<td></td>
</tr>
</tbody>
</table>

Figure 1: Typical heat treatment used for hot worked die steels used for forging tools.

Die costs can range from 8-to-30% of the total part cost for hot or warm forging\(^{(10,11)}\), so maximizing the life of forging tools is crucial for minimizing production costs. Bayramoglu et al.\(^{(12)}\) noted there are a number of parameters that impact die life during forging, including die heat treatment, wear resistance, surface condition, cavity shape, alignment, surface stress distribution during the forging operation, and surface treatments.
The main failure mechanisms that compel replacement of a forging die are shown schematically in Figure 2, and include wear (both adhesive and abrasive), plastic deformation, and fatigue (both mechanical and thermally induced)\(^ {11,13}\). Note that failure mechanisms vary with location in the die and with die geometry. Gronostajski et al.\(^ {11}\) provided statistics detailing reasons for tool retirement, shown in Table 3, which suggest that loss of dimensional accuracy due to abrasive wear is by far the most common.

**Figure 2:** Common failure mechanisms and locations for forging dies, including (1) abrasive wear, (2) thermal fatigue, (3) mechanical fatigue, and (4) plastic deformation\(^ {8}\).

**Table 3:** Reasons for forging tooling retirement\(^ {11}\).

<table>
<thead>
<tr>
<th>Reason</th>
<th>% of Tools</th>
</tr>
</thead>
<tbody>
<tr>
<td>Loss of dimensional accuracy due to abrasive wear</td>
<td>70%</td>
</tr>
<tr>
<td>Plastic deformation</td>
<td>25%</td>
</tr>
<tr>
<td>Fatigue cracking</td>
<td>5%</td>
</tr>
</tbody>
</table>

Images of typical wear conditions on the surface of the forging dies has been provided by Yilkiran et al.\(^ {14}\), and are reproduced in Figure 3 to show the differences between cracking, pitting, adhesion, and abrasion in the morphology of surfaces that interact with the component being forged.
3. Conventional Lubricants Used During Forging

Excellent summaries of the types and performance of conventional lubricants used in the forging industry have been provided by Wiederholt\(^\text{(15)}\) and Clarke and Van Tyne\(^\text{(16)}\). Conventional lubricants used in forging include oil-based graphite, oil-in-water emulsion graphite, water-based graphite and synthenotics, and these are needed to optimize part quality, increase tool life, and increase productivity. There are a number of lubrication mechanisms occurring during the forging process including solid film lubrication, fluid film lubrication, vapor barrier lubrication and boundary layer lubrication. During a forging operation lubricants can degrade, physically move away from locations where lubrication is necessary (moved by the deforming forging blank or not in the desired location by inconsistent initial placement), or occupy volumes into which metal is intended to flow\(^\text{(16)}\). Thus, minimization of the amount of lubricant required during a forging operation may improve process control and repeatability.
4. **Surface Engineering Technologies**

Surface engineering of die materials is becoming more commonly used across a wide range of metals processing technologies, as it imparts characteristics to the surface of materials that are otherwise unachievable. For metals processing methods such as forging, rolling, machining, extrusion, and die casting, there are three types of surface engineering technologies commonly used: conventional surface treatments, hard coatings, and weld overlays. Different types of technologies within these three broad groups are listed in Figure 4\(^{(17)}\).

![Figure 4: Classifications of surface engineering techniques (Modified from Ref. 17).](image)

The technologies shown in Figure 4 are briefly summarized in the following sections (4.1-4.3) of this report, which are primarily extracted from refs. (17) and (18).

4.1 **Traditional Surface Treatments**

As shown in Figure 4, surface treatments applied to steels for forming tools include nitriding and ferritic nitrocarburizing. During nitriding, atomic N is diffused into the surface of the steel to create a hard surface layer. The N then combines with elements in the surface layer, producing hard nitride compounds. Nitriding is typically performed at
temperatures between roughly 500 and 550°C (~925 and 1025°F), below the tempering temperature of hot worked steels, allowing the surface of the die to be nitrided (hardened) without compromising the overall bulk hardness of the tool.

Ferritic nitrocarburizing is a surface hardening process for steels that involves diffusing both C and N into the steel’s surface to provide a hard, wear-resistant surface. In general, ferritic nitrocarburizing is performed below 675°C (1250°F), but for the types of hot working steels used for forging, it should be performed at or below ~550°C (1025°F), to avoid over-tempering. With respect to forging, Altan and Shirgaokar\(^{(10)}\) noted that conventional surface treatments such as nitriding provide a significant increase in surface hardness but have not been found to provide substantial improvements in die life.

4.2 Hard Thin-Film Coatings

Figure 5 shows typical application temperatures and coating thicknesses for a range of hard coating processes. For the application of coatings to forming tools for processes such as forging, coating thicknesses are typically kept to 10 \(\mu\)m or less, to avoid spalling of the coating due to the severe loads experienced during the forging operation. In addition, the temperature of the substrate (the forging tool) needs to be maintained below 550°C (1000°F) or so during application, again to avoid over-tempering. As shown in Figure 5, the thin-film coating process physical vapor deposition (PVD) meets these criteria, and is most commonly used to coat forging tools.

![Figure 5: Ranges of processing temperatures and achievable coating thicknesses for various thin-film coatings\(^{(18)}\).](image-url)
PVD is an atomistic deposition process, where atoms of the material to be deposited are physically vaporized normally from a solid source. Coatings produced via PVD can be single elements (such as Ti or Cr), alloys (such as Ti-Cr), or compounds (TiN, CrN, CrC, AlCrN). In addition, multilayer or graded compositional coatings can also be produced. Figure 6 shows a 5.1 μm thick CrN coating deposited using a PVD process onto a steel substrate.

![Figure 6: Secondary electron micrograph of a cross-section of a 5.1 μm thick CrN thin-film coating on a steel substrate. Coating was produced by PVD (courtesy of Phygen Coatings).](image)

The two most common PVD techniques used to deposit wear resistant coatings for forming dies are cathodic arc evaporation (CAE) and sputtering. In both cases, metal ions (e.g., Cr) are released from a solid source called a target and these ions combine with gaseous atoms within a vacuum chamber (e.g., N), to form thin-film compounds (CrN) on the surface of the dies (called a substrate).

Sputtering takes place within a plasma environment. A plasma is one of the four fundamental states of matter (the others being solid, liquid, and gas). The formation of a plasma decreases or increases the number of electrons in the atoms of a gas within the reaction chamber, creating positive or negative charged particles called ions. However, only a small number of the atoms present are ionized (about 0.01%), so there are still more neutral atoms than ions. Within the plasma, the number of ions and electrons are approximately equal, so the plasma has an overall charge neutrality.

The sputtering process uses a “glow discharge”, which is a plasma formed by the passage of electric current through a low-pressure gas created by applying a voltage between an
anode and a cathode within a vacuum container. When the voltage exceeds a certain value, called the striking voltage, the gas in the tube ionizes, becoming a plasma, and the ionized gas can now conduct electricity (Figure 7). This concept is used in fluorescent lights.

For the sputtering process, a small amount of Ar is introduced into the vacuum chamber, and Ar+ ions are generated by the plasma (Figure 8). The Ar+ ions are attracted to the cathode (target), hitting the target at high energy, which causes ejection of atoms from the target. Atoms ejected from the target metal can combine with reactive gasses in the plasma (such as N, C or O) to produce nitrides, carbide or oxides, etc., and these ceramic compounds are deposited onto the substrate (the forging die) to produce the coating.
The advantages of sputtering include the ability to produce very smooth coatings and the ability to generate coatings from most solid materials. The main disadvantage is that the overall level of ionization is low for the sputtering process, especially when compared to cathodic arc, and so the properties of the coatings can be less favorable, especially for mechanical applications.

With the cathodic arc process, a high current density travels along the surface of the cathode target, causing the evaporation of metallic ions from the target. Again, these ions can combine with gasses present within the reaction chamber, and these compounds are deposited onto the substrate (the forging die), to produce the thin-film coating. For the cathodic arc process, the targets must be electrically conductive (normally a metal), to allow the production of the arc.

One of the main disadvantages of the cathodic arc process is the generation of macro-particles, which are molten metal droplets about 1-5 μm in size that are ejected from the target and can be deposited in the coating. As these macro-particles are about the same size as the film thickness, they create regions of weakness within the coatings (Figure 9). One solution used in industry is the use of filtered arcs, which utilize magnetic fields to steer the plasma through a curved duct (Figure 10). The macro-particles are relatively large, so they are not influenced by magnetic field, and so are not deposited on the substrate.
Figure 9: Macro-particles deposited into a thin-film coating\textsuperscript{(19)}.

Figure 10: Schematic of the Filter Arc process used to minimize macro-particle deposition for the cathodic arc process\textsuperscript{(17,18)}.

One major disadvantage of both sputtering and cathodic arc is they are line-of-sight processes, meaning that deposition only occurs on the side of the substrate facing the
target. Therefore, to uniformly coat a cylindrical component such as a pin, the pin has to be rotated in front of the target.

As shown schematically in Figure 11, the chemical vapor deposition (CVD) process uses volatile gases for the production of the thin coatings. The substrate is heated to a high temperature, causing breakdown of the gasses within the chamber, producing the chemical species that deposit onto the substrate.

![Figure 11: Schematic of the CVD process](image)

There are a number of advantages of the CVD process, including the ability to coat complex shapes (not a line-of-sight process), the ability to produce uniformly thick coatings over large areas, and to control coating composition by gas premixing.

However, as discussed earlier, there are disadvantages that preclude CVD from being used for coatings for forging dies. The main disadvantage is that it requires the use of a high temperature to decompose the gasses used to produce the coatings. Such high temperatures limit the use of some substrates such as hot worked steels, as they will over-temper and soften.

Plasma Enhanced Chemical Vapor Deposition (PECVD) is shown schematically in Figure 12 and utilizes the energy of charged particles in a plasma to reduce the temperature required for the decomposition of the gas. This allows the production of coatings at much lower temperatures than for thermal CVD, and so PECVD can be used for forging tools.
Finally, it’s worth mentioning the recent development of multi-layer PVD hard coatings for metal forming tools. As shown schematically in Figure 13, the interfaces present in multi-layer coatings can assist in crack deflection to improve the cracking resistance of these relatively brittle ceramic coatings. For example, if a single layer coating fractures, the crack runs from the surface to the steel substrate below allowing premature failure of the coating. With multi-layer coating architectures, however, the interface between the coating layers can provide a barrier to crack propagation, thereby increasing the toughness of the coating, potentially extending its life.

Moore and co-workers\textsuperscript{(20,21)} recently described the function and purpose of each layer in multi-layer coating architectures that provide a transition from the base die material to the working surface, as summarized below in sequential order:

1. **Modification of the die surface** – The die surface should be hardened by nitriding or ferritic nitrocarburizing to produce a hard and corrosion resistant surface with increased mechanical support. Typical nitriding thickness is about 150 µm. Without
good load support, an “eggshell effect” can be created, such that when the shell (the coating) is loaded, cracking and delamination can readily occur.

2. **Adhesion layer** – A thin metallic layer between the steel substrate and the ceramic coating, typically about 50 to 100 nm in thickness, which enhances the adhesion of the ceramic thin-film coating to the substrate, and also helps to accommodate coefficient of thermal expansion mismatch between the steel substrate and the ceramic coating. Typically, adhesion layers are produced from refractory or near-refractory metals (usually not nitrides) such as Ti, Cr, or W.

3. **Intermediate layers** – The role of the intermediate layer is to provide accommodation of the stresses either inherent in the deposition process, or that develop during thermal cycling of the forging dies. Typical intermediate layer materials include nitrides, carbides, and borides of metals such as Ti, Cr, Al or V. Intermediate layers can be single material, graded structures to provide accommodation of structural differences between the adhesion layer and the working layer, or multi-layered structures.

4. **Working layer** - This layer will be directly exposed to the workpiece, where the environment tends to be most severe. Critical characteristics of the working layer for forging applications include excellent wear resistance, good corrosion and oxidation resistance, and excellent lubricity. The total thickness of the intermediate and working layers should be around 2-5 µm.

![Figure 14: Schematic diagram of a multi-layer coating architecture](image)

*Figure 14: Schematic diagram of a multi-layer coating architecture*
4.3 Weld Overlays

The third surface engineering technique identified in Figure 4 is weld overlays. Weld overlays are used to deposit a hard layer on the face of a substrate (such as a forging die), to increase its wear resistance. Table 4 lists the compositions of some weld-overlays that have been used for hot forging dies\(^{(13)}\).

**Table 4:** Composition of weld overlays used for forging dies\(^{(13)}\).

<table>
<thead>
<tr>
<th>Material</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Mo</th>
<th>W</th>
<th>Fe</th>
<th>Ni</th>
<th>Co</th>
<th>Other</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stellite 6</td>
<td>1.2</td>
<td>1.0</td>
<td>1.0</td>
<td>---</td>
<td>---</td>
<td>28.0</td>
<td>---</td>
<td>5.0</td>
<td>2.5</td>
<td>2.5</td>
<td>Bal.</td>
<td>---</td>
</tr>
<tr>
<td>Stellite 21</td>
<td>0.25</td>
<td>1.0</td>
<td>1.0</td>
<td>---</td>
<td>---</td>
<td>27.0</td>
<td>5.5</td>
<td>---</td>
<td>2.5</td>
<td>2.5</td>
<td>Bal.</td>
<td>---</td>
</tr>
<tr>
<td>Inconel 625</td>
<td>0.05</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>21.0</td>
<td>9.0</td>
<td>---</td>
<td>1.0</td>
<td>Bal.</td>
<td>---</td>
<td>4Nb</td>
</tr>
</tbody>
</table>

5. Application of Thin-Film Coatings in Metal Forming Operations

PVD-type coatings are currently being used in a number of wear-sensitive applications, including cutting tools, firearms, automotive, aerospace, and die casting. For example, Voevodin et al.\(^{(22)}\) noted the recent transition of cutting tools from wet to dry machining, where traditional liquid-based lubricants are no longer necessary but instead lubrication is provided by thermally stable high temperature tool materials with hard and wear-resistant coated surfaces. They report that recent tool coatings consist of composite hard coatings with complex chemistries and compositions that provide optimum hardness, toughness and high temperature stability.

A detailed review of the architecture of PVD coatings for metal cutting applications has been provided by Inspektor and Salvador\(^{(23)}\). They reported that the first commercial PVD coating was TiN used on high speed drills circa 1980. Since then, the types of coatings used on metal cutting tools have continued to increase in complexity, and Inspektor and Salvador\(^{(23)}\) indicated that these coatings have progressed from single layer monolithic PVD coatings, through multi-layer coatings, to nanostructured coatings. For example, Figure 15 shows a multi-layer coating where an AlCrN layer is applied to provide adhesion to the substrate, the bulk of the coating is a TiAlN/AlCrTiN multi-layer structure, and a working (top) layer of AlCrN provides improve chip flow. The total coating thickness is about 10 µm.
A chart published online by Iscar (24) provides information about the current types of coatings commercially used on machine tools. Common coatings include TiAlN, TiCN and multi-layer TiN/TiCN. Kobelco, a division of Kobe Steel, also recommend a number of coatings applied to machine tools for both wet and dry machining of a number of metals (25), including multilayer TiAlN, TiCrAlSiN, Si added TiCrAlN and rare earth added TiCrAlN multilayer.

Voevodin et al. (22) recently reported that research on coatings for machine tools is currently focusing on coatings with the adaptive capability to form high temperature lubricating oxides of V, Mo, and other Magnéli phases. These types of coatings are described in more detail later in this review.

PVD coatings have also been used for many years in die casting applications. The coatings are typically used in applications where portions of the die cavity such as core pins or cavity inserts are exposed to extreme conditions, especially high temperatures, and where application of conventional liquid-based lubricants is difficult. PVD coatings used in die casting operations include CrN, AlTiN and AlCrN. It is worth noting that although the term “lubricants” is used for die casting applications, the “lubricants” used in die casting are really parting agents, that prevent the molten die casting alloy from contacting and adhering (alloying) to the die steel.
As described earlier in this report, a prior project at CSM introduced the concept of using PDV thin-film coatings to reduce or eliminate lubrication during die casting\(^{(3-6)}\). Reducing die lubrication has the potential to make significant improvements to the die casting process, including reducing residual porosity and entrapped gasses in the castings, lowering production costs, extending die life, improving cycle time, and reducing housekeeping issues. The results of laboratory testing and initial plant trials suggested that an AlCrN coating applied to the die casting die has the potential for dramatically reducing die lubricant. Commercial plant trials have shown that an AlCrN coating applied to an entire die reduced the application of conventional liquid-based lubricant by 80-90\%, and to date more than 20,000 shots have been produced using an AlCrN coated die. In addition to reducing spray, cycle time has been reduced by about 12\%, and testing has suggested that the reduction of spray increased the quality of the castings.

While laboratory and plant testing of thin-film PVD coatings in die casting applications has shown that PVD thin-film coatings can reduce or eliminate soldering (sticking) of the solidifying alloy to the steel substrate, and reduce erosion of the steel, the coatings typically have little-or-no impact on heat checking (thermal fatigue) caused by the cyclical heating and cooling of the steel substrate during the production of each casting\(^{(18)}\). However, as noted above, the use of thin-film coatings in die casting can significantly reduce the need for lubricants to be sprayed onto the die prior to each shot, and this reduction in spraying is expected to significantly reduce thermal fatigue in die casting operations, potentially doubling die life. As the thermal fatigue mechanism during forging is similar to that with die casting, it is possible that the use of die coatings, and the resultant reduction in die spray, could also reduce thermal fatigue (heat checking) and thereby extend die life during forging.

5.1 Forging Tools

For forging applications, PVD coatings are currently commercially available from a number of PVD coating companies. For example, for hot forging operations, Oerlikon Balzers\(^{(26)}\) recommends ALCRONA while Phygen Coatings\(^{(27)}\) recommends FortPHY, both of which are AlCrN PVD coatings. In 2011, Altan and Deshpande\(^{(13)}\) noted that studies examining the performance of thin-film ceramic coatings have been focused on die casting and extrusion applications, with few studies focusing on the application of thin-film ceramic coatings for forging applications. However, a number of studies have been performing focusing on the application of thin-film coatings for forging applications since then, and these are reviewed below. It is worth noting that few studies have explicitly studied utilizing coatings to improve lubricity.
Several researchers examined the impact of PVD hard coatings on wear and friction at the die-work piece interface, and coatings examined have included CrN, AlCrN, AlTiN, CrAlTiN, TiB₂, Ti(B,N), superlattice (Ti,Hf,Cr)N, and multilayer TiN/Ti-B-N\(^{(28-33)}\). These studies do not provide much information regarding highly lubricious coatings.

Other studies, however, were more useful. For example, Schrader et al.\(^{(34)}\) utilized a Double Cup Extrusion (DCE) test to examine the impact of PVD coatings on the tribological conditions during cold forging. Their data, shown in Figure 16, showed that while the type of conventional lubricant used during the DCE test had a large impact on the magnitude of the friction factor, the type of coating had only a minor influence. The research did show, however, that two multilayer coatings (TiC and AlCrN) exhibited excellent wear resistance when used in Punching–Forward Extrusions Tests.

![Figure 16: Friction factors of different coatings measured by Schrader et al.\(^{(34)}\).](image)

Leskovsek et al.\(^{(35)}\) performed a study that examined the impact of two types of 2 μm thin-film coatings on the performance of forging dies. Both coatings were produced by PECVN, one being a commercial TiCN coating (TiN base layer plus TiCN top layer), and the other a complex multi-layer architecture incorporating a TiN base layer, a multi-layer TiN/Ti-B-N zone, and a top TiB₂ layer. The Ti-B-N layer had an extremely fine grain size of around 5-7 nm, which the authors reported increased the coating’s overall resistance to plastic deformation and abrasion. The PECVN coatings were placed onto an H11 steel substrate, (47.5 HRC) surface hardened by plasma nitriding.

Coefficient of friction and wear rates results are shown in Figure 17, showing that the heat treated and polished (HP) sample of TiCN exhibited the best performance. However, the nitrided (N) samples of TiCN exhibited higher values for both coefficient of friction (CoF) and wear, due to lack of adhesion and spallation of the coating. In industrial forging trials,
a TiCN coated insert showed great improvement in forging tool life, although Leskovsek et al.\(^{(35)}\) did not report specific die life numbers.

![Graph showing friction and wear rate](image)

**Figure 17:** Average coefficient of friction (CoF) and steel ball wear rates measured by Leskovsek et al.\(^{(35)}\) for coated specimens.

Altan and Deshpande\(^{(13)}\) described wear tests performed on a (TiAl)N coating produced by PVD processing. The best wear resistance at 600°C (~1110°F) was observed for samples with a (TiAl)N coating on a nitrided surface. Altan and Deshpande\(^{(13)}\) reported that the nitrided layer enhanced the load bearing capacity of the system and hence reduced the difference in hardness between the substrate and the ceramic coating.

Podgrajsek et al.\(^{(36)}\) examined the failure mechanism of a 2.5 µm thick PVD processed TiAIN coating placed on a nitrided Dievar steel forging die. Podgrajsek et al.\(^{(36)}\) did not report using an adhesion layer, but indicated that the coating was “multi-layer”, although they only reported one composition for their coating (TiAIN). They also reported that life of the PVD coated die was 57% shorter as compared to a similar nitrided die (without the PVD coating). The PVD coated die exhibited cracking and spalling of the coating, with the cracking extending into the nitrided layer. The authors emphasize that, if PVD coatings are to be used, the die needs to be carefully designed to avoid plastic deformation of the steel substrate, which will lead to cracking and damage of the brittle ceramic coating.

The impact of various PVD and PECVD coatings on wear resistance has been evaluated during hot forging\(^{(14,37)}\). The results, reproduced in Figure 18, showed that CrN and CrVN coatings provided the best wear resistance.
Figure 18: Total wear rates measured by Yilkirana et al.\textsuperscript{(14)} at convex flash path radius after defined numbers of forgings.

Bayramoglu et al.\textsuperscript{(38)} examined the impact of die coatings and weld overlay on die life during the hot forging of Mn-Cr steels at 1200°C (~2200°F). As shown in Table 5, they found that the performance of various surface treatments in terms of die life increase from best to worst was the weld overlay coated dies (Co-Cr-Mo-Ni weld overlay), multilayer TOKTEK coated dies, single layer AlTiN coated dies, plasma nitrided dies and finally the as-received dies.

Table 5: Number of polishing operations and parts obtained with experimental dies\textsuperscript{(38)}.

<table>
<thead>
<tr>
<th>Operation</th>
<th>As-Received</th>
<th>Nitrided</th>
<th>AlTiN Coated</th>
<th>TOKTEK</th>
<th>Weld Overlay</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>V: nitride layer 250 μm nhd</td>
<td>V: nitride layer 100 μm nhd + TiBN layer</td>
<td>V: nitride layer 100 μm nhd + CrVN layer</td>
</tr>
<tr>
<td>1\textsuperscript{st} forging</td>
<td>1440</td>
<td>3920</td>
<td>4810</td>
<td>4660</td>
<td>8690</td>
</tr>
<tr>
<td>2\textsuperscript{nd} forging</td>
<td>1260</td>
<td>2140</td>
<td>1980</td>
<td>2030</td>
<td>8160</td>
</tr>
<tr>
<td>3\textsuperscript{rd} forging</td>
<td>1320</td>
<td>2110</td>
<td>1860</td>
<td>1750</td>
<td>8240</td>
</tr>
<tr>
<td>4\textsuperscript{th} forging</td>
<td>---</td>
<td>---</td>
<td>1720</td>
<td>1830</td>
<td>---</td>
</tr>
<tr>
<td>5\textsuperscript{th} forging</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>1780</td>
<td>---</td>
</tr>
<tr>
<td>Total</td>
<td>4290</td>
<td>9420</td>
<td>12280</td>
<td>13210</td>
<td>27810</td>
</tr>
</tbody>
</table>
6. Recent Advances in Lubricious Thin Film Coatings

Although, as noted above, there have not been a large number of studies that examined the impact of PVD coatings on lubricity for forging dies, a number of authors have examined the application of thin-film PVD coatings for lubrication applications for general metal processing applications at elevated temperatures. To-date, these coatings do not appear to have been applied to forging tools, but instead have focused on applications such as aerospace bearings and machine tools. However, it appears that they could also be utilized to provide high temperature lubrication on forming tools such as forging dies.

Table 6 shows information adapted from Voevodin et al.\textsuperscript{(22)} that shows examples of lubricants that can be applied to metal forming process at different temperature ranges. For example, Aouadi et al.\textsuperscript{(39)} noted that traditional liquid-based lubricants (oils and greases) provide lubrication at relatively low temperatures, but will vaporize at higher temperatures, leading to breakdown of lubricating conditions. At higher temperatures, solid lubricants can be used, and Aouadi et al\textsuperscript{(39)} noted that traditional solid lubricants fall into two classes:

- Intrinsic lubricants, with an atomic structure that can easily shear (i.e., MoS\textsubscript{2}).
- Extrinsic lubricants, which require the influence of an external additive from the surfaces to activate a low-shear mechanism (i.e., graphite in water).

Table 6: Lubricants used at various temperatures (adapted from reference 22).

<table>
<thead>
<tr>
<th>Mechanism</th>
<th>Material Examples</th>
<th>Temperature Range</th>
<th>Challenges</th>
</tr>
</thead>
<tbody>
<tr>
<td>Traditional lubricants</td>
<td>Oils and greases</td>
<td>&lt;~250°C</td>
<td>Vaporize at higher temperatures</td>
</tr>
<tr>
<td>Atomic structures that can easily shear</td>
<td>MoS\textsubscript{2}, Graphite</td>
<td>&lt;300°C</td>
<td>Oxidation at higher temperatures</td>
</tr>
<tr>
<td>Diffusion of soft metals to contact surface</td>
<td>Ag, Au, Cu encapsulated in TiN, CrN, VN, YSZ, CrAlN</td>
<td>300 – 500°C</td>
<td>Fast diffusion to surface depletes metal lubricant reservoirs</td>
</tr>
<tr>
<td>Lubricious oxide formation at contact surface</td>
<td>V\textsubscript{2}O\textsubscript{5}, MoO\textsubscript{3}</td>
<td>500 – 1000°C</td>
<td>Rapid loss due to oxidation can reduce lubricious effect</td>
</tr>
</tbody>
</table>
Aouadi et al. \cite{39} reported that most conventional intrinsic and extrinsic lubricants tend to operate at low temperatures (25 - 300°C, 70 - 570°F), but lose their lubricating properties at temperatures above 300°C (570°F), as they oxidize and become abrasive. They did report, however, that there are three classes of solid lubricants that can be used at higher temperatures, and these materials provide lubrication by plastically deforming and/or forming low-shear-strength surfaces at elevated temperatures. These materials are:

- Soft metals, such as silver, copper, gold, lead and indium
- Fluorides, such as CaF$_2$, BaF$_2$ and CeF$_3$
- Metal oxides, including V$_2$O$_5$ and Al$_2$Mo$_2$O$_7$

The materials, however, are typically not lubricous at relatively low temperatures, and so researchers have combined them with conventional lower temperature lubricants to create adaptive or chameleon coatings that can adapt their surface conditions to minimize friction while cycling at temperatures between room temperature and 1000°C (1830°F) – this will be discussed in more detail later in section 6.5\cite{39}. Information about the different types of coatings are summarized below.

6.1 MoS$_2$-Containing Coatings

Molybdenum disulfide (MoS$_2$) is classified as a transition metal dichalcogenide, and has excellent lubricating properties due to its layered structure (see Figure 19)\cite{40}. However, MoS$_2$ exhibits the lowest coefficient of friction in a vacuum or dry atmosphere, but the presence of humidity can increase its CoF by more than two orders of magnitude. Rigato et al.\cite{41} noted that MoS$_2$ can be deposited using sputtering PVD processing through the use of a pure MoS$_2$ target. Due to the soft nature of MoS$_2$, however, rather than depositing a homogeneous layer onto the face of a metal working tool, it is more common to co-sputter harder supporting materials in either multi-layer or dispersed structures. For example, Rigato et al.\cite{41} reported that while single-layer MoS$_2$ coatings detached from the substrate abruptly and almost immediately upon the application of a load, Ti-MoS$_2$ coatings were much tougher and could better withstand mechanical loading.
Renevier et al.\textsuperscript{[42]} reported on industrial trials of Ti-MoS\textsubscript{2} PVD coatings for a number of metal cutting and forming operations, including drilling, end milling, piercing, blanking and drawing. For example, Figure 20 reproduces their data for an M4 punch coated with a 3-3.5 µm layer of TiCN plus a 1.2 µm top-layer of MoS\textsubscript{2}-Ti. As shown in Figure 20, the punch with the multilayer TiCN/MoS\textsubscript{2}-Ti coating was able to pierce more than 200,000 parts before failure, a greater than ten-fold increase over the uncoated tool.

Bobzin et al.\textsuperscript{[43]} noted that AlCrN coatings can be used for cold forging processes, due to their high hardness and excellent resistance to abrasion wear, but suggested that the addition of MoS\textsubscript{2} could provide additional lubricity. They examined the impact of chemical composition on the performance of (Cr\textsubscript{1-x}Al\textsubscript{x})N/Mo\textsubscript{y}S\textsubscript{2}-type coatings. Their results are
shown in Figure 21, indicating the coated samples performed better than the uncoated substrate (M2 tool steel), and the coating with a composition of \((\text{Cr}_{54.3}\text{Al}_{45.7})\text{N/MoS}_{2.13}\) exhibited the lowest value of the coefficient of friction. However, overall coefficient of friction values were still relatively high.

![Figure 21: Tribological tests using pin-on-disk tribometer performed at room temperature](image)

- **a)** Uncoated substrate
- **b), c) & d)** utilizing \((\text{Cr}_{1-x}\text{Al}_x)\text{N/MoS}_{y}\text{SZ}-type coatings**

Another potential MoS\(_2\)-containing coating is i-Kote, which is available from Tribologix\(^{(44)}\). i-Kote is a nanocomposite coating that can shift lubricating mechanism depending upon operating environment.

### 6.2 Hard Coatings

Many of the hard coatings used for tooling (TiN, CrN, AlCrN) can provide wear resistance, but not lubricity. However, a coating material that has the potential to provide excellent lubricity together with high hardness is diamond-like-carbon (DLC). DLC is a class of carbon having an amorphous crystal structure, and is normally used commercially in the form of a thin-film coating. As shown in Figure 22, DLC can present low values of CoF, while still providing high hardness and resistance to wear.
As noted in Figure 20, TiCN is another hard coating that has the potential to provide lubricity. It has been reported to exhibit a coefficient of friction of 0.35 in cutting applications\(^{(46)}\).

6.3 Noble Metal Solid Lubricants

Solid lubricants that can be used at higher temperatures than MoS\(_2\) include noble metals (see Table 6). Voevodin et al.\(^{(22)}\) reported that Ag and Au are reliable solid lubricants for use in both air and in vacuum, as they exhibit low shear stress from room temperature to their melting points. These noble metals perform best in lubricating conditions when applied with a hard, supporting substrate such as a thin-film coating. According to Voevodin et al.\(^{(22)}\), the best lubricating performance is achieved when the noble metal is in the thickness range of 300-1000 nm.

Hard nano-composite coatings have been developed to provide lubricating conditions utilizing these noble metals, while maintaining good wear resistance. An example of a noble metal solid lubricant includes Y-stabilized zirconia and Au nanocomposite coatings containing up to 20 at% Au, which exhibited a hardness of about 15 GPa. Other examples include TiC-Ag, YSZ-Ag, Ta\(_2/\)Cr\(_2\)AlC-Ag, CrN-Ag, CrAlN-Ag, Mo\(_2\)N-Ag and MoCN-Ag\(^{(22)}\).

One problem with these noble-metal lubricants is that the noble metal can rapidly diffuse to the surface of the coating, as shown in Figure 23. For example, Voevodin et al.\(^{(22)}\) reported that a one micron-thick YSZ-based composite coating containing 20 at.% Ag as nano-inclusions will be depleted of all Ag after less than 5 minutes at 500°C (930°F). Researchers have examined several methods to modulate the diffusion of the noble metals to the surface of the coatings, and such techniques include depositing a TiN diffusion barrier patterned with an array of holes.
**Figure 23:** Metal diffusion to the surface from nanoscale size inclusions in ceramic matrices\(^{(22)}\)

Left: schematic of Ag or Au diffusion out of an adaptive nitride or oxide matrix hard coating. Right: cross-section (top) and surface (bottom) morphology of a CrN-Ag adaptive hard coating after heating to 650°C (1200°F).

Mulligan et al.\(^{(47)}\) also examined methods to slow down the diffusion of the noble metal to the surface. They examined the impact of 200 nm thick pure CrN diffusion barrier cap surface layers on the performance of sub-surface CrN hard coatings containing dispersions of silver. Their data are reproduced in Figure 24, showing the results of elevated temperature (550°C, 1020°F) tribological sliding tests of this coating system against alumina balls. The data in Figure 24 shows the CoF (μ) versus cycle number, where each cycle corresponds to a distance of 16 mm (0.63 in). The CrN–Ag composite coating without the cap layer shows an initially low friction coefficient of 0.16, which Mulligan et al.\(^{(47)}\) attributed to Ag transport to the surface, and after 6,000 cycles (a linear distance of about 96m, 315 ft), the friction coefficient increased to 0.39 as the Ag reservoir in the coating became depleted. However, the system capped with a dense 200 nm thick layer of CrN exhibited a higher initial friction coefficient of 0.43, which decreased to 0.23 after an initial run-in period. Mulligan et al.\(^{(47)}\) attributed the decrease in the friction coefficient to the development of openings in the barrier layer (generated by the wear), which allowed the Ag lubricant to diffuse to the sliding top surface.
6.4 Highly Lubricious Oxide Coatings

The final type of high temperature solid lubricants identified in Table 6 are highly lubricious oxide coatings. Aouadi et al.\(^{39}\) noted that for applications at extremely high temperatures, it is unreasonable to expect materials to resist oxidation for extended periods of time, and so they examined and reviewed materials that form low interfacial shear-strength oxides at higher temperature ranges. Aouadi et al.\(^{39}\) indicated that there are five mechanisms reported in the literature for the origin of the lubricious behavior in these lubricious oxide coatings:

1. Easy-shearing due to screening of cations by surrounding anions (crystal chemical model)
2. Material softening that occurs when the working temperature is between 0.4 and 0.7

Finally, it is worth making a few comments regarding the cost of these noble metal-containing coatings. Although the price of Ag and Au are high (around $550/kg and $41,000/kg, respectively, at time of writing), the actual value of noble metal in these types of coatings is relatively low, due to the thin nature of the coating applied to the forging die. For example, for a 2µm thick layer of coating containing 20% noble metal applied to a 12-inch by 12-inch tool face, the actual value of noble metal in the coating is about $0.20 of Ag and $30 of Au. Now the actual cost of deposition might be higher due to coating of the noble metal onto the inside of the deposition chamber, but these costs are small compared to the overall cost of a forging die.
of the absolute melting point \((T_m)\), which corresponds to the brittle-to-ductile transition of most oxides (similar to the lubricious behavior observed for soft metals)

3. Melting of the oxide by exceeding \(T_m\) (similar to the mechanism that reduces friction in liquids)

4. Creation of a material with a lamellar crystal structure with weak inter-planar cohesive bonds (similar to the shearing mechanism that occurs in graphite and molybdenum disulfide)

5. Shearing in textured nanocrystalline grains due to dislocation glide resulting in intra-crystalline slip

Several authors have published results documenting the lubricating properties of these lubricious metal oxides. For example, Voevodin et al.\(^{(22)}\) reported that hard coatings produced from transition metal nitrides, such as VN, MoN, WN, are oxidized at high temperatures, producing binary oxides that are lubricious at these temperatures. For example, MoO\(_3\) was identified to have a CoF of 0.2 at 700°C (1300°F)\(^{(22)}\).

Franz and Mitterer\(^{(48)}\) reported that many of the lubricious oxides form a series of compounds commonly referred to as Magnéli phases, named after A. Magnéli who first investigated their crystal structures. An example is V\(_2\)O\(_5\), whose atomic structure is shown in Figure 25, and has a layered structure similar to graphite and molybdenum disulfide (the latter is shown in Figure 19). Franz and Mitterer\(^{(48)}\) noted that this phase is responsible for the reduction in friction in vanadium-containing hard phases at elevated temperatures.

![Figure 25: View of layered structure of V\(_2\)O\(_5\), where the V atoms are represented as grey balls, and the O atoms as red balls. Weak van der Waals bonds between layers are omitted for clarity\(^{(48)}\).](image)

Franz and Mitterer\(^{(48)}\) reported that the addition of vanadium to nitride-based PVD hard coatings enable the formation of lubricious oxides at elevated temperatures, and that that
Coefficient of friction values in the range from 0.1 to 0.2 at temperatures between 600 and 700°C (1110 and 1300°F) can be achieved. Although V₂O₅ can provide low CoF values, its wear resistance is also poor, so Franz and Mitterer⁴⁸ noted that the best performance for metal forming operations would be to combine these vanadium-based phases with hard coatings, such as TiAlN/VN multi-layer coatings, or TiAlVN or CrAlVN single layer coatings, as shown in Figure 26.

**Figure 26:** Lifetime of single-layer and multi-layer coatings in milling of 42CrMo4 steel, compared to the TiAlN reference coating⁴⁸.

However, Franz and Mitterer⁴⁸ reported that V₂O₅ melts at temperatures between 670-685°C (1240-1265°F), so the low CoF values measured at 700°C (1300°F) can also be attributed to a liquid lubrication mechanism.

Franz and Mitterer⁴⁸ noted that, similar to the noble metal solid lubricants discussed previously, rapid loss of vanadium due to oxidation in these coating can lead to a reduction in the lubricious effect, and therefore incorporating oxidation resistant layers might assist in slowing the oxidation rate, and thereby increasing the time over which the coatings are effective.

Bobzin et al.⁴⁹ examined similar self-lubricating high temperature coatings, based on the AlCrN hard coating system and containing either vanadium or tungsten. Again, the AlCrN coating provides for wear protection, while the vanadium or tungsten generates friction reducing oxides at high temperatures. Figure 27 shows the results of tribological testing performed at room temperature, 600°C (1110°F), and 800°C (1470°F), showing that the friction coefficient drops to 0.05 at 800°C for the (Cr,Al,V)N coating.
Figure 27: Mean friction coefficient $\mu$ at RT, 600 °C and 800 °C of the reference material 1.2999 and both coatings [(Cr$_{0.59}$Al$_{0.21}$V$_{0.20}$)N and (Cr$_{0.53}$Al$_{0.14}$W$_{0.33}$)N] (49).

Stone et al. (50) examined the performance of Ag-Ta-O thin film coatings with various Ag/Ta ratios and oxygen contents, as well as TaN/Ag produced by sputtering from Ta and Ag targets in a reactive oxygen atmosphere. Coating compositions examined by Stone et al. (50) are shown in Table 7, and CoF values at different temperatures are shown in Figure 28. As shown in Figure 28, the CoF dropped as the temperature was increased and the coating S4 exhibited a very low CoF of about 0.04 at a temperature of 750°C (1380°F). Coating S1 which did not contain silver, had the highest CoF at all temperatures.

Table 7: Power to the Ta ($P_{Ta}$) and Ag ($P_{Ag}$) sources, oxygen partial pressure and elemental composition for selected tantalum oxide (S1) and silver tantalum (S2, S3 & S4) samples (50).

<table>
<thead>
<tr>
<th>Sputtering Parameters / Chemical Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample</td>
</tr>
<tr>
<td>--------</td>
</tr>
<tr>
<td>S1</td>
</tr>
<tr>
<td>S2</td>
</tr>
<tr>
<td>S3</td>
</tr>
<tr>
<td>S4</td>
</tr>
</tbody>
</table>
Figure 28: Steady-state CoF for silver tantalite coatings as a function of temperature.

Regarding TaN/Ag coatings, the TaN/Ag coating has a CoF of about 0.25 at 750°C, (1380°F), as shown in Figure 29.

Aouadi et al.\textsuperscript{(51)} deposited nanocomposite coatings of Mo$_2$N/MoS$_2$/Ag onto Inconel and silicon substrates by sputtering, using individual targets of Mo, MoS$_2$ and Ag. The compositions of the five samples are listed in Table 8. Four of the samples contain between 16 and 25% at. % Ag, while three of them contain MoS$_2$. 
Figure 29: Steady-state CoF for TaN and TaN/Ag as a function of temperature\(^{(50)}\).

Table 8: Nomenclature, powers to the Ag and MoS\(_2\) target and compositions, of Mo\(_2\)N/MoS\(_2\)/Ag coatings obtained by magnetron sputtering of Mo, Ag, and MoS\(_2\) targets\(^{(51)}\).

<table>
<thead>
<tr>
<th>Coating</th>
<th>S-48-20</th>
<th>S-60-60</th>
<th>S-30-20</th>
<th>S-42-0</th>
<th>S-0-0</th>
</tr>
</thead>
<tbody>
<tr>
<td>(P_{\text{Ag}}) (W)</td>
<td>48</td>
<td>60</td>
<td>30</td>
<td>42</td>
<td>0</td>
</tr>
<tr>
<td>(P_{\text{MoS}_2}) (W)</td>
<td>20</td>
<td>60</td>
<td>20</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Mo (At. %)</td>
<td>40</td>
<td>40</td>
<td>45</td>
<td>47</td>
<td>63</td>
</tr>
<tr>
<td>N (At. %)</td>
<td>35</td>
<td>27</td>
<td>34</td>
<td>28</td>
<td>37</td>
</tr>
<tr>
<td>S (At. %)</td>
<td>4</td>
<td>12</td>
<td>5</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Ag (At. %)</td>
<td>21</td>
<td>21</td>
<td>16</td>
<td>25</td>
<td>0</td>
</tr>
</tbody>
</table>

The results of their testing at different temperatures are shown in Figure 30 and summarized below:

- **Testing at room temperature** – S-48-20 samples had the lowest CoF at room temperature, about half that of the S-0-0 (consisting only of Mo\(_2\)N). However, none of the coatings exhibited the low coefficient of friction expected from coatings containing MoS\(_2\), which Aouadi et al.\(^{(51)}\) attributed to the MoS\(_2\) coating in their tests being amorphous, and thus not having the layered structure necessary for effective lubrication.
• **Testing at 350°C (660°F)** – For all the coatings that contained silver, the coefficient of friction was lower at 350°C (660°F) as compared to room temperature. The CoF was unchanged for the S-0-0, a coating that was silver-free.

• **Testing at 600°C (1110°F)** – The coefficient of friction was lowest for three of the samples at 600°C (1110°F), and the CoF values are quite low (in the range of 0.10-0.15). Aouadi et al.\(^{(51)}\) attributed this to the formation of a number of lubricous silver molybdate phases at the surface of the coatings (\(\text{Ag}_2\text{Mo}_4\text{O}_{13}\), \(\text{Ag}_2\text{MoO}_4\), and \(\text{Ag}_2\text{Mo}_2\text{O}_7\)).

![Figure 30: Impact of coating composition on coefficient of friction at different temperatures\(^{(51)}\).](image)

6.5 **Adaptive Tribological Coatings**

Aouadi et al.\(^{(51)}\) describe a new class of materials that, although in their infancy, can adapt the friction and wear characteristics over a range of temperatures. One example they provide is a YSZ/Au/MoS\(_2\)/DLC (YSZ is Yttria stabilized zirconia, DLC is diamond-like carbon) where:

• Load bearing is provided by the YSZ and the DLC
• Lubrication is provided by the other phases
  - MoS\(_2\) provides lubrication at <300°C (660°F) in a vacuum or dry environments
  - Graphite is a byproduct of DLC, and provides lubrication in humid environments
- Au provides lubrication at >300°C (660°F)

In a later paper, Aouadi et al.(52) extended the concept, with the goal of developing an adaptive coating that can provide lubricious behavior from 250 to 1,000°C (480 to 1830°F). They examined a two-phase nanocomposite coating that consists of inclusions of Ag in a VN matrix. They chose this nanocomposite composition expecting that Ag and Ag vanadate phases would form on the surface of these coatings. The goal was that the Ag phase would reduce friction from room temperature to mid-range, while at high temperature the Ag and V would react with O to produce a lubricious silver-vanadium-oxygen phase.

The results from their tribological testing is shown in Figure 31. The lowest friction coefficients recorded for samples with identical compositions were 0.35, 0.30, 0.10 and 0.20 at 25, 350, 700 and 1000°C (70, 660, 1290, and 1830°F), respectively.

![Figure 31: Coefficient of friction values as a function of temperature for sliding tests performed against Si3N4 balls(52).](image)

7. Plasma Sprayed Coatings

Although not strictly a PVD coating, it is worth describing plasma-sprayed solid lubricants that have been developed by NASA for high temperature applications. The most recent, PS400, comprises a nickel-molybdenum binder that provides strength, creep resistance and oxidation resistance, while silver and barium–calcium fluoride are added for
lubrication\(^{(53)}\). As shown in Figure 32, the solid lubricant has a splat-layered morphology typical of thermally sprayed coatings, and coefficient of friction data at various test temperatures is shown in Figure 33.

**Figure 32:** Micrograph from a cross-section of PS400 coatings showing a splat layer morphology\(^{(53)}\).

![Micrograph from a cross-section of PS400 coatings showing a splat layer morphology.](image)

Test conditions: load 5 N, 3 m/s sliding velocity, air atmosphere 50 percent relative humidity, Inconel X-750 pins

**Figure 33:** Coefficient of friction data from PS400 solid lubricant\(^{(53)}\).

![Coefficient of friction data from PS400 solid lubricant.](image)
8. Laser Textured Surface

Finally, another surface modification technique worth mentioning is laser texturing. Wu\textsuperscript{(54)} examined the impact of laser texturing for forging applications in a PhD program performed at The Ohio State University, and found that values for coefficient of friction were reduced by as much as 50% after texturing, with the reduction being due to a lower area of contact between the die and forging.
References


18. Steve Midson, Applications of Surface Engineering for Die Casting Dies, in preparation for NADCA.


47. C.P. Mulligan, T.A. Blanchet & D. Gall, Control of Lubricant Transport by a CrN Diffusion Barrier Layer during High Temperature Sliding of CrN-Ag Composite Coating, Surface and Coatings Technology 205 (2010) 1350-1355.