Plasma nitriding offers a way to significantly improve the surface properties of tool steel stamping dies, and is not limited by their size to its application, as are some alternative surface treatments.

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Advanced high-strength steels (AHSS), such as dual phase (DP), transformation-induced plasticity (TRIP), and some other high-strength steels (HSS) for auto body parts require the use of stamping dies having improved durability and wear resistance[1-5]. In addition, most of these new automotive steels also are coated with a zinc or iron-zinc alloy galvanized coating before stamping, which greatly affects the coefficient of friction during stamping[3]. AHSS also have a higher volume fraction of very hard martensite, which requires improved tool surface finish.

Stamping of these materials is accompanied by higher contact pressures between the tool and stamped material and higher tool temperature from frictional heat, which places additional requirements on tool surface engineering to prevent powdering, galling, and other damage to the galvanized coating[6]. It is important to prevent damage to the coating to ensure the quality of stamped parts when Class A surface is required and to lower die maintenance costs.

Galvanically applied hard chromium, which has been extensively used to coat stamping dies, cannot withstand some of the high contact stresses generated during stamping and forming AHSS. A combination of various surface treatments (i.e., duplex surface engineering) allows producing a surface composite having a combination of properties unobtainable through any individual surface technology. For example, combining induction or flame hardening with consecutive gas or plasma nitriding treatments and coating with a hard PVD layer can produce a superior tool capable of withstanding the most difficult stamping applications[7]. For large tools where size limits the application of a hard PVD layer, coated inserts or welded draw beads can be used[8]. Other existing surface engineering techniques can provide duplex diffusion and coating layers, and often are used to treat small stamping dies. More demanding applications can require the formation of even more complex duplex diffusion and coating layers, such as with plasma nitriding, to withstand severe contact stress applied to the tool[9].

While there are many different combinations of surface treatments that can be applied to stamping dies, a critical limiting factor in the ability to apply a specific treatment is the unavailability of surface engineering chambers large enough to accommodate the work, a limitation that does not apply to plasma nitriding technology. Plasma (ion) nitriding alone is recognized as a prime hardening treatment of the larger size stamping dies[8, 10]. Figure 1 shows examples of such large dies plasma nitrided at the Advanced Heat Treat Corp. Monroe, Mich., facility.

For nitrided dies used to produce stampings having a Class A surface,
the lowest possible tool surface roughness and a smooth tool-hardness gradient across the nitrided layer are recommended. Low surface roughness comparable to chrome plating can be achieved by subsequent polishing of the ion-nitrided tool surface.

Many die materials, such as those listed in Table 1, have chemical compositions that make them good candidates for plasma nitriding. Full size stamping dies are cast from all of the materials in Table 1 except M2 high-speed tool steel, which is used for inserts and welded beads, especially in the most stressed area of the stamping tools.

Importance of Proper Processing and Die Finishing

Plasma nitriding of large tools requires a good starting vacuum and clean chamber atmosphere to produce a proper case structure having maximum hardness (with or without a nonporous compound zone of limited thickness). This cannot be achieved without proper cleaning of dies before loading them to the nitriding chamber. The most common impurities that must be removed are lubricants adsorbed on the die surface during tryouts and paint in the case of processing repaired dies. These impurities contaminate the vacuum system and result in doping of the plasma; that is, they increase the nitriding potential of the plasma resulting in formation of the unwanted case structure having a porous \( \varepsilon \) (epsilon) plus \( \gamma' \) (gamma prime) compound zone, or they drastically reduce the kinetics of nitriding when too much oxygen-bearing gases are present\[12\]. The porous near-surface tool layer contains some oxygen (Fig. 2) that affects die properties, and, as a result, the surface hardness is slightly reduced as shown in Fig. 3. Final polishing must always be applied to remove this layer to achieve maximum hardness and minimize surface roughness of the plasma nitrided die. A typical microindentation hardness test may not reveal the presence of the layer or any variation in the near-surface hardness region because the first microhardness indentation is typically 0.001 in. (0.025 mm) below the surface as shown on the plasma nitrided M2 sample in Fig. 4. However, after polishing, the hardness is very high (~1,280 Knoop at 100 g load). Figure 5 shows the microstructure of the nitrided M2 sample.

Extremely high surface hardness can also be achieved on plasma nitrided D2 cold work tool steel (Fig. 6), which is commonly used for casting die-inserts or smaller dimension dies. Figure 7 shows the microstructure of the nitrided D2 sample. Similar to the M2 material, it is necessary to have a nitrided layer with the proper structure to avoid a brittle condition; mainly the absence of both a compound zone and network of nitrocarbides at the grain boundaries.

Response of Modern Die Materials to Plasma Nitriding

Common die materials have a very good response to conventional heat treatments such as quenching and tempering after flame or induction heating, and they also respond very
well to plasma nitriding (Table 2). Surface hardness after nitriding is very high (over 90 HR15N) for all the tested materials except Carmo, which formed the lowest case depth and had low core hardness due to its annealed condition. The other materials formed a sufficiently deep case to resist indentation of the Rockwell hardness indenter under the 15 kg load, or they had high core hardness (such as with M2, which has good resistance to tempering at nitriding temperatures). Superficial hardness measurements taken using a portable Vickers instrument ranged above 850 HV1 (more than 65 HRC equivalent). Photomicrographs of the plasma nitrided samples tested with corresponding hardness profiles are

### Table 1 — Nominal chemical composition of die materials evaluated

<table>
<thead>
<tr>
<th>Material</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Cu</th>
<th>Al</th>
<th>Mo</th>
<th>V</th>
<th>W</th>
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</thead>
<tbody>
<tr>
<td>M2</td>
<td>0.85</td>
<td>0.35</td>
<td>0.35</td>
<td>0.03</td>
<td>4.25</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>5.00</td>
<td>2.00</td>
<td>6.00</td>
</tr>
<tr>
<td>D2</td>
<td>1.5</td>
<td>0.6</td>
<td>0.6</td>
<td>—</td>
<td>12</td>
<td>0.3</td>
<td>—</td>
<td>—</td>
<td>0.95</td>
<td>1.1</td>
<td>—</td>
</tr>
<tr>
<td>Cast Cut#1</td>
<td>0.60</td>
<td>1.45</td>
<td>0.60</td>
<td>0.12</td>
<td>1.50</td>
<td>0.25</td>
<td>0.40</td>
<td>0.10</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Caldie</td>
<td>0.7</td>
<td>0.5</td>
<td>0.5</td>
<td>—</td>
<td>5.0</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Carmo</td>
<td>0.6</td>
<td>0.8</td>
<td>0.35</td>
<td>—</td>
<td>4.5</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>2.3</td>
<td>0.5</td>
<td>—</td>
</tr>
</tbody>
</table>

M2 is a high-speed tool steel. D2 is a cold-work tool steel. Cast Cut#1 is a registered trademark of Nelson & Assoc. Research Inc., Clinton Township, Mich. Caldie and Carmo are trademarks of Böhler Uddeholm Corp, Rolling Hills, Ill.

### Table 2 — Average hardness of nitrided and quenched and tempered die materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Nitrided 950°F/15 h in 25% N₂-75% H₂</th>
<th>Nitrided 900°F/15 h in 5% N₂-95% H₂</th>
<th>Quenched and tempered HRC</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Surface, HR15N</td>
<td>Surface, HV1</td>
<td>Surface, HR15N</td>
</tr>
<tr>
<td>M2</td>
<td>—</td>
<td>—</td>
<td>94.5</td>
</tr>
<tr>
<td>D2</td>
<td>—</td>
<td>—</td>
<td>92.8</td>
</tr>
<tr>
<td>Cast Cut#1</td>
<td>91.1</td>
<td>874</td>
<td>91.5</td>
</tr>
<tr>
<td>Caldie</td>
<td>92.9</td>
<td>880</td>
<td>90.1</td>
</tr>
<tr>
<td>Carmo</td>
<td>90.0</td>
<td>930</td>
<td>83.0</td>
</tr>
</tbody>
</table>

![Fig. 4](image1.png) — Hardness profile in the M2 sample plasma nitrided at 900°F; total case depth is 0.0036 in.

![Fig. 5](image2.png) — Photomicrograph of the M2 sample plasma nitrided at 900°F showing the compound zone-free and network-free structure of the nitrided layer. Etchant: 2% nital.

![Fig. 6](image3.png) — Hardness profile in the D2 sample plasma nitrided at 900°F. Total case depth 0.004 in.

![Fig. 7](image4.png) — Photomicrograph of the D2 sample plasma nitrided at 900°F showing the compound zone-free and network-free structure of the nitrided layer. Etchant: 2% nital.
Many die materials have chemical compositions that make them good candidates for plasma nitriding.

Table 3 — Scratch test results on nitrided die materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Penetration depth(a), μm</th>
<th>Residual depth, μm</th>
<th>Coeff. of friction, μ</th>
</tr>
</thead>
<tbody>
<tr>
<td>M2 nitrided at 900°F</td>
<td>4.75</td>
<td>2.0</td>
<td>0.23</td>
</tr>
<tr>
<td>Cast Cut #1 nitrided at 950°F</td>
<td>3.28</td>
<td>1.43</td>
<td>0.32</td>
</tr>
<tr>
<td>Cast Cut# 1 nitrided at 900°F</td>
<td>3.00</td>
<td>1.10</td>
<td>0.27</td>
</tr>
<tr>
<td>Cast Cut# 1 nitrided at 900°F + CrN coating</td>
<td>2.83</td>
<td>1.26</td>
<td>0.19</td>
</tr>
</tbody>
</table>

(a) Penetration depth of Rockwell indenter at a normal force of 2 N.

Many die materials have chemical compositions that make them good candidates for plasma nitriding.

shown in Figs. 8 through 13.

Cast Cut #1 (Nelson & Assoc. Research Inc., Clinton Township, Mich.) and Caldie and Carmo (Böhler Uddeholm Corp., Rolling Meadows, Ill.) alloys readily form a compound zone (white layer) of the γ nitrides (Fe_N) when nitrided at 950°F (510°C) even with only 5% nitrogen in the atmosphere. The compound zone may play the same role as the hard coating does in many applications, because its crystallographic structure and properties are similar to a certain degree to many PVD and CVD coatings.

It should be noted that the case hardness of nitrided M2 is much higher than the other steels, and is very close to the high hardness (1,500 HV) of chromium nitride[13]. Chromium nitride is widely used as a wear-resistant coating, although it has a high coefficient of friction. The coating is well known for its very low internal stresses[10]. The hard zone in nitrided M2 is many times deeper than any of the other coatings, and therefore, could perform as well as any other steels coated with CrN. This is illustrated by the scratch-test results in Table 3. Performance of variously treated samples of the Cast Cut #1 and M2 steels is compared with the CastCut #1 nitrided and coated with the 2-μm thick CrN layer. The coefficient of friction of the CrN coated sample in the scratch test using the Rockwell C indenter is slightly lower than that for the nitrided-only M2 sample.

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