VALVE MATERIALS

What's new and what's old but still great…

BY **FERNANDO CURELLO**

The evolution of valve materials is an amazing story being written since the early days of our beloved internal combustion engines. There is a symbiotic relationship between them because of the constant demand for higher specific power outputs (HP per cubic inch), lower fuel consumption (higher mileage) and emissions controls, forced the development of new alloys. At the same time, these alloys that were added allowed new improvements in the engine characteristics. Let us not forget to include in this scenario the never-ending pressure for improved durability and lower costs for volume engine productions.

The metallurgy and technology of valve materials is a special field of heat resistant steels and other high temperature alloys. These materials must operate reliably over long periods of time at elevated temperatures and in corrosive environments under high dynamic loads. Therefore, valve materials must maintain, even at the highest operating temperatures, good hot hardness to resist indentation and wear, high tensile strength and fatigue strength, excellent hot corrosion, and oxidation resistance under widely varying environment conditions. Also desirable are a low thermal expansion coefficient and compatibility with guide and seat materials.

With that said, let's get to the point and talk about valve materials families. Valve materials families is the best way we can better understand what has strategically happened in this field, in the many years of engine and valvetrain developments.

So, in trying not to go too deep in the metallurgy and chemical considerations, we can split basic valve materials in five families, plus another four families for seat fortifications coatings (hard facing alloys), adding finally the stems (or overall valve) coatings or thermochemical treatments. These are as follows:

Martensitic Steels: Iron-Carbon alloys that can be hardened by the heating and quenching process and are magnetic at room temperature.

Austenitic Steels: Iron-Carbon alloys that cannot be hardened by quenching and require the so-called "precipitation" process, also nonmagnetic at room temperature.

High Nickel Alloys: Not Iron-Carbon alloys but based on approximately 70% Nickel on weight, and 15 to 23% Chromium. Also known as "superalloys", they are not magnetic and hardenable only via the precipitation process.

Medium Nickel Alloys: Developed in the '80s and '90s for cost reduction purposes due to the ever-increasing Nickel price worldwide. These alloys can match the high temperature characteristics of High Nickel alloys, at a lower cost.

Light Alloys: Basically Titanium-Aluminum alloys that initially were only used in racing applications for weight reduction, but extended to high performance engines, high revving motorcycles and any high revs applications. As you all well know the huge jump in pricing for these titanium alloys against the above-mentioned alloys, plus the engine designs trend for increasing number of valves per cylinder, making smaller valves with less weight, kept their usage only to those fields.

Hard Seat Face Coatings: Developed to extend seat face durability, reduce wear, and valve recession during usage. We have several Cobalt base alloys, some Nickel base alloys, and some iron base alloys that, in combination with Chromium, Nickel, Carbon and Molybdenum, offer a lower cost alternative, and in certain specific cases even more appropriate performances. The application is via a Plasma Transferred Arc Process (PTA), welded to the valve seat area.

Overall or Stems-only Valve Coatings: Finally, we must include the coatings or additional treatments to protect stems from abnormal wear, to reduce the guide-stem wear by lowering friction and improving the tribological characteristics of the two materials in contact. Here we have the well known and widely used hard chromium layer, the carbonitriding processes, and the PVD (Physical Vapor Deposition) processes. The most common PVD are Titanium Nitride, Chromium Nitride, and DLC (Diamond Like Carbon). Also used for protecting titanium valve stems is the Plasma Jet Spray with Molybdenum powder.

So, let's talk about all these alternatives, as said "what, why, when and how"…

Martensitic Alloys

These are steels with medium Carbon content (from 0.20% to 0.50%, and 0.80-0.90%), with the addition of Chromium, Silicon, and Manganese, which have been used for the last 80 years as intake valve materials (and the basic ones remain the same… so, nothing new under the sun, but still good!). They are magnetic at room temperature, can be hardened by heating and quenching in oil, with additional tempering to get the required tensile strength and minimum hardness needed for intake valve usage.

Why do we need to state this? Because the operation temperatures play the most important role in valves fatigue strength and durability. The combustion heat inside the chamber is transferred to the valves, from the valve seat face to their valve seat inserts and ultimately to the cylinder head water jacket. Intake valves usually reach between 650 to 950ºF at the head, allowing these martensitic steels to be used with good results. Still today, the martensitic intake valve materials represent the highest percentage of usage at OEM level worldwide. They also maintain good usage in the race engines field, for intake applications only, and where operation temperatures are low (natural aspirated, regular fuels, etc.).

The other huge application field for these steels are valve stems for bimetallic intake or exhaust valves; usually a mid-stem weld, as hardened tips welded to the end of austenitic steel or "superalloys" one-piece forged valves.

Let's explain a bit here… as you see in Figure 1, as valves cool down from the head to the stem end because the heat absorbed from the combustion chamber and exhaust gases is being transferred first to the seats and then to the guides along the stems. Ideally, temperatures are lower in the stems so there is no need to use the same material on heads and stems. Martensitic steels are

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welded via friction welding process. The head portions that can be high resistant alloys. Also, as we explain later, these high resistant alloys cannot reach the higher hardness values to withstand the friction and loads exerted by rocker arms. In addition, when multiple grooves are used in valve locks to improve valve rotation, we need to increase the hardness on that area to withstand friction and wear at the grooves. Thus, a hardening quenching on the keeper areas and tip area, or the so called "wafer welded tips" gives the hardness we need.

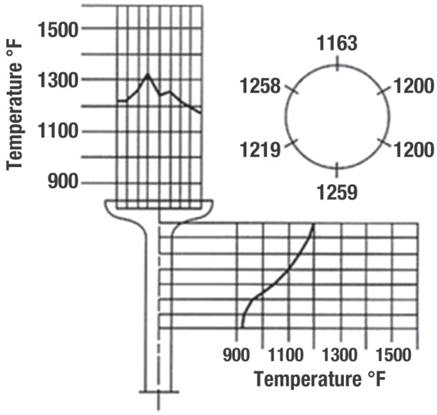

Figure 1: Typical diesel exhaust valve temperatures, across the head and along the stem.

The most popular and best-known intake valve alloy is commercially known as SIL#1 (Silchrome #1), or using an old SAE name, HNV3. This steel contains the principal alloying element Chromium (9.0%), with the addition of Silicon (3%) to increase thermal fatigue properties and oxidation resistance. Manganese (0.5%) is added to control transformation during hardening. 50-55 HRC can be reached by quenching and tempering. This steel performed so well when introduced in the 1930s, it was only used for exhaust valves!

There are other alloys in this family, both up and down in strength and corrosion resistance, like SAE 3140 or 8645 that are only used today for stems. For instance, a Stainless Steel like the commercially known 422 SS (with 12% Chromium plus a bit of Nickel, Molybdenum, Vanadium), after quenching and tempering, has very good corrosion resistance and hardness. But let's say that, in this section, SIL#1 is still king.

Austenitic Steel Alloys

Please allow us a little introduction here… Austenite is a microscopic constituent in steels, formed as a solid solution of Carbon in Iron. It is found in the Iron-Carbon alloys. When heated over a specific temperature depending on the type of steel (usually around 1330-1350ºF), it becomes nonmagnetic at those temperatures due to a modification of the crystalline net of atoms. When high Chromium content is present in the steel alloy and we add Nickel over 1-2% (or Manganese over 12%), the steel becomes austenitic at room temperature, that means that it is nonmagnetic. To increase its hardness and mechanical properties requires a different heat treatment than just heat and quench. This process is known as "precipitation hardening" and requires two steps. First heating at approx. 2100ºF (solution annealing), quenching in water, followed by 14-16 hours at 1400ºF in a furnace (aging). During this aging process, there is a precipitation of chromium carbides

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and other hard carbides at the borders of the microscopic grains of the alloy. This process increases its hardness and strength, as well as its resistance to stress at high temperatures (known as creep resistance).

The most popular steels here are the well-known 21-4N, (or SAE EV8) containing 21% Chromium, 4% Nickel and 9% Manganese, the 21-2N (SAE EV12). Several other similar compositions like the initially called "modified 21-4N" with additions of small percentages of Niobium and Tungsten or the 23-8N (or EV 16) with 23% Chromium and 8% Nickel plus a significant addition of Nitrogen to increase hardness and mechanical properties with and excellent hot corrosion resistance.

Do you know when the famous 21-4N "appeared on scene"? It was after the WW II, when the tetraethyl lead was beginning to be added to gasoline. This process allowed increased compression ratios without experiencing detonation. The highly corrosive lead oxides present in the combustion gases accelerated corrosion and oxidation of the martensitic steels used before. New solutions had to be developed and one of them was this nonmagnetic stainless steel that is still widely used for exhaust valves worldwide. In fact, a few years after its introduction at the OEM production level, the automotive industry discovered that using only 2% less Nickel, the high temperature resistance and mechanical properties were almost not affected. Due to the high cost of Nickel, a reduction in price could be obtained practically with the same properties… and that's how the 21-2N became the most popular exhaust valve steel alloy at both OEM level and aftermarket worldwide and this is still true today.

21-4N and the other slightly modified 21-4N alloys are still today the preferred alloys for exhaust valves, but they are also the preferred intake valves in the racing world. The solution treatment and aging are fine-tuned to match the racing engine requirements as opposed to streetcar requirements. Why is this? Because in a racing engine we need all the high temperature characteristics we've been mentioning, but there is another important factor…the valves must not be fragile. The valve must be able to bend and not break under conditions like over-revving or even minor piston impact. Of course, that target cannot always be hit. If the impacts are hard, or a valve is slightly bent and the engine keeps running, sooner or later a fatigue failure will occur… these steels are marvelous but they still don't perform miracles!

Another important thing to mention about austenitic valve steels is that the aging temperature we mentioned (1400ºF) earlier is approximately the same as the exhaust gas temperatures leaving the combustion chambers in most N.A. engines (even higher temps in turbocharged engines). So, while the engine is running, the aging process is taking place… slowly and constantly, hard carbides are precipitating and making the alloy harder, and at the same time slightly more brittle. That's why in racing it is important to change the valves, mainly the exhaust. This need is dependent upon the application, fuel type, exhaust temperatures, rpms, etc. In Figures 2 and 3, we can see the effects of carbide precipitation (darker zones) in a new valve, and one used in too many races.

High Nickel Alloys

Also known as "superalloys", have been in usage for the last 50 years, exclusively for exhaust valves. The leading reasons were the higher exhaust temperatures generated by the new and constantly increasing emissions controls. Add to that, the usage of EGR and the pressure for leaner fuel-air ratios to reach greater thermal efficiency in our quest for reducing fuel consumption. All these factors made the austenitic steels not good enough for many applications. They had insufficient high temperature strength; hot hardness too low to work in the above-mentioned complex environments. From the very beginning, circa the 1970's, American manufacturers, and some years later some Japanese manufacturers, began using one alloy that is still today one of the best technically speaking for these high temperature environments, Inconel 751 (SAE HEV-3). It contains approx. 72% Nickel, with Chromium at 15.5%, very low Carbon (0.065%), plus several other elements in low percentages (Cobalt, Titanium, Aluminum, etc.) to enhance the hot hardness retention, tensile strength at elevated temperatures, as well as better fatigue and creep resistance.

European manufacturers developed a very similar alloy, which is also in use today called the Nimonic 80 (and Nimonic 90 afterwards), having slightly higher Chromium content (19.5%), but its high temperature properties are almost the same as Inconel, perhaps a little better for specific conditions on diesels applications.

Over the years, new alloys were developed in an effort to achieve certain characteristics for special applications like diesel fuels with high Sulfur content, and at the same time reduce the material cost with somewhat less Nickel content. This generated alloys like the Pyromet 31 V or Waspaloy, but these are with very limited usage in today's engines.

The enhancement of mechanical properties like hardness, stress resistance and fatigue behavior at elevated temperatures are obtained through


Figure 2. Microstructure of 21-4N steel with low carbide precipitation and ductile response after an impact with the piston.

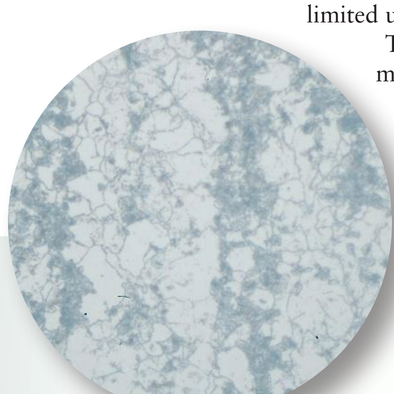


Figure 3. Microstructure of 21-4N steel with high carbide precipitation (darker zones), brittle, with breakage after an impact with the piston.

contains 87-91% Titanium, 5.5-6.5% Aluminum and 3.5-4.5% Vanadium.

For the Exhaust usage, the alloy 6-2-4-2 containing 6% Aluminum, 2% Tin, 4% Zirconium, 2% Molybdenum is used. A very low Silicon content can also be added sometimes to enhance high temperature characteristics. Typical heat treatment, after rough forging at 1900ºF, is followed by stress relief, then air cooling, plus solution treatment and water quench. At the end, the aging process takes 6-8 hours at approximately 1025ºF.

The corrosion resistance of Titanium alloys is well known. They withstand very well, the hot and corrosive combustion gases, but the failure mode is not like austenitic steels. When overheated, they can present brittle fractures with no advance notice. For instance, no stretching before the breakage. For that reason, paying attention to, and keeping track of the too lean conditions or excessively high combustion temperatures will help support the required changing Titanium valves that must be more often than the Stainless-Steel valves.

Another downside of Titanium is that the alloys cannot be used naked against any other mating parts. They require a coating that preserves the contact surfaces from scuffing, galling and abnormal wear. This is often applied on the stems or the whole valve, and we'll explain those coatings in the coating's section.

Titanium Aluminides

During the mid '90s and early 2000s, some new light alloys were developed and quickly found place in Formula 1 engines called the Titanium Aluminides (powder metal Vacuum Remelted Titanium-Aluminum alloys). Weighing closer to pure Aluminum (4 gr/cc), require a double vacuum melting process, extruded bars followed by hot forging, and several steps of heat treatments based on solution and precipitation aging under vacuum. The cost was enormous! The initial problems found in F1 engine valves were the unstable characteristics of the forgings. The early valves did not tolerate the hard impact and high revving conditions of the V10 engines being used. Some failures exposed themselves as some severe brittle breakages and blown-up engines. These problems were finally solved, but the huge cost per valve made the FIA (Federation International de l'Automobile) to prohibit these alloys in 2006, for Formula 1 and other high end European classes.

The Titanium Aluminides are currently used today in aerospace and turbine applications, where there is a steady dynamic output behavior, more constant flow of power and combustion process.

Hard Seat Face Coatings

With the usage of lead-free gasolines, with additives to compensate the withdrawal of tetraethyl lead, the increasing of specific power outputs, turbocharged engines, Direct Fuel Injection, gaseous fuels like LPG, CNG, biogas, diesel fuels with high Sulfur contents, etc., higher hot hardness was required at the valve surface seat faces to withstand indentation, abrasion and adhesive wear usually found in both Intake and Exhaust valve seat faces.

For many years, this protection was afforded by using the Stellite® coatings, mainly with exhaust valves using austenitic head alloys. Today, there are many other alloys that target specific applications which will be explained later this article.

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The basic characteristic that a hard facing coating must exhibit is hot hardness, which means to maintain its hardness even at high temperatures during continuous operation and maximum engine loads. But it is not the only one, because the wear resistance is also a result of the two parts in contact, the valve and the seat insert. The words are "tribological compatibility" between the two surfaces working together with friction, sliding, rotating, and impacting under hot exhaust gases that can reach as high as 1900ºF in some turbocharged applications before the turbo's turbine.

Even though intake temperatures are much lower, some Intake valves also require hard facing due to abrasion wear, like dirty inlet air, fuels compositions (pit natural gas, etc.).

There are four main groups of hard facing alloys, the Stellites® family which are basically Cobalt base alloys containing Cobalt, Chromium, Carbon and Tungsten. Here we have different compositions like Stellite 6, Stellite F, Stellite 1. The most popular is Stellite 6, meanwhile the hardest is Stellite 1. The selection is a careful decision based on the kind of engine, operating temperatures, fuel type, valve seat material, kind of service (light duty, medium duty, severe duty), expected service intervals, ease of access to service working location, etc. (a valve job located up in a mountain mine is not as convenient as in a city shop…).

Some years ago, the welding process was just an oxyacetylene torch and wire, but nowadays the application is automatic welding with a two steps process. First, a round channel is turned on the valve face to accommodate the area required to apply the hard facing material. Then the Stellite® welding process is done through a PTA equipment (Plasma Transferred Arc), that automatically, via a CNC program, generates a torch of melted alloy that impacts the channel while the valve rotates and performs a 360º perfect weld. Figure 4 shows the appearance of said PTA Stellite® welding on a valve before machining and grinding the seat face.

Figure 4. Automatic PTA welding on valve face.

Another hard facing group is also a Cobalt based material, but containing Molybdenum, Chromium and Silicon that generate stable intermetallic compounds. This group is known as Tribaloy® and has the highest hot hardness of all hard facing alloys, usually recommended for gaseous fuels under severe duty services and with highly corrosive environments. It is also applied by the PTA process.

(continued)

The other hard facing alloys are Nickel-based with the addition of Carbon, Tungsten, Molybdenum or Cobalt smaller percentages. The main objective for developing was to offer a lower cost alternative to Cobalt based alloys, with similar performance in specific applications. Here we have the SAE VF 10, VF 11 and Eatonite 6® as regular commercial options.

And then we have the Iron-based alloys, some of them being austenitic stainless steels with high Chromium and Molybdenum percentages, dispersed in an Iron-Chromium-Nickel matrix. These alloys can be a good and lower cost alternative to the expensive Cobalt and Nickel based facings but depends on each application to be carefully selected.

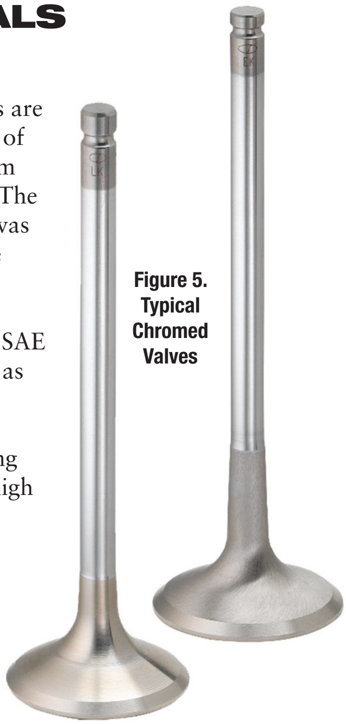

Figure 5. Typical Chromed Valves

As you see, nothing is an easy and good-for-all recipe in the current valves and seats world!

Overall or Stems-only Valve Coatings

In order to reduce the friction coefficient between the stem and the valve guide, and at the same time to increase the surface hardness of the valve stems, mainly under the emissions constraints that reduced the amount of lubrication oil that goes through the guide (take into account that nowadays these two surfaces in contact, under high alternative speeds and lateral forces, have the less amount of oil in the whole engine) several coatings were developed.

The most popular and still having the biggest usage at OEM and aftermarket stem valves worldwide is the hard chromium layer. It possesses a few characteristic properties like relatively high hardness, good corrosion resistance and an oil self-retention effect. (The microscopic structure is full of voids, which helps oil retention on the surface, with a big effect of friction reduction.)

This coating is generally used as “chromium flash”, meaning a thin layer between 2 and 7 microns (0.000000050-0.000000275"). In certain OEM designs, when a higher thickness is specified, it is imperative to grind or lap the stem afterwards, because chromium in thicker layers develops hard nodules that can wear the guides at the fastest rate.

Another important coating, in this case, covering the whole valve including the valve seat surface, is the nitriding, or nitrocarburizing process. These are typically the “black” valves, which go through two or more steps in salt melt baths or gaseous furnaces.

The salt melt consists of alkali cyanate and alkali carbonate; during the process a reaction takes place between the cyanate and carbonate, and the surface of the components, incorporating nitrogen in the crystalline net of the alloyed steels, generating Chromium nitrides, Vanadium nitrides, Tungsten nitrides, etc., depending on the steel alloy. The higher the content of nitride-forming elements, the harder the surface, with a hard zone and a diffusion zone beneath. That’s why we say that it is not a layer, is a compound beneath the surface of the steel, that enhances hardness,

reduces friction coefficient and depending on the salt's combination and steels alloys, can improve the corrosion resistance of said steels.

A second process usually takes place on racing and high-performance austenitic steel valves, following the carbonitriding, which is an additional salt bath to carry out an oxidative treatment that enhances the corrosion resistance on certain steels, and gives the valve surface a slippery and shiny black appearance.

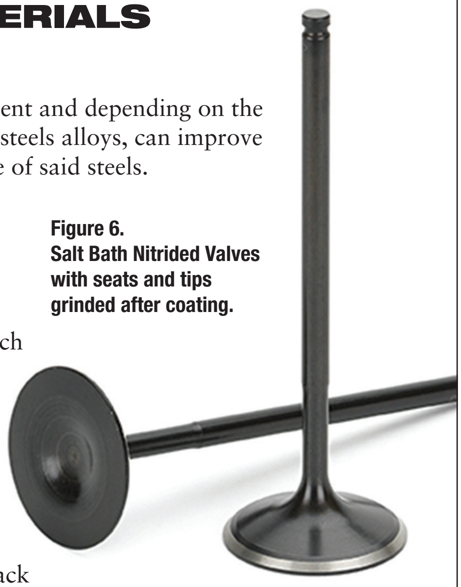

Figure 6. Salt Bath Nitrided Valves with seats and tips grinded after coating.

Similar results can be obtained with a gaseous nitrocarburizing flow inside a furnace, creating an atmosphere rich in Nitrogen and Carbon. The chemical combinations on the valves surfaces increases the hardness and other characteristics as explained above.

Plasma Coatings

Over the years, response to the environmental regulations for cleaner or safer procedures, drove the search for alternative coatings utilizing different material applications (like over titanium, for instance), the "plasma spray" or the "plasma voltage deposition" were developed – like the "jet spray guns", the PVD, (Physical Vapor Deposition), DLC (Diamond-like Carbon), etc.

Let's talk a bit about the basics… Plasma processing is a technique that giving energy to a gas produces the ionization of the atoms and molecules, loosing electrons from their external orbits and generating a flow of particles that impacts to the part to be coated. When these particles are very slippery, like Molybdenum, or hard, like several Nitrides and Carbides that are formed by chemical combination in the high temperature plasma, they are deposited on the surface (like Molybdenum on titanium stem valves), or even penetrating below the surface and interacting with the crystal net of the base material, like with PVD or DLC processes.

The layers so formed have very low friction with the guides, or at the same time high surface hardness like Titanium Nitride (yellow in color), or the nowadays widely used Chromium Nitride (gray color).

The other important advantages for the PVD and DLC processes is the low temperature process for application, not over 800ºF, thus with no modifications at all on the valve material previous heat treatments, and due to their very thin layer (less than 5-6 microns in general for valves), enables to coat a finished valve stem without substantially altering the valve-guide clearance.

During the more recent years, a tendency of CrN via PVD process has gained popularity on the Performance titanium valve market, because it has higher hardness than Moly coating (meaning less wear and better durability), and still very low friction coefficient. Though not so hard as yellow Titanium nitride (TIN), this last one does not work well with powder metal guides or Cast Iron ones.

DLC, on the other hand, has the greatest hardness of all PVD processes (not really a "pure" PVD process, requires

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an additional process to generate chemical reactions between the ions bombing and the base material). Its hardness is so high due to the formation of carbon crystals that are like the diamond structure, but of course, not so hard. With the highest hardness, also comes more brittleness. Being that it is not popular in valve usage because it has a high processing cost compared to the previously mentioned coatings and treatments, we may not see much of the coating outside of racing.

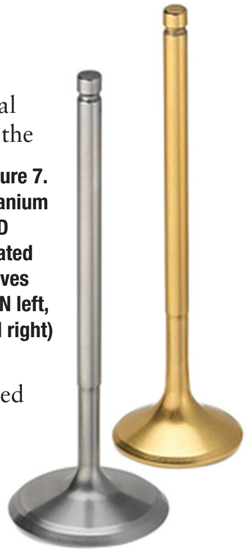

Figure 7. Titanium PVD Coated Valves (CrN left, TIN right)

How do the different processes create the "plasma jet" or ions/electrons beams to impact on the components surfaces to be coated?

The first one mentioned "spray gun plasma", heats an inert gas through combustion to the point of ionization, making electrons from the gas to jump off their orbits, becoming a high temperature jet of particles that melts powder materials to be used as coatings. This mixed jet of particles impacts onto the component surface and forms the thicker layers of coating materials. This is the process to apply the Moly coatings on titanium valve stems. A final grinding is required to get the appropriate low surface roughness and dimensional accuracy for the final valve stem diameter.

The jet spray guns are also used for general applications, like several hard coatings, ceramic coatings, anti-wear coatings, thermal barrier coatings, etc. that are currently in usage for different industries, and have been also used on valves for specific race applications like hard ceramic coatings for valve tips, or seat face coatings for Titanium Aluminide valves, etc.

The PVD, or DLC coatings on the other hand, require a much bigger, expensive, and complex equipment that is called the PVD reactor, in which a high voltage is applied to a gas inside for instance Nitrogen, after a very high vacuum is reached. This high energy ionizes the gas, the high vacuum makes materials like pure Titanium or Chromium to become gaseous and again ionizes, losing electrons from their external orbits and becoming positive particles. A cathodic-anodic reaction takes place, being the components to be coated the cathodic (negative) end, hardly impacted by the positive ions of Nitrogen, Chromium or Titanium, creating the chemical bonds of Chromium Nitrides or Titanium Nitrides inside the components surface and crystal net of said materials.

So, our long-suffering readers, as you have seen in these pages, in a modern engine valve, the material's science and applied technology is unbelievable, so the future is even more exciting, don't lose it and be a part of it! ■

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