TECHCOMMENTARY

ION NITRIDING

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IMPROVE METALLURGICAL PROPERTIES AND INCREASE PRODUCTIVITY USING ION NITRIDING

Heat treaters use a number of surface hardening processes to enhance the wear and fatigue resistance of metal components. The most popular are carburizing and nitriding. In carburizing, carbon is diffused into the surface layers of the workpiece using a special atmosphere or a pack of carbonaceous material in a high-temperature furnace. Similarly, in nitriding, the workpiece surface is alloyed with nitrogen. Conventionally this has been done in an ammonia atmosphere or a special nitrogen-containing salt bath.

A welcome innovation has been ion nitriding which offers many advantages over conventional processes. Ion nitriding produces **more uniform cases**, enables the operator to exercise **greater control** over the process, and is **cost effective**.

This issue of TechCommentary describes the basic concept and advantages of ion nitriding as well as technical and economic factors to consider when deciding if the process could benefit a particular application or product.

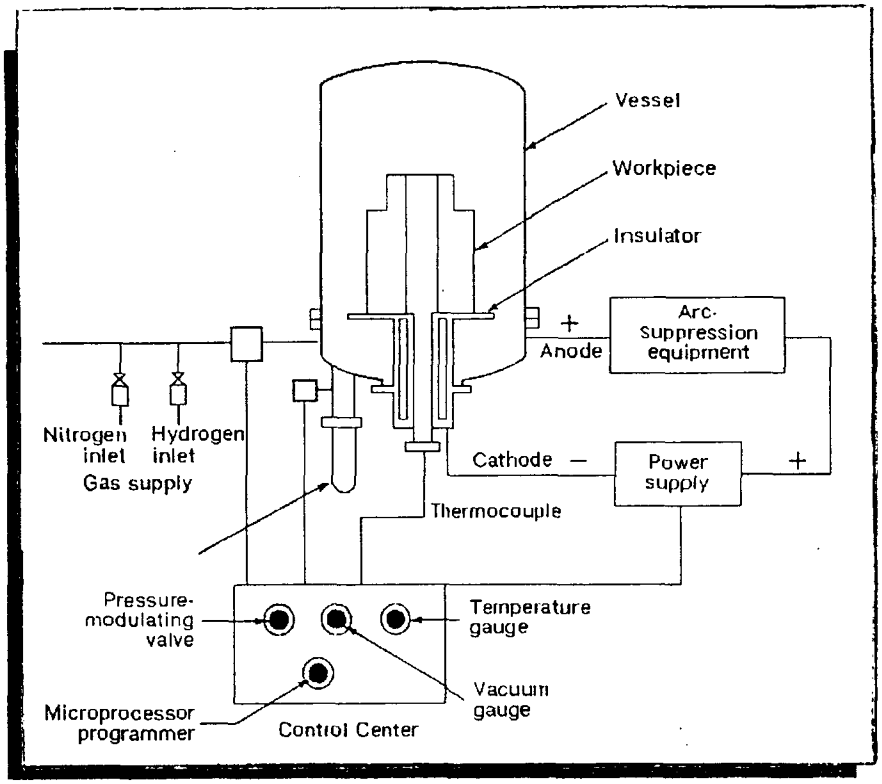

Figure 1. **Schematic arrangement of an ion-nitriding system.**

The Process

The ion nitriding process uses an electrically charged gas of ions to alloy metal surfaces with nitrogen. The process requires a vacuum vessel in which the workpiece becomes the cathode in a dc circuit. The vessel wall becomes the anode. (Figure 1). The vessel is evacuated to remove oxygen and other contaminants, and backfilled with a reactive gas such as an atmosphere containing nitrogen.

When the electric power is turned on, the gas becomes ionized Positive ions strike the workpiece surface and electrons are emitted to the anode producing a glow discharge around the workpiece.

In steel this process forms a solid solution of nitrogen in the surface which then decomposes into ion nitrides Fe_2N, Fe_3N, and Fe_4N. A compound layer consisting of these lower nitrides can also be formed without forming a high energy nitrogen rich (FeN) surface layer. The hardness, thickness and composition of these cases depends on the material being nitrided and the control of the following variables: time, temperature, gas composition, pressure, voltage and current.

Frequently the vessel is initially filled with an inert gas. When power is applied, only sputtering occurs and the workpiece is cleaned. Since parts can be sputter cleaned in the ion nitriding vessel itself, the need for separate cleaning equipment is often eliminated. It is recommended, however, that heavily oiled or dirty parts be cleaned prior to nitriding.

Advantages

Ion nitriding offers numerous advantages over conventional nitriding and carburizing processes including:

Increased Control and Improved Properties — In a conventional nitriding process the furnace is set

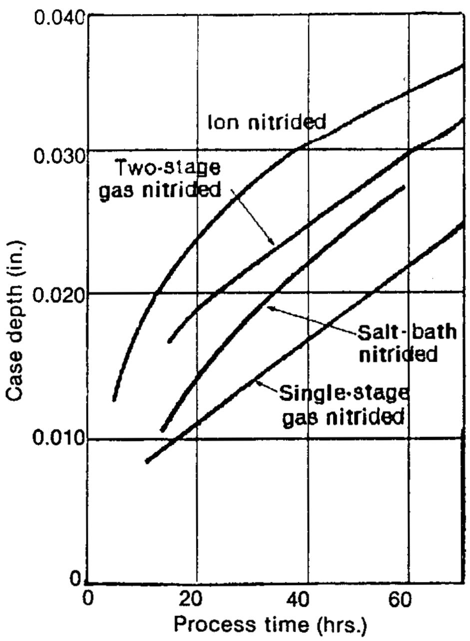

***Figure 2.* Rates of case development for AISI 4340 steel.**

between 975 and 1100 F and the operator controls the length of time and the composition of the gas in the furnace. The compound layer thickness cannot be as easily controlled and contains a mixture of the gamma prime and epsilon crystals. It is brittle and tends to spall or chip off during service.

By contrast, in ion nitriding other parameters such as temperature, time, gas composition, pressure, voltage and current can be controlled. The process can be used to create a diffusion zone of nitrogen dissolved in the surface layers of the workpiece. The result is surface toughness.

By varying the parameters a diffusion zone and a compound layer of either gamma prime or epsilon crystal structures can be achieved resulting in a surface that has both toughness and resistance to wear.

More Uniform Cases — The glow discharge surrounds the part forming a more uniform case and making the process ideal for complex parts such as gears, splines and shafts.

Negligible Thermal Shock and Distortion — Parts are heated to the desired temperature at a preset rate thus avoiding the thermal shock and distortion prevalent in a salt bath process. Since ion nitrided parts do not require quench hardening, as in carburizing, another source of distortion and cracking is eliminated.

Broader Treatment Range — The treatment range is 700 to 1200 F. The workpiece is heated to the desired temperature using the glow discharge and, in some cases, auxiliary electric-resistance heating elements. Lower temperatures help maintain workpiece dimensions during heat treatment. Keeping the temperatures 50 F or more below the tempering temperature of the steel maintains the core hardness of the parts and eliminates the need for any final heat treatment.

Faster Cycle Times — Heat treatment cycle times can be 20 to 50 percent shorter (Figure 2) and can favorably affect productivity.

Lower Energy Consumption — Lower temperatures and faster cycle times reduce energy consumption.

Easier Masking — Mechanical and water-based masking paints are used to leave chosen areas untreated. This avoids masking by electroplating and subsequent stripping procedures.

Increased Safety — Safety problems attendant with the toxic, flammable, or explosive salts or gases used in conventional processes are either eliminated or greatly reduced with the vacuum environment and control systems available.

Typical Applications

The range of products which can be ion nitrided is broad because the process can impart either hardness, toughness or both while maintaining dimensional accuracy. Some typical products which are ion nitrided are listed in Table 1.

Improvements in the service life of the part are often dramatic. (Figure 3). In some reports the service life of hot forging dies has been increased 30 to 50 percent, and cutting tools by a factor of 2 or 4. The spindles for many machine tools require a high degree of dimensional accuracy, making them prime candidates for low treatment-temperature ion nitriding.

Technical Considerations

There are a number of technical factors to consider before deciding to invest in ion-nitriding equipment.

***Table 1.* Typical applications of ion nitriding.**

Part	Attributes Required From Surface Heat Treatment	Optimum Surface Treatment By Ion Nitriding
Plastic molding equipment	Wear resistance	Diffusion zone + gamma prime layer
Machine tools	Wear resistance, dimensional accuracy	Diffusion zone
Drive gears for heavy machinery	Wear and fatigue resistance, nonadhesion properties	Gamma prime layer
Rotary engine housings	Wear resistance, nonadhesion properties	Epsilon layer
Hot forging dies	Wear resistance, toughness	Diffusion zone + gamma prime layer
Cold forging dies, sheet metal stamping dies	Toughness, nonadhesion properties	Diffusion zone
Cutting tools (e.g., drills, taps, end mills, etc.)	Toughness	Diffusion zone
Automotive components (e.g., gears, crankshafts, lifters, valves, rocker-arms, camshafts, etc.)	Fatigue resistance, wear resistance, antigalling resistance	Diffusion zone + epsilon or gamma prime layer

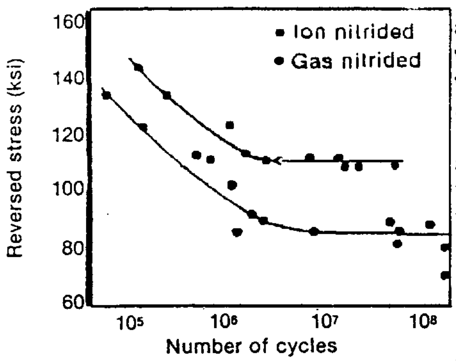

***Figure 3.* Fatigue strength of AISI 4340 surface hardened via conventional gas nitriding or ion nitriding.**

Use—How the part is used determines the case depth, type of compound layer, layer thickness and areas to be hardened. See again Table 1.

Previous Processing—The processing steps that the part has already been subjected to determine whether or not additional steps such as stress-relief annealing or re-tempering are necessary prior to ion nitriding. When deciding on the ion nitriding process, all prior treatments must be considered.

Surface Condition — As with other processes, prior to nitriding, excessive scale or foreign matter must be removed by grit blasting, degreasing, or other methods. Otherwise arcing, common during the sputtering operation, can cause equipment and workpiece damage.

Material Composition—Almost all irons and steels can be ion nitrided. These include:

- Plain-carbon steels and cast irons
- Low-alloy and microalloyed steels
- Tool steels (high speed and hot and cold work die steels)
- Stainless steels (ferritic, austenitic, martensitic, and precipitation hardening)
- Maraging steels.

Depending on the particular material and application, a diffusion zone alone (0.025 in. max.) or in combination with either an epsilon (0.001 in. max.) or a gamma prime (0.0005 in. max.) compound layer is imparted to the workpiece. Steels containing certain alloying elements such as chromium, vanadium, aluminum, silicon, and molybdenum will also form other types of nitrides which increase surface hardness and wear resistance.

Some nonferrous materials can also be ion nitrided. These include titanium alloys which are treated at temperatures of approximately 1600 F.

Geometric Restrictions—To permit adequate development of the glow seam around the workpiece, parts should not be densely packed into the nitriding vessel. Likewise, parts with small crevices or holes cannot be ion nitrided.

Economic Considerations

Extensive cost analyses have yet to be performed for this new process. However, the following factors are important when comparing ion nitriding to conventional nitriding:

Equipment Costs—Ion nitriding equipment is generally more expensive. However auxiliary equipment needed in conventional nitriding such as ammonia dissociators and cooling pits are not required. Floor space requirements can be reduced 50 percent.

Energy Costs—Energy requirements are lower because of reduced process time and temperature. Also the equipment is shut down completely when not in use and need not be maintained at idling temperatures.

Gas Costs—The inert and reactive gas consumption is negligible.

Labor Costs—Handling and stacking prior to treatment is similar in all nitriding processes. However masking and stripping is easier, and ion nitriding requires no washing, grinding to remove a brittle layer, or other handling of parts following treatment. Eliminating washing reduces water and waste disposal costs.

Other Competitive Processes

Before deciding on ion nitriding, there are a number of other surface hardening processes that you should evaluate. The advantages and disadvantages of some other processes are discussed below.

Induction, Laser and Electron Beam Hardening—These methods harden iron and steel surfaces without the use of additional alloying elements or surface coatings. In all cases, the surface is rapidly heated to temperatures of approximately 1500 to 1700 F and is water or self quenched. The final surface hardness is determined by the carbon content of the workpiece material. Because equipment costs are high, such processes are of greatest use in high production operations such as in the automotive industry.

Conventional Processes—Gas and salt bath carburizing, nitriding and carbo-nitriding are economical processes for many applications. Typically they produce case depths of 0.003 to 0.030 in. Ion nitriding is generally selected over these processes when shallow cases (0 to 0.005 in.) are needed, or when distortion or non-uniform case depth must be avoided.

Hard Chromium Plating—Ion nitrided surfaces yield better fatigue properties because of the absence of surface cracks and porosity. They are also harder (Vickers hardness of 1200 vs 830 for hard chromium plating).

Ion Implantation—It can produce very thin cases (0.0003 in. or less) that are quite hard. A focussed high-energy beam of ions penetrates the surface and combines with the part's surface atoms. The maximum temperature rarely exceeds 400 F. The major disadvantage is that only those portions of the workpiece in line with the beam are hardened.

Chemical Vapor Deposition (CVD) and Physical Vapor Deposition (PVD)—These methods apply a thin hard surface coating (less than 0.0003 in.) such as titanium carbide or titanium nitride. CVD uses a chemical reaction and subsequent vapor deposition. PVD uses a sputtering reaction. Process temperatures are 1700-2000 F (CVD) and 500-900 F (PVD). Drawbacks include the need for high processing temperature (CVD) and for ultraclean starting surfaces (PVD).

Ion Nitriding Process Variables

Several parameters must be controlled during processing:

Gas Composition—This determines the composition and

Gas Composition	Type of Surface Layer Obtained
5% Nitrogen, balance inert gas	Diffusion zone only
15 to 30% Nitrogen, balance inert gas	Gamma prime layer, (short times) Gamma prime layer + diffusion zone (longer times)
60 to 70% Nitrogen, 1 to 3% methane, balance inert gas	Epsilon layer + diffusion zone

Table 2. **Typical gas compositions used in ion nitriding.**

properties of the case hardened layer on the part. Table 2 gives typical selections.

Time and Temperature — Higher temperatures require shorter times to achieve a given case depth. Temperatures below 930 F are usually used to produce diffusion zones without a compound layer.

Voltage and Current — A minimum voltage is required for consistent results. The minimum voltage depends on the type and pressure of the gas, electrode spacing, and workpiece material but usually does not exceed 700 volts.

The amount of gas which is ionized and which strikes the workpiece is primarily controlled by the current. Current also affects the workpiece temperature. In general one milliamp per square centimeter of workpiece surface area is used for parts heated by the glow discharge. When auxiliary heating is used, values as low as 0.2 milliamps per square centimeter can be used. Newer systems use computers to control voltage and current.

Pressure — Pressure determines the thickness of the glow discharge seam. Pressure does not influence case depth but only its uniformity. Typical operating pressures are between 1 and 10 torr (1 to 10 mm of mercury). Higher pressures are used to penetrate blind holes and to develop uniform cases on parts such as gear teeth and complex injection molding dies.

In Summary

Ion nitriding is an attractive method for surface hardening steel parts. Its advantages over conventional nitriding and carburizing treatments include better control of surface structure and properties, less distortion, and shorter cycle times. The process is used for machine tools, heavy equipment, automobiles, and injection molding dies for forming plastics.

The information discussed in this issue of TechCommentary is an overview and intended only to familiarize you with the basic aspects of ion nitriding. If you are interested in more detailed background, please contact CMF or one of the manufacturers of ion nitriding equipment. Please refer to the sources listed below for additional information. ☐

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