COURSE EBOOK

Essentials for Beginners



Fundamentals of Plasma Nitriding

Discover the surface hardening technique that delivers top-tier metallurgical results without harming the environment.



Fundamentals of Plasma Nitriding

Discover the surface hardening technique that delivers top-tier metallurgical results without harming the environment.

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Introduction

In the dynamic realm of the metalworking industry, efficiency is crucial for maintaining competitiveness and achieving manufacturing excellence. While plasma nitriding emerged in the 1920s as **an environmentally friendly alternative** to cyanide salt bath nitriding, it is still sometimes regarded as a form of 'modern-day alchemy.'

Far from any mystical connotations, **plasma nitriding is grounded in solid science**, a cutting-edge integration of physics, chemistry, and metallurgy that significantly enhances metal properties, with the added benefit of delivering consistent results. We designed this course to leverage our 40-plus years of expertise as heat treaters and equipment manufacturers, not just to disseminate knowledge but to inspire engineers, technicians, and business leaders to adopt greener practices, **achieve precise targets**, and extend the lifespan of components.

We'll cover the fundamental principles so you can gain a comprehensive understanding of this technology and unlock substantial improvements in your production lines. Have any comments or questions? Feel free to reach out.

Ready to heat things up?

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Plasma Nitriding Furnace Viewport by ION HEAT



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UNIT 1

Preliminary Concepts



1.1. The Need for Hardening in Tool and Machinery Part Manufacturing

Designing the part or tool is step one in the manufacturing process, followed closely by selecting the right raw material—usually a type of steel that meets the performance requirements of the piece. At this initial stage, **'soft steels' are chosen** for their ease of machining.

After shaping, the parts undergo heat treatments to **fine-tune their mechanical properties to match design specifications**, such as:



- Enhancing mechanical strength, fatigue, wear resistance, and consequently, hardness
- Preventing adhesive/abrasive wear to improve load capacity and crack resistance.

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Heat treatment is the process that maximizes steel's potential, transforming the part into a final product that withstands wear and meets the demanding conditions of its intended use.





1.2. Which path to take?

Heat Treatments

VS

Heat treatments, like the well-known hardening and tempering, involve cranking up the heat on parts, holding them at those high temps for a set spell, and then cooling them down fast. **This thermal jolt shuffles the steel's atoms**, meaning a switch-up in its crystal grid.

Based on how hot things get, how long they stay there, and how quick the cool-down is, you can end up mainly with one of three types of structures:



FERRITE

That's a cubic crystal setup where atoms chill at the body's center, rocking a low hardness but high ductility phase.



AUSTENITE

This face-centered act allows more carbon to dissolve in iron, making for a more even-keeled and packed structure that means more hardness and strength than ferrite, though it dials back on ductility.



MARTENSITE

Now that's an uber-hard phase, but it's also brittle as all get-out, freezing atoms in a kind of twisted standoff.

Thermochemical Treatments

Thermochemical treatments, such as carburizing, carbonitriding, cementation, nitriding, and nitrocarburizing, involve not only precise temperature control but also the alteration of the steel's surface chemical composition. This combination expands the spectrum of potential benefits and applications.

Broadly, thermochemical treatments are grounded in the **diffusion of atoms through the metal's vacancies** (those minuscule imperfections or empty spaces within the crystal lattice). It's critical to point out that only exceedingly small atoms—like Carbon, Hydrogen, Nitrogen, and Oxygen—are able to migrate through these interstitial spaces.

These 'incoming atoms' bond with the intrinsic alloying elements of the steel, forming hard alloys or compounds that enhance its mechanical properties.

Heat treatments lead to a 'phase change.' We're talking about the metal's crystalline structure getting a makeover during temperature shifts, tweaking the metal's core properties. While that's central to boosting hardness, toughness, and strength in parts, it throws us a few curveballs:



THERMAL EXPANSION AND CONTRACTION: When things heat up and cool off, the metal swells and shrinks, leading to size shifts and stirring up internal stress that can throw the part out of whack.



UNEVEN PHASE

TRANSFORMATION: That's internal stress coming from not-so-uniform expansion or contraction.



HIT-OR-MISS HEAT DISTRIBUTION that can leave

That's why most heat treatments call for follow-up moves, like grinding, to straighten out the geometry, surfaces, and dimensions. Carburizing and carbonitriding are high-temperature treatments, thus classified as austenitic processes. This entails a phase transformation, which can result in part deformation and slight dimensional changes.

Conversely, **nitriding and nitrocarburizing are regarded as ferritic processes** since they can be executed at lower temperatures within the ferritic phase zone. Their principle relies on bombarding the metal surface with nitrogen ions, which then diffuse into the surface, tailoring its properties.

The endgame of this process is the creation of an exterior layer whose chemical makeup is distinct from that of the core.

> This achieves a combination of properties: a surface that's hard and wear-resistant, with a core that remains tough.

Key points to note:

- The absence of a phase transformation means dimensional changes are virtually nonexistent.
- Fatigue resistance is bolstered by the compressive stresses on the surface, brought on by the diffusion of nitrogen atoms into areas where they previously weren't.

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Download the summary infographic on this topic in PDF format: Thermal Processes Vs. Thermochemical Processes, Which Path to Take?





1.3 What is plasma?

In our everyday experience, we're typically familiar with three states of matter: solid, liquid, and gas. They differ in how molecules interact and attract each other.



The fourth state of matter is plasma. Ionization means that electrons are separated from atoms, creating a **mix of electrically charged particles, known as ions.** Unlike gases, plasma acquires unique properties, such as electrical conductivity, magnetic field generation, light emission, and high kinetic energy.

Plasma is the most abundant state of matter in the universe, present in stars and the intergalactic medium. In materials engineering, specifically in Plasma Nitriding, its applications are revolutionary for **altering the chemical composition and enhancing the properties of metals.**

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Download the summary infographic on this topic in PDF format: What is Plasma?



1.4 Plasma in nitriding

In the context of thermochemical processes, Plasma Nitriding involves forming a mixture of nitrogen and hydrogen gases and subjecting it to enough energy to release electrons from its atoms or molecules (turning it into plasma).

During the process, a set of electrically charged particles is created, composed of positive ions and free electrons. This is achieved by generating a magnetic field that accelerates the ions toward the surface of the part, which then diffuse into its vacancies, forming nitride compounds with the metal's alloying elements and, consequently, a layer that is harder than the base material. This enhances wear resistance, fatigue strength, and corrosion resistance.



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In summary...

What you should take away from this unit as an introduction to Plasma Nitriding:

/ It is a thermochemical process.

In plasma nitriding, there are NO phase changes in the metal structure.

This is because the process operates under a ferritic temperature, which does not alter the body-centered crystalline structure of the metal atoms.

Austenitic stainless steels can be plasma nitrided without losing their stainless properties.

Running at a 'low' temperature, the deformations of the part are almost nonexistent, so post-process grinding is usually not necessary.

The process is based on the diffusion of nitrogen ions into the vacancies of the metal's crystalline structure.

Vacancies' are small irregularities or empty spaces inherent in the crystalline lattice of metals.

One technique for diffusing nitrogen into the metal surface is plasma-assisted nitriding.







At ION HEAT, we're manufacturers of plasma nitriding furnaces and retrofits.

UNIT 2

Plasma Nitriding and Expected Results

In the world of industrial heat treatments, Plasma Nitriding has marked a before and after in surface engineering. This Unit explains the reactions that occur in the parts up to the **formation of the** **nitrided layer**, which is what Plasma Nitriding really aims for.

Let's start with the Top 10 fundamentals that will pave your way to fully understand this process

2.1 Plasma Nitriding in 10 Tips



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Download the summary infographic on this topic in PDF format: Plasma Nitriding Fundamentals





04	LAYER DEPTH RANGES?	The process forms a high hardness surface layer that can measure from a couple of microns in highly alloyed materials, up to 0.7 mm in low alloy materials.	``	
05	WHAT IS THE PROCESS TEMPERATURE?	For steel hardening, the process operates at a 'low' temperature, in the range of 350°C – 600°C (662°F - 1112°F). On the other hand, plasma nitriding of titanium does require a higher temperature: 850°C (1562°F).		
06 💽	DOES THE PROCESS PRODUCE PHASE CHANGES?	At the temperature at which the process is maintained, the diffusion of nitrogen occurs in the ferritic phase of the steel , so no phase transformation occurs during the cooling of the substrate.	Ċ	
07	WHAT HARDNESS IS EXPECTED?	The obtained surface hardness largely depends on the metal's composition and microstructure. This can range from 400 HV in carbon steels to 1400 HV in highly alloyed grades.	Û	
08	WHAT ARE THE RECOMMENDED USES?	Plasma Nitriding guarantees excellent results in components that need adhesive wear resistance and fatigue properties, require masking of certain areas, and where final grinding is not desired.	r L	
09	IS THE PROCESS ENVIRONMENTALLY FRIENDLY?	Yes, because it uses hydrogen and nitrogen as process gases. These are not corrosive and ensure an efficient and clean process. It is worth highlighting that Plasma Nitriding does not use toxic gases or chemi- cals, like ammonia or cyanide salts.		
10	WHAT ARE THE ADVANTAGES? Plasma Nitriding is a highly advanced process, and the equipment which it is carried out has a high technological development in i instrumentation. This means that simply applying the correct re can achieve repeatable results, including:		Ĩů	
		↑ Increases ↓ Decreases ↓ Avoids		
		Surface hardness and resistance to wear, fatigue, and corrosion.Corrosion, adhesive wear, wear, and premature failures.Distortions from conventional heat treatment and additional grindings.		





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2.2 How does plasma nitriding work?

The furnaces in which Plasma Nitriding is carried out are **exceptional pieces of engineering** and the main stars behind the process's success. At ION HEAT, we manufacture hot wall furnaces that deliver excellent metallurgical results.

In very broad terms, one of these furnaces is equipped with:



A chamber (bell or retort) where the parts to be nitrided are introduced. •—



Gas panel that injects the exact gas mixture defined in the metallurgical recipe.

Pumping system that creates a vacuum in the



Bipolar pulse power suppl the electrica percessary to

Bipolar pulsed plasma power supply, generating the electrical power necessary to ionize the gases in the mixture.



Heating/ventilation sources that maintain a uniform process temperature.



Software that controls all the process variables. •—





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Now, the steps in the nitriding process are:







2.3 Reactions occurring on the part's surface during nitriding

In the previous lesson, we reviewed what the Plasma Nitriding team needs to do to generate nitrogen ions and diffuse them into the surface of the parts to be nitrided Now, we'll focus on the reactions that occur on the parts' surface during the process, which allow achieving that **layer of great hardness**. We can mention three stages:



STAGE 1: SPUTTERING

After machining (before starting the nitriding process), the parts' surfaces often contain non-uniform layers of material and various contaminants, such as oxides, organic residues, greases, dust deposits, and particles.

However, when ionic bombardment begins, the ions themselves prepare **the piece for homogeneous and efficient metallurgical results. Their impact, aided by a reducing reaction with hydrogen, removes iron oxides and other contaminants** present on the metal surface. This action is known as sputtering.

STAGE 2: LAYER FORMATION

As a result of the ions' impact and the presence of atomic nitrogen on the surface, the formation of the iron nitride layer (FeN) begins. This occurs when nitrogen ions diffuse through the surface of the part.

STAGE 3: DECOMPOSITION OF FeN

The continuous plasma bombardment causes the instability of FeN, which ends up decomposing as follows:

- $2\text{FeN} \rightarrow \text{Fe}_2\text{N} + \text{N}$
- $3Fe_2N \rightarrow 2Fe_3N + N$ (e-phase)
- $4Fe_{3}N \rightarrow 3Fe_{4}N + N (g' phase)$
- $Fe_4N \rightarrow 4Fe + N$ (Iron/nitrogen compound zone)

This layer of iron, mainly (Fe_3N and Fe_4N), and other nitrides, are responsible for **creating the 'Compound Zone' or 'White Layer,'** which is a kind of ceramic mantle of high hardness, low coefficient of friction, and corrosion resistance.





2.4 Unveiling the Compound Zone (White Layer).

When observing a cross-section of a part that has undergone Plasma Nitriding under a microscope, two zones can be distinguished:





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Here it's important to ask: In metallurgical terms, **is the white layer advantageous or disadvantageous?**

It's a fact that surface hardness and impact resistance are inversely proportional. Therefore, the White Layer has advantages and disadvantages, making it suitable only for certain applications.



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#### ADVANTAGES

- Increased surface hardness: Significantly enhances wear resistance to sliding.
- Reduced friction: Especially beneficial for parts requiring low-friction properties.
- Improved corrosion resistance: Increases surface corrosion resistance.

#### DISADVANTAGES

- Potential brittleness: May increase brittleness, reducing impact resistance.
- Machining difficulty: The hardness may make post-treatment machining challenging.
  - Inconsistency: Thickness and properties may vary depending on nitriding conditions, affecting performance.

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- Components requiring high wear resistance: Gears, bearings, brake discs.
- **Replacement for hard chrome:** When surface hardness and corrosion resistance are crucial.
- Not recommended for components subjected to high impact loads due to potential brittleness.

#### APPLICATIONS THAT BENEFIT

- Gears and transmission components: To improve wear resistance.
- Some cutting tools: To maintain edge and durability.
- Sliding surfaces:
   Where low friction and high wear resistance are needed.





### 2.5 Industrial applications.

It's clear that Plasma Nitriding is used across a wide range of applications where enhancing the durability and performance of metal components under demanding operating conditions (combined properties) is necessary. Adding to this the fact that nitriding equipment features precision technology that ensures repeatable and standardizable results, we can understand why industries requiring quality certifications (such as Nadcap) are increasingly attracted to 'upgrading to plasma'.

SOME OF THESE INDUSTRIES INCLUDE:



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### In summary...

#### What you should take away from this unit:

- Plasma Nitriding can be used on parts made of steels (including stainless), titanium, iron castings, and nickel alloys.
- The main goal of the process is to achieve a high-hardness surface layer without altering the properties of the core.
- $\heartsuit$  The maximum thickness of the nitrided layer is 0.7 mm.
- $\sim$  The process temperature is considered 'low', as it ranges between 350 600 °C for steels.
- The obtained surface hardness depends on the metal's composition and the initial hardness, ranging from 400 HV in plain carbon steels to 1400 HV in highly alloyed grades.
- Plasma Nitriding guarantees excellent results for parts requiring improved properties of resistance to adhesive wear and fatigue.
- The principle of the process is ionic bombardment. This requires a nitriding furnace that features: a) a vacuum chamber with a controlled, low-pressure atmosphere of nitrogen  $(N_2)$  and hydrogen gas  $(H_2)$ . b) A heating system that provides a uniform temperature. c) An electrical power source that ionizes the gas. d) Software that controls all the process variables.
- Ionic bombardment is key for: a) Performing sputtering to clean the part to be treated. b) Diffusing nitrogen ions into the vacancies of the steel's crystalline structure. c) Decomposing iron nitride layers into high hardness phases, with low friction coefficient and increased corrosion resistance.
- The nitrided layer is composed of two zones: The outermost layer, just 25 micrometers thick, with high hardness and resistance, known as the 'white layer'. And the Diffusion Zone, up to 0.7 mm thick, highly resistant to fatigue and wear. These two zones complement each other to provide exceptional mechanical properties in critical components and demanding mechanisms.
- Plasma Nitriding is recommended for gears, bearings, brake discs, injection molds, biocompatible components, and parts requiring sliding surfaces with high wear resistance. However, it is not indicated for parts that require high impact resistance.

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Download the summary infographics on these topics in PDF format: • Reactions that occur at the surface of the part during nitriding.

- Unveiling the Compound Zone (White layer).
- White layer, asset or liability?
- Which industries have upgraded to plasma nitriding?





An aluminum extrusion die undergoing plasma nitriding in a NitrEos machine by ION HEAT.

# UNIT 3

# Optimization and Customization of Nitriding Processes

Plasma nitriding is a technique of proven versatility that can be fine-tuned to fulfill specific criteria. As highlighted, it enables the creation of detailed formulas, consistent outcomes, and exceptional metallurgical quality in certain types of components, notably those that require

# enhanced properties for resistance to adhesive wear and fatigue.

Therefore, to finely conclude this topic and this course, it's crucial to address three key points that you should contemplate before deciding to 'upgrade' to plasma nitriding

# 3.1 Comparison with alternative nitriding methodsProcesses

The most well-known methods for diffusing nitrogen into the surfaces of metal parts are **gas nitriding, cyanide salt baths, and plasma nitriding.** While the latter offers unique advantages in terms of precision, control, and surface treatment quality, there is still resistance among traditional heat treaters towards technological migration.

Some reasons that explain this phenomenon include: the familiarity within the industry with 'time-honored' methods, the high initial investment required for acquiring plasma nitriding equipment, and the mistaken belief that this is a complex process to implement.

Among supporters and critics of each of the technologies, there's a kind of 'clash of titans' where there are no winners or losers. Therefore, before deciding on one of them, the healthiest and most responsible approach is to have a general overview of their advantages and disadvantages.

It's important to clarify that each method has its merits and optimal applications.

# THE FOLLOWING TABLE COMPARES THEM IN BROAD STROKES:











	GAS	SALT BATH	PLASMA
PROCESS DESCRIPTION	It involves exposing steel parts to an ammonia-rich atmosphere at elevated temperatures. The gas dissociates, releasing nitrogen atoms that diffuse across the material's surface.	Submerging metal parts in a cyanide salt bath. At high temperatures, the salts dissociate into cyanide and sodium ions. The cyanide ions are the source of nitrogen and carbon that diffuse onto the surface.	It consists of subjecting the parts to a bombardment of nitrogen ions, which are absorbed by the surface and form nitride compounds with the alloying elements of the material.
PRINCIPLE	NH3   Ammonia Diffusion $2NH_3 \rightarrow N_2 + 3H_2$	NaCN   Cyanide Salt Diffusion $4NaCNO \rightarrow 2NaCN + Na_2CO_3 + CO + 2N$ $3Fe + 2CO \rightarrow Fe_3C + CO_2$	N+   Nitrogen Ion Bombardment Electrically Charged Gas
		A CONTRACTION OF CONT	$\rightarrow \bigcirc \bigcirc$
TEMPERATURE	<b>520 - 600°C /</b> 932 - 1112°F	<b>570 - 620°C</b> / 1058 - 1148°F	<b>350-800°C /</b> 662-1382 °F
PROCESS TIME	10-120 hours	2-4 hours	Hasta 120 hours
NITRIDED LAYER DEPTH	<b>0.05 - 0.7 mm</b> (50 - 700 micrometers)	<b>0.05 - 0.15 mm</b> (50 - 150 micrometers)	<b>0.05 - 0.7 mm</b> (50 - 700 micrometers)

* General estimates. The process variables may vary depending on the material, the geometry of the part, and the expected outcomes.











	GAS	SALT BATH	PLASMA
ADVANTAGES	<ul> <li>Deep nitrided layers.</li> <li>Flexibility in part size and ease of furnace loading.</li> <li>Capability for both nitriding and FNC (Ferritic Nitrocarburizing).</li> </ul>	<ul> <li>Short treatment times.</li> <li>Thick white layer.</li> <li>Cost-effective solution.</li> </ul>	<ul> <li>Deep nitrided layers.</li> <li>Sputtering cleaning.</li> <li>Repeatable results.</li> <li>Environmentally friendly gases.</li> <li>Low decarburization.</li> <li>Low gas consumption.</li> <li>Easy masking of selected surfaces.</li> <li>Capability for both nitriding and FNC (Ferritic Nitrocarburizing).</li> </ul>
DISADVANTAGES	<ul> <li>Use of corrosive gases.</li> <li>No cleaning effect.</li> <li>Hard to nitride stainless steels.</li> <li>Not easy to control thermodynamic reactions.</li> <li>High gas consumption.</li> </ul>	<ul> <li>Parts with unsealed seams or complex geometries can trap the saline solution, even after rigorous rinsing.</li> <li>Corrosion/pitting in case of prolonged immersion.</li> <li>Shallow case depth.</li> <li>Banned in some places due to pollution.</li> <li>Poisonous.</li> </ul>	<ul> <li>Deep holes and cuts.</li> <li>Hollow cathode + Edge effects.</li> <li>Furnace loading requires attention to detail.</li> </ul>

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Download the summary infographic on this topic in PDF format: What is the best nitriding method?



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# 2.5 The difference between nitriding and ferritic nitrocarburizing

There's a 'sibling' process to nitriding: ferritic nitrocarburizing. They have in common that both are surface treatment processes that involve ion diffusion on the part's surface. **The difference lies in that, in ferritic nitrocarburizing, carbon is used in addition to nitrogen, in order to increase the thickness of the white layer,** a useful property in parts that will be exposed to **corrosive environ**- ments or where adhesion resistance is a critical factor, such as in valves, shafts, and food industry equipment.

We recommend familiarizing yourself with the differences between these processes to get the most out of your equipment and achieve better results:









	NITRIDING	FERRITIC NITROCARBURIZING
ELEMENTS TO DIFFUSE	Nitrogen	N Nitrogen + C Carbon
OBJECTIVE	<ul> <li>Formation of a fine and hard nitride compound on the surface, as well as a good and strong diffusion zone underneath, with the purpose of enhancing:</li> <li>Surface hardness.</li> <li>Load capacity.</li> <li>Wear resistance.</li> <li>Fatigue resistance.</li> </ul>	<ul> <li>Formation of hard nitrides and carbides on the surface, to increase the thickness of the white layer and improve:</li> <li>Load capacity.</li> <li>Fatigue resistance.</li> <li>Adhesive wear resistance.</li> <li>Corrosion resistance.</li> <li>Lubricity.</li> </ul>
PROCESS	Focused on the diffusion zone rather than the white layer.	Creates a thicker white layer and a less deep diffusion zone than nitriding. The introduction of carbon into the surface saturates the chemical composition of the steel, turning it into a compound.
MATERIALS	Medium to high carbon steels   Iron castings   Stainless steels   Nickel alloys   Titanium.	Low carbon steels Iron castings.
CONSIDERATIONS	When the best relationship between hardness and ductility is sought in parts with high fatigue resistance.	Aims to improve sliding properties and corrosion resistance.
APPLICATIONS	Gears, camshafts, cutting tools, engine components, drills, etc.	In addition to the above, parts exposed to corrosive environments, such as valves, shafts, and food industry equipment.
SUITABLE MECHANISM	Rotational fatigue resistance, load capacity.	Sliding wear.
PROCESS TEMPERATURE	390°C - 530°C Up to 800°C for Titanium	550°C - 650°C







# 3.3 Choosing gases for nitriding

This topic is important because selecting the gases and their specific proportions is key to controlling the process, tailoring the properties of the nitrided layer, and achieving desired outcomes in various materials and applications.

Furthermore, it's worth noting that **plasma nitriding does NOT use corrosive gases**, an

advantage that impacts the lifespan of the equipment, reduces maintenance costs, and ensures an environmentally friendly process, enhancing quality standards and metallurgical results.

THE GASES USED IN PLASMA NITRIDING ARE:









#### NITROGEN Primary gas

When subjected to an electrical discharge in a vacuum chamber, the nitrogen molecule dissociates, and atomic nitrogen is ionized to create a plasma containing active nitrogen species ( $N_2$ +),  $N_2$ -,  $N_4$ + y  $N^{2*}$ , which form nitrides on the part's surface, improving its hardness and wear resistance, among other properties.





#### HYDROGEN Transport and dilution

Hydrogen acts as a reducing gas and, when ionized, helps to activate the nitrogen gas by providing free electrons. It helps to dilute the nitrogen to the correct nitriding potential and assists in reducing oxides present on the surface, thereby activating the surface for further nitriding.





#### ARGON Carrier & sputtering

Provides a medium for the transport of reactive species, helps stabilize the electrical discharge and ensure that the plasma remains consistent during the process. Ar is a heavier atom, thus increasing the kinetic energy with which it hits the surface creating a stronger sputtering effect.





#### **METHANE** Process gas

It is a carbon donor gas for plasma FNC or ferritic nitrocarburizing. The atoms of Carbon and Nitrogen form hard compounds in the surface layer of the material, making steel harder and more wear-resistant. Methane is used in low quantities, therefore a single bottle will probably last for a couple years.







# As a closing note...

Throughout this course, we've delved into the basic principles of plasma nitriding, facing its complexities and benefits in the field of surface hardening head-on. As we prepare to close this chapter of learning, we leave on the desk a reflection on the importance of combining efforts to lead the metalworking industry toward adopting cleaner processes.

In a world increasingly aware of sustainability, our industry is called to reduce the emission of harmful gases and the use of dangerous chemicals, without sacrificing the demand for precise, reliable, and competitive results. In this light, learning about plasma nitriding is more relevant than ever.

As manufacturers of plasma nitriding furnaces and providers of heat treatment/thermochemical services, we hope to be contributing to the improvement of industrial practices, towards a cleaner and more prosperous world for the generations to come.





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