Vacuum Heat Treating of Fastener Materials

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Vacuum processing is a highly repeatable heat treating method and one that results in optimum surface finish.

Certain types of fastener materials, especially those made from stainless steels and certain superalloy materials, either require or can benefit from processing in vacuum as opposed to using a protective atmosphere. In general, applications involving vacuum heat treating can be broken down into three main categories:

- Processes that can be done in no other way than in vacuum.
- Processes that can be done better in vacuum from a metallurgical standpoint.
- Processes that can be done better in vacuum from an economic standpoint.

A principal difference between vacuum heat-treating and all other forms of heat treatment is the absence of, or the precise control of, surface reactions. In addition, vacuum processing can remove contaminants, and under certain circumstances, de-gas or convert oxides found on the surface of a material.

What is Vacuum?

The word vacuum comes from the Latin, "vacuus" which means empty or "vacare" meaning to be empty. When we think of an empty space, what comes to mind is something entirely devoid of matter. Such a space does not exist nor can it be produced. In practical terms then, a vacuum must be considered a space with a highly reduced gas density. In heat treating, gas molecules and contaminants are removed from a vacuum vessel using a pump. Air is the most important of all gases to be eliminated since it is present in every system (**Table 1**).

A vacuum system, like the typical vacuum system seen in **Figure 1**, provides a space in which the pressure can be maintained below atmospheric pressure at all times. The primary advantage of a vacuum heat treatment is its versatility. In almost all cases it provides a "safe" environment with respect to the surface of the components being treated, is self-contained and uses cycles/recipes that can be reproduced consistently. When not in use, like an electric light, it is simply turned off. When turned back on, minimal conditioning time is required. A typical vacuum furnace shop is seen in **Figure 2**.

Table 1.[1] Composition of Air.

Gas Present in Air	Volume (approximate)
Nitrogen (N_2)	78.1
Oxygen (O)	20.9
Argon (Ar)	0.9
Carbon Dioxide (CO_2)	0.03
Other Gases (Neon, Helium, Methane, Krypton, Hydrogen, Nitrogen Dioxide, Xenon	0.07

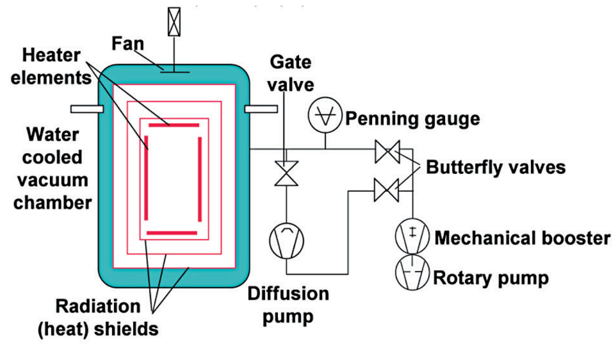

Fig. 1 — Typical vacuum system.

Fig. 2 — Typical vacuum furnace shop (photo courtesy of Nevada Heat Treating, Inc.).

Vacuum Hardening

The various vacuum hardening technologies for fasteners will be discussed by type of material in the following paragraphs.

Hardening by Oil Quenching (Plain Carbon and Alloy Steels). Oil quenching takes place in horizontal vacuum furnaces equipped with integral quench tanks (**Figure 3**). The design of the quench tank is similar to its atmosphere counterpart—fixed or variable-speed oil circulation via agitators or pumps located on one or both sides of the tank and internal baffles to guide the respective oil flow around and through the load. Cold or preheated oil, in the 120°F to 150°F (50°C to 65°C) range, are the most common, and special (hot) oils, which run at 275°F to 350°F (135°C to 175°C) have been used with success. Heaters control the oil temperature and the oil is cooled via double-wall construction or external heat exchangers usually employing air, for safety reasons.

Vacuum quench oils are distilled and fractionated to a higher purity than normal quench oils, which is important in producing the better surface appearance of quenched parts. In practice, the quenching in vacuum furnaces is frequently done with a partial pressure of nitrogen above the oil between 400 and 500 torr (540 and 675 mbar). In general, high partial pressures above the oil can be advantageous in producing full hardness on unalloyed or very low-alloy materials, whereas low pressures above the oil produce higher hardness and lower distortions on components made of medium or highly alloyed steels.

Medium alloy steels (**Table 2**) and most case hardening steels are hardened either by oil quenching or high gas pressure quenching (up to 20 bar).

Hardening by Gas Quenching (Alloy Steels). Hardening by inert gas pressure quenching (see **Figure 4**) at pressures of 2 to 20 bar is the most popular method of quenching in vacuum furnaces. Nitrogen and then argon are the most common quenching gases. Cooling in argon produces the slowest heat transfer rates, followed by nitrogen, then helium and finally hydrogen. All these gas mixtures are used, but nitrogen is the most attractive especially from a cost standpoint, but there are limitations with certain alloys (e.g., titanium). Theoretically, there is no limit to the improvement in cooling rate that can be obtained by increasing gas velocity and pressure. Practically, however, very high-pressure and very high-velocity systems are complex and costly to construct. In general, the power required for gas recirculation increases faster than benefits accrue.

The trend today is to "dial in" the quench pressure, that is, use only the highest pressure required to properly transform the material. Recent changes in material chemistry and pressure quench design (e.g., alternating gas flows, directionally adjustable blades and variable speed drives) have made this possible, and gas quenching is now used to produce full hardness in many traditional oil hardening materials.

Fig. 3 — Two-chamber oil quench vacuum furnace (photo courtesy of SECO/WARWICK Corporation).

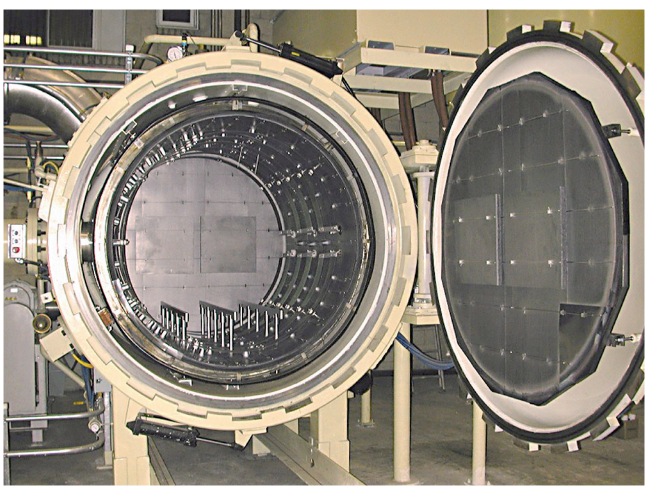

Fig. 4 — Typical vacuum pressure quench furnace (photo courtesy of Vac-Aero International).

Martensitic Stainless Steels

All grades of martensitic stainless steel fastener grades can be processed in vacuum furnaces using similar austenitizing

Table 2.[2] Quenchant Type for Select Alloy Steels.

Material Grade (AISI/SAE)	Hardening Temperature [a] °F (°C)	Quench Medium [b]	Typical Surface Hardness [c] (HRC) diameter values in inches (mm)			
			0.5 (12)	1 (25)	2 (50)	4 (100)
1340	1525 (830)	OQ, PQ	58	57	39	32
3140	1525 (830)	OQ, PQ	57	55	46	34
4140	1570 (855)	OQ, PQ	57	55	49	36
4150	1525 (830)	OQ, PQ	64	62	58	47
4340	1475 (800)	OQ, PQ	58	57	56	53
4640	1475 (800)	OQ, PQ	57	54	39	30
6152	1550 (845)	OQ, PQ	61	60	54	42
8740	1525 (830)	OQ, PQ	57	56	52	42
9440	1525 (830)	OQ, PQ	56	51	41	28

Notes:
[a] Austenitizing temperature in vacuum is often 25°F to 50°F (15°C to 30°C) higher than atmosphere processing.
[b] Cooling nomenclature: OQ = oil quench; PQ = pressure quench.

Table 3.[3] Typical Hardening Cycles for Select Martensitic Stainless Steels.

Material Grade (AISI/SAE)	Preheat 1 [a] °F (°C)	Preheat 2 °F (°C)	Hardening Temperature °F (°C)	Quench Medium [b]	Typical Surface Hardness [c] (HRC)
410	1000 (540)	1400 – 1450 (760 – 790)	1695 – 1850 (925 – 1010)	OQ, PQ	40 – 44.5
420	1000 (540)	1400 – 1450 (760 – 790)	1795 – 1950 (980 – 1065)	OQ, PQ	47.5 – 55.5
440A	1000 (540)	1400 – 1450 (760 – 790)	1850 – 1950 (1010 – 1065)	OQ, PQ	52 – 57
440B	1000 (540)	1400 – 1450 (760 – 790)	1850 – 1950 (1010 – 1065)	OQ, PQ	56 - 59
440C	1000 (540)	1400 – 1450 (760 – 790)	1850 – 1950 (1010 – 1065)	OQ, PQ	60 - 62

Notes:
[a] Rapid heating rates can cause distortion and/or cracking. In vacuum heating rates of 15°F/minute to 25°F/minute are recommended for small parts or intricate shapes.
[b] Certain parts will benefit from an initial preheat at the temperatures shown.
[c] Cooling nomenclature: OQ = oil quench; PQ = pressure quench.
d] As quenched (oil) data shown.

temperatures and general considerations as those used in atmosphere furnaces (**Table 3** seen on the previous page of this article).

Since the austenitizing temperatures are usually below 2000°F (1100°C), vacuum levels in the 10^{-3} torr (10^{-3} mbar) range are very often used, which result in clean and bright part surfaces. In order to avoid evaporation of certain alloying elements, processing is also done at vacuum levels ranging from 10^{-1} to 1 torr (10^{-1} to 1.3 mbar), with some sacrifice to brightness.

Because of the differences in the hardenability of the various martensitic stainless alloys, there is a limitation on the section sizes that can be fully hardened by recirculated nitrogen gas quenching.

Other types of cooling gas (for example, helium) can be utilized, but the economic benefits must be carefully considered. The actual values of section size limits depend on the type of cooling system and the capability of the specific furnace employed.

Precipitation Hardening of Stainless Steels

The heat treatment temperature for precipitation hardened stainless steels depends on the particular alloy grade, the type of part/component being treated and the required properties (as seen in **Table 4**).

In several instances, multiple heat treatments are specified. In other cases, such as 17-4 PH stainless steel, material is purchased in the so-called Condition "A" which requires only an aging operation to be performed (this aging operation is typically not done in the vacuum process).

To result in optimum creep and creep rupture properties, the high side of the solution annealing temperature range is typically utilized. For optimum strength during relatively short-term service at high temperatures, a low-end annealing temperature is utilized. A final aging heat treatment results in a finely dispersed precipitate throughout the microstructure, which significantly increases the room-temperature yield strength.

Superalloys

Superalloys covers a wide range of materials—typically nickel, cobalt or iron based—and are generally intended for high-temperature applications. Most of these alloys are hardened using a solution treating and aging process (as seen in **Table 5**). Solution treating involves heating the alloy to a temperature in the range of 1800°F (982°C) or higher, followed by gas quenching. In most cases, a very fast quench speed is not required and gas quenching with nitrogen at pressures of 2 bar or less is often sufficient.

This is followed by aging at an intermediate temperature for extended periods of time. Normally, the complete solution treat and aging cycles can be programmed into the furnace so that unloading is not required between cycles. However, certain superalloys require other special treatments to develop required properties.

Summing Up

If your heat treating needs call for vacuum processing, you will find it to be a highly repeatable process that will produce the best surface finish of all the heat treating methods.

There is certainly a cost factor that must be considered when selecting any heat treatment process. However, it is certainly worth an investigation of performing your heat treating process in vacuum.

To learn more about vacuum heat treating of fastener materials, visit the website listed below.

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Table 4.[3] Typical Solution Heat Treating and Aging Cycles for Select Precipitation Hardening Stainless Steels.

Material Grade (AISI/SAE)	Solution Heat Treat Temperature °F (°C)	Method of Cooling [a], [b]	Aging Temperature °F (°C)	Aging Time (hrs)	Method of Cooling [a], [b]
A-286	1800 (980)	OQ, PQ	1325 (720)	16	AQ
13-8 Mo					
15-5	1900 (1040)	OQ, PQ	900 – 1150 (480 – 620)	4	AQ
17—4 PH	1900 (1040)	OQ, PQ	900 – 1150 (480 – 620)	4	AQ
17 – 7 PH	1925 (1050)	PQ	950 – 1100 (510 – 595)	4	AQ
Custom 455	1525 (830)	WQ, PQ	900 – 1050 (480 – 565)	4	AQ
Rene 41	1975 (1080)	WQ, PQ	-	-	-
Udiment 700	2150 (1175)	PQ	1550 (845)	24	AQ
Waspaloy	1975 (1080)	PQ	1550 (845)	24	AQ

Notes:
[a] Cooling nomenclature: WQ = water quench; OQ = oil quench; PQ = pressure quench; AQ = air cool.

Table 5.[3] Typical Solution Heat Treating and Aging Cycles for Select Wrought Superalloys.

Alloy	Solution Heat Treat Temperature °F (°C)	Solution Heat Treat Time (hrs)	Method of Cooling [a], [b]	Aging Temperature °F (°C)	Aging Time (hrs)	Method of Cooling [a], [b]
A-286	1800 (980)	1	OQ, PQ	1325 (720)	16	AC
Incoloy 925	1850 (1010)	1	AC	1350 (730)	8	FC
Inconel 625	2100 (1150)	2	[c]	-	-	-
Inconel 718	1800 (980)	1	AC	1325 (720)	8	FC
Inconel X-750	2100 (1150)	2	AC	1550 (845)	24	AC
Rene 41	195 (1065)	0.50	AC	1400 (760)	16	AC
Udiment 700	2150 (1175)	4	AC	1550 (845)	24	AC
Waspaloy	1975 (1080)	4	AC	1550 (845)	24	AC

Notes:
[a] Cooling nomenclature: FC = furnace cooling; AC = air cooling; RAC = rapid air cool; OQ = oil quench; PQ = pressure quench.
[b] Air cooling equivalent is defined as cooling at a rate not less than 40°F/minute to 1100°F and not less than 15°F/minute from 1100°F to 1000°F. Below 1000°F any rate may be used.
[c] To provide adequate quenching after solution heat treatment, cool below 1000°F (540°C) rapidly enough to carbide precipitation. Oil or water quenching may be required on thick sections.

References:

[1] *Herring, D. H., What is Vacuum? Industrial Heating, November 2006.*

[2] *Modern Steels and Their Properties, Handbook 268, Bethlehem Steel, 1949.*

[3] *Heat Treater's Guide: Practices and Procedures for Nonferrous Alloys, Candler, Harry (Ed), ASM International, 1996.*