Using Vacuum Technology *for the* Heat Treatment of Fastener Materials

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Many fastener materials, in particular stainless steels and certain superalloys, benefit from or actually require vacuum processing for heat treatment instead of using protective atmospheres. In general, there are three main categories of applications that involve vacuum heat treatment:

1. Processes that can be done in no other way than in vacuum;
2. Processes that can be done better in vacuum from a metallurgical standpoint;
3. Processes that can be done better in vacuum from an economic standpoint.

The absence of surface reactions or the ability to precisely control them is the main difference between vacuum heat treatment, and all other forms of heat treatment. Vacuum processing can also remove contaminants from parts, and in some instances, degas or convert oxides found on the materials surface.

Vacuum Defined

The word vacuum is derived from the Latin "vacuus" meaning 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 the heat-treating industry, gas molecules and contaminants are removed from a vacuum vessel using a pump. Air (Table 1) is the most important gas to be removed, as it is present in every vacuum system.

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

A vacuum system (Fig. 1) provides a space in which the pressure can be maintained below atmospheric pressure at all times. Versatility remains the primary advantage of vacuum heat treatment. In addition to being self-contained, vacuum heat treatment provides a "safe" environment for the surface of the parts being treated and uses consistently reproduced cycles and recipes. When not in use, like an electric light, it is simply turned off. When turned back on, minimal conditioning time is required.

Figure 1. Typical Vacuum System
(Photograph Courtesy of Invensys Eurotherm)

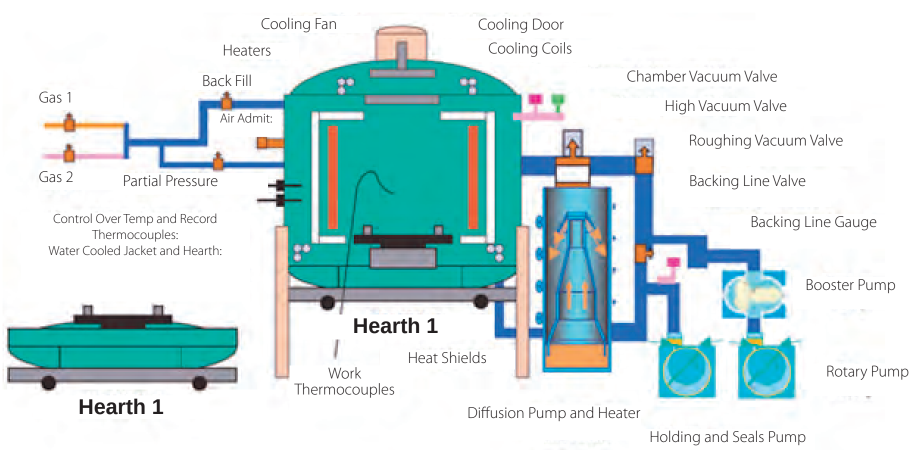

Figure 2. Typical Vacuum Furnace Shop

(a) (b)

(a) Vertical Style Vacuum Furnace (Photograph Courtesy of Vac-Aero International)
(b) Horizontal Style Vacuum Furnaces (Photograph Courtesy of Nevada Heat Treating, Inc.)

Vacuum Hardening

The various equipment (Fig. 2) and processes for vacuum hardening of fasteners will be discussed by type of material.

Hardening by Oil Quenching (Plain Carbon and Alloy Steels)

Oil quenching takes place in horizontal vacuum furnaces equipped with integral quench tanks (Fig. 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 50˚C - 65˚C (120˚F - 150˚F) range, are the most common and special (hot) oils, which run at 135°C - 175°C (275°F - 350°F), 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.

Compared to normal quench oils, vacuum quench oils are distilled and fractionated to higher purity levels. This allows for a better surface appearance on quenched parts. It is common that the partial pressure of nitrogen above the quench oil be between 540 mbar (400 Torr) and 675 mbar (500 Torr). Usually, higher partial pressures above the quench oil can be advantageous in obtaining full hardness on both unalloyed or very low alloy materials. Low partial pressures above the quench oil produce higher hardness values, as well as lower distortion on parts consisting of medium or highly alloyed steels.

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

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

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

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

Hardening by Gas Quenching (Alloy Steels)

The most popular method of quenching used for hardening in vacuum furnaces is inert gas pressure quenching used at pressures of 2 – 20 bar. Nitrogen and argon are the most common quenching gases. Cooling in argon produces the slowest heat transfer rates, followed by nitrogen, then helium and finally hydrogen. Nitrogen is the most attractive gas mixture from a cost perspective, however limitations exist 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 particular, 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. This has been made possible due to recent changes in both material chemistry and pressure quench design (e.g. alternating gas flows, directionally adjustable blades, variable speed drives). Gas quenching is now being used to produce full hardness in many materials that in the past have been traditionally oil quenched.

Martensitic Stainless Steel

All grades of martensitic stainless steel fastener grades can be processed in vacuum furnaces. Austenitizing temperatures and general heat treatment considerations are similar to those used in atmosphere furnaces (Table 3). Since the austenitizing temperatures are usually below 1100°C (2000°F), vacuum levels in the range of 10-3 mbar (10-3 torr) are very often used which result in clean and bright part surfaces. To avoid evaporation of certain alloying elements, vacuum levels in the range of 0.1 – 1.3 mbar (10-1 to 1 torr) are required, resulting in some sacrifice of brightness.

Due to 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 (e.g. helium) can be used 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.

Figure 3. Two Chamber Oil Quench Vacuum Furnace (Photograph Courtesy of SECO/WARWICK Corporation)

Precipitation Hardening of Stainless Steels

Determining the heat treatment temperature for precipitation-hardened stainless steels (Fig. 4) depends on a number of factors such as the alloy grade, the type of parts being treated, and the required mechanical properties (Table 4). It is not uncommon, for multiple heat treatment steps to be specified. In other cases, material is purchased in the so-called Condition "A" requiring only an aging operation to be performed (this is typically not done in vacuum). For optimum creep and creep rupture properties, the high side of the solution annealing temperature range is typically used. A low-end annealing temperature is used to obtain optimum strength during relatively short-term service at high temperatures. A final aging heat treatment produces a finely dispersed precipitate throughout the microstructure significantly increasing the room-temperature yield strength.

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

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

Notes
[a] Rapid heating rates can cause distortion and/or cracking. In vacuum heating rates of 8°C (15°F)/minute - 15°C (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.

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

Material Grade (AISI/SAE)	Solution Heat Treat Temperature °C (°F)	Method of Cooling [a], [b]	Aging Temperature °C (°F)	Aging Time (hrs)	Method of Cooling [a], [b]
A-286	980 (1800)	OQ, PQ	720 (1325)	16	AQ
13-8 Mo					
15-5	1040 (1900)	OQ, PQ	480 – 620 (900 – 1150)	4	AQ
17—4 PH	1040 (1900)	OQ, PQ	480 – 620 (900 – 1150)	4	AQ
17 – 7 PH	1050 (1925)	PQ	510 – 595 (950 – 1100)	4	AQ
Custom 455	830 (1525)	WQ, PQ	480 – 565 (900 – 1050)	4	AQ
Rene 41	1080 (1975)	WQ, PQ	-	-	-
Udiment 700	1175 (2150)	PQ	845 (1550)	24	AQ
Waspaloy	1080 (1975)	PQ	845 (1550)	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 °C (°F)	Solution Heat Treat Time (hrs)	Method of Cooling [a], [b]	Aging Temperature °C (°F)	Aging Time (hrs) (小时)	Method of Cooling [a], [b]
A-286	980 (1800)	1	OQ, PQ	720 (1325)	16	AC
Incoloy 925	1010 (1850)	1	AC	730 (1350)	8	FC
Inconel 625	1150 (2100)	2	[c]	-	-	-
Inconel 718	980 (1800)	1	AC	720 (1325)	8	FC
Inconel X-750	1150 (2100)	2	AC	845 (1550)	24	AC
Rene 41	1065 (1950)	0.50	AC	760 (1400)	16	AC
Udiment 700	1175 (2150)	4	AC	845 (1550)	24	AC
Waspaloy	1080 (1975)	4	AC	845 (1550)	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 per minute to 1100°F and not less than 15°F per 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.

Figure 4 Load of Custom 630 Fasteners for Solution Heat Treatment and Aging (Photograph Courtesy of Solar Atmospheres Inc.)

Superalloys

Superalloys cover a wide range of materials; typically nickel, cobalt or iron based alloys are generally intended for high temperature applications with most of them being hardened using a solution treating and aging process (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, gas quenching with nitrogen at a pressure of 2 bar or less is sufficient. This is followed by aging at an intermediate temperature for an extended period of time. In some cases, the complete solution treatment and aging cycles are programmed into the furnace instrumentation so that unloading is not required between cycles. Certain superalloys, however, require other special treatments to develop required properties.

Summing Up

Vacuum processing of fasteners is a highly repeatable process that will produce the best surface finish of all the heat treatment methods. While there is always a cost premium, its benefits with respect to metallurgy, properties and repeatability make it a technology to consider.

References

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