Technology Trends in Vacuum Heat Treating, Part One: Markets, Processes and Applications

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The last decade has seen double-digit growth in the use of vacuum heat treating and increased vacuum market share (Figs. 1–5) throughout the Americas. Vacuum processing is growing more than any other technology, due in large part to the demand for high quality, precision and repeatability of part performance in ever more sophisticated and demanding service applications.

Each of the common vacuum processes will be discussed in this and future articles in this series.

Annealing

Annealing treatments are undertaken primarily to soften a material, to relieve internal stresses and/or to modify the grain structure. These operations are carried out by heating to the required temperature and soaking at this temperature for sufficient time to allow the material to stabilize, usually followed by a slow cooling at a predetermined rate. The choice of vacuum annealing is primarily influenced by the cleanliness and high quality of surface finish (Fig. 6) that can be obtained relatively easily compared to controlled-atmosphere heat-treatment operations.

Copper and Copper Alloys

Annealing of copper alloys is normally performed to soften the material after work (strain) hardening and to retain bright surface finishes.

Stainless Steels

Processing of stainless steel components in vacuum furnaces is often specified not only because of the cleanliness of the finished product, but also because of the fast gas-quench capability in vacuum. Stainless steel grades are usually gas quenched in nitrogen for general commercial applications. However, austenitic steel grades stabilized with titanium or columbium (niobium) require argon or helium quenching to avoid sensitization (nitrogen pickup) that degrades the corrosion resistance, particularly for nuclear, medical and aerospace applications.

A number of different annealing methods – full, isothermal, subcritical – are commonly used for stainless steel. Austenitic stainless steels cannot be hardened by heat treatment, but they do harden by cold working. Annealing not only allows recrystallization of the work-hardened grains, it also places chromium carbides – precipitated at grain boundaries – back

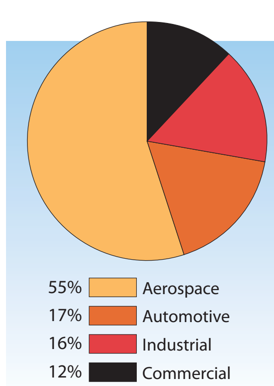

Fig. 1. Vacuum market in the Americas by industry

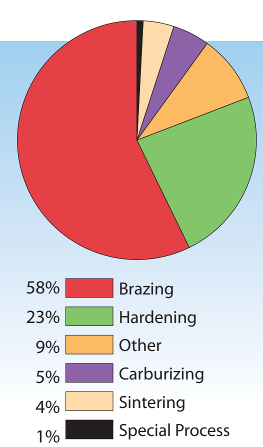

Fig. 2. Vacuum market in the Americas by process

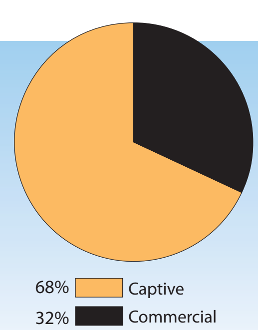

Fig. 3. Vacuum market in the Americas by segment

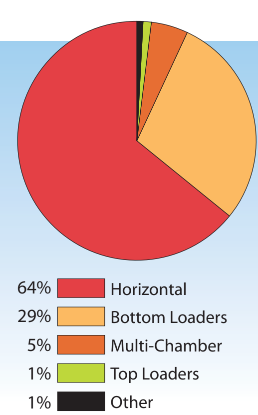

Fig. 4. Vacuum market in the Americas by equipment type

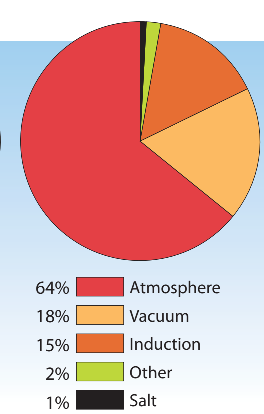

Fig. 5. Vacuum market in the Americas by technology

Fig. 6. Bright annealing of copper diodes for heat sinks

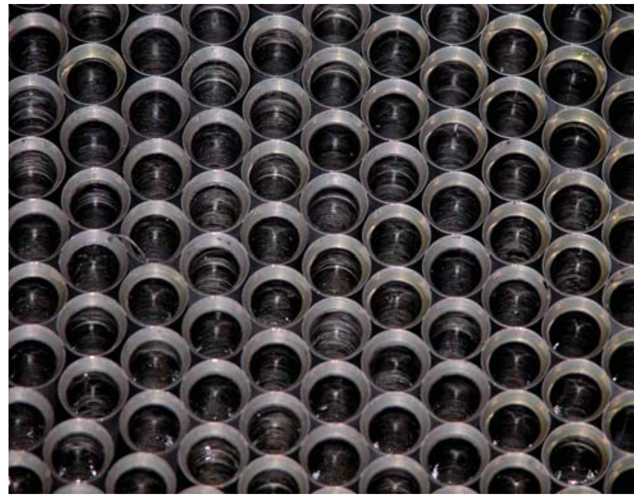

Fig. 7. Die-cutting punches after oil quenching in a horizontal batch vacuum furnace

into solution. Annealing can also be used for homogenization of castings or welds and to relieve stresses from cold working.

Time at temperature is often kept short to minimize surface oxidation and to control grain growth, which may lead to a surface phenomenon called "orange peel." Some chromium evaporation can take place during the annealing of stainless steels, but normally the amount lost is not significant because of the short time at heat and the slow diffusion rates of chromium in steel.

Annealing temperatures range from 630 – 900°C (1150 – 1650°F) for ferritic and martensitic stainless steels to above 1040°C (1900°F) for austenitic (stabilized and unstabilized) alloys. When fine grain size is desired the annealing temperature must be closely controlled.

Carbon and Low-Alloy Steels

These materials are only processed economically in applications where cleanliness of the products or the prevention of carburization or decarburization of the part surfaces is critical.

Tool Steels

Vacuum annealing is often used on tools that have been improperly hardened so that they can be reworked to meet required specifications on re-hardening. Annealing using other types of furnace equipment is impractical because all working surfaces of the tools might need to be reground due to intergranular oxidation or carburization/decarburization, thus losing the dimensional precision required.

Hardening by Oil Quenching

Oil quenching in horizontal (Fig. 7) or vertical (Fig. 8) vacuum furnaces is common using integral oil-quench designs. The design of the quench tank is similar to its atmosphere counterpart. Fixed- or variable-speed oil-circulation agitators or pumps are located on one or both sides of the tank, and internal baffles guide the respective oil flow around the load. The oil is commonly heated in the 50 – 65°C (120-150°F) range but with special oils can run at 135 – 175°C (275 – 350°F). Heaters control the temperature, and the oil is cooled via external oil cooler, usually employing air, for safety reasons.

A peculiarity of quenching in vacuum furnaces is that the low pressure above the oil causes standard quench oils to degas violently. The duration of this degassing process depends on the amount of air or nitrogen absorbed by the oil during the loading and unloading of the furnace. Vacuum oils have been created to minimize these problems. Oils that are not degassed properly have a worse quenching severity and produce discolored components.

Vacuum quench oils are distilled and fractionated to a higher purity than normal 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 540 mbar (400 torr) and 675 mbar (500 torr).

It is well known, however, that a pressure increase just before initiating the quench also changes the oil-cooling characteristics. The pressure increase shortens the vapor-blanket phase, thus increasing the quench severity at high temperatures (in the pearlite-ferrite transformation range). On the other hand, it lowers the quench rate in the convective cooling phase (in the bainite or martensite transformation range). Thus, 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 alloyed steels.

Very low pressures (<50 mbar/<35 torr) above the oil and very high quenching temperatures in the area of 1200°C (2200°F) can lead to carbon deposition and/or pick-up on the surface of parts. This is due to the thermal decomposition of the quench oil as has been experienced in hardening certain tool steels. The carbon originates from the fractionation of the oil vapor in contact with the hot surface of the load in the initial phase of the quench process. High nitrogen pressures (>150 torr/>200 mbar)

Fig. 8. Landing-gear load being assembled for hardening and oil quenching in a vertical vacuum furnace (Photograph courtesy of Vac-Aero International)

tend to reduce or eliminate this effect.

Hardening by Gas Quenching

Inert-gas pressure quenching in the range of 2–20 bar is the most popular form of quenching in vacuum furnaces. There is an interest in the use of hydrogen for cooling in the 25–40 bar range due to its extremely high heat-transfer rates. In gas quenching, part dimensional changes, although repeatable, are different than when quenching in oil. 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, variable-speed drives) has made this possible, and gas quenching is now used to produce full hardness in many traditional oil-hardening steels.

Flow rate and density of the cooling gas blown onto the surface of the load are important factors for achieving high heat transfer (high cooling) rates. In addition to high gas velocities, high gas pressures are needed to through-harden a wide variety of steel parts with appreciable dimensions. Calculations of the heat-transfer coefficient alpha (α) show that it is proportional to the product of gas velocity and gas pressure.

$$\alpha \approx (vp)^n \qquad (1)$$

where v is the gas velocity and p the pressure of the gas. The exponent n depends on the furnace design, the load and the properties of gas. It lies typically in the range of 0.6 – 0.8. The exponential behavior of the heat transfer makes it clear that the difference in the increase of heat transfer is considerable with the first few bars of pressure but decreases with increasing pressure. And the (negative) influence of equipment limitations can contribute to non-uniformity of cooling or the use of higher pressures or velocities that can contribute to increased distortion.

The critical transformation range for most steel is between 800°C (1475°F) and 500°C (930°F). Lambda (λ) values – numbers that represent the time required to pass through this temperature range divided by 100 seconds – are now available for many types of materials as a relative measure of the required cooling rate.

Cooling in argon produces the slowest heat-transfer rates, followed by nitrogen, then helium and finally hydrogen. All these gas mixtures are popular, but nitrogen is the most attractive from a purely cost standpoint. 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.

There are pressure/gas combinations that achieve heat-transfer coefficients within the range of those produced by still and mildly agitated oil quenchants. Gas quenching has certain advantages over liquid (oil or salt) quenching. The cooling rate can be easily changed by altering gas velocity or pressure, allowing not only the heat treatment of a wide variety of materials but also complex shapes and components of large or variable cross section. The effect of load weight on the resultant cooling speed during gas quenching is more pronounced than, for example, in liquid quenching. Maximum section size (ruling section) is an important consideration as well.

Fig. 9a. H-11 tool-steel die for hardening by high-pressure gas quenching (Photograph courtesy of Ipsen USA)

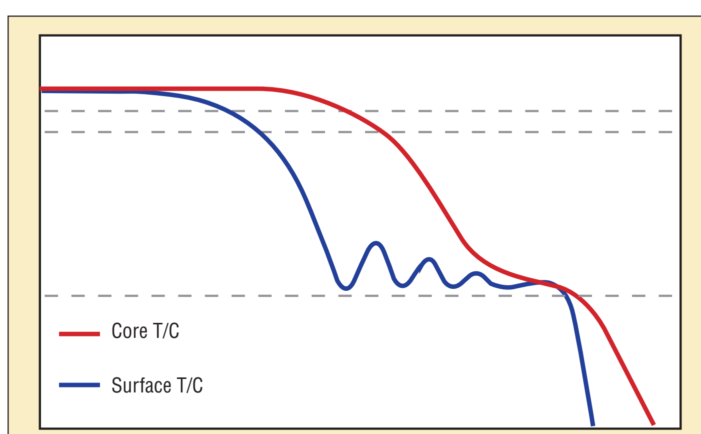

Fig. 9b. Isothermal quench cycle

Tool Steels

For most tool steels, equivalent end-product performance, surface hardness, and mechanical and microstructural properties (carbide size and distribution) can be achieved by vacuum hardening as compared to alternative technologies such as salt bath or atmosphere processing. A major advantage of using vacuum furnaces to process parts in this way is that surfaces are neither carburized nor decarburized and consequently exhibit superior performance.

In general, the air-hardening tool-steel parts are hardened in much the same way as in atmosphere furnaces. They are preheated, heated to a high austenitizing temperature and cooled at a moderate rate. The medium-alloy air-hardening steels in the A-series and the high-carbon, high-chromium steels in the D-series are regularly hardened in gas-quench furnaces using nitrogen up to 6 bar.

A rough vacuum in the range of 1.3 to 1.3 x 10^{-1} mbar (1 torr – 1 x 10^{-1} torr) is used in the heat treatment of tool steels. This level of vacuum is required mainly because of the relatively high vapor pressures of chromium, manganese and other easily vaporized elements.

Today, many grades of hot-work tool steels such as H11 (Fig. 9a) and H13 are high-pressure nitrogen-gas quenched at 10-bar pressure as opposed to oil quenched. In many cases, an isothermal hold (Fig. 9b) is introduced to minimize distortion. Gas quenching has been reported to achieve cooling rates in the 40 – 70°C/minute (100 – 160°F/minute) range, adequate for most service applications. Faster cooling rates extended life and enhanced performance. Oil quenching can achieve rates of up to 150°C/minute (300°F/minute).

Martensitic Stainless Steels

All grades of martensitic stainless steels have been processed in vacuum furnaces using the same austenitizing temperatures and considerations as those used in atmosphere furnaces. 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, processing is also done at vacuum levels ranging from 10^{-1} to 10^{-3} mbar (10^{-1} – 10^{-3} torr) with some sacrifice to 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 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.

Alloy Steels

Higher gas pressures (up to 20 bar) and the proper choice of quench gas enable some low-to-medium alloy steels and most case-hardening steels to be hardened. The automotive and aerospace industries are taking advantage of this for their heat-treatable steels that are normally oil hardened.

Sintering

Vacuum sintering, compaction (CIP, HIP) and secondary heat-treatment operations are performed on both conventional powder metal (PM) as well as particulate (CIM, PIM, MIM) materials. In an industry dominated by atmosphere processing, increased interest in controlled-atmosphere sintering arises from factors such as:

- The purity of the vacuum environment and its effect on part microstructure
- The use of sub-atmospheric (partial) pressure to improve the efficiency of the sintering reactions, especially with highly alloyed materials that require elevated sintering temperatures
- The ability of the vacuum process to reduce pore size and improve pore-size distribution
- The higher furnace-temperature capabilities that permit faster sintering reactions carried out much closer to the melting point and with alloys of higher melting point interstitial elements

The limitation on the application of sintering in vacuum furnaces is the vapor pressure of the metals being processed at the chosen sintering temperature. If the vapor pressure is comparable with the working pressure in the vacuum furnace, there will be considerable loss of metal by vaporization unless a sufficiently high partial pressure of inert gas is used. In certain instances, the partial-pressure gas can react with the surface of the part, creating a surface layer that may need to be removed.

Stainless Steel

Vacuum sintering of stainless steel powder-metal parts is a common process, employed for 300 series (e.g. 304, 316) and 400 series (e.g. 410, 420) as well as precipitation-hardening grades – 17-4PH, 17-7, 13-8Mo. These products are very often superior to those sintered in hydrogen or dissociated-ammonia atmospheres with respect to their corrosion resistance and mechanical properties.

High-Speed Steel

Powder-metallurgy manufacturing methods have been developed for producing finished and full-density cutting tools of high-speed tool steel. Applications include such items as complex geometry hobs, pipe taps and reamers. Special isostatic compacting techniques have been developed that use neither lubricants nor binders for these types of components. The pressed compacts are sintered in vacuum furnaces under precise control of heating rate, sintering time, temperature and vacuum pressure in order to eliminate porosity. The result is predictable densification of the pressed compact with final size tolerances of ±0.5 – 1.0%. Full-density, sintered high-speed steel tools have been shown to be at least equivalent to conventional wrought material in cutting performance. Grindability is dramatically improved in particular for the high-alloy grades such as M4 and T15. This is attributed to a finer and more uniform carbide distribution.

Tempering and Stress Relief

Where surface finish is critical and clean parts are desired to avoid any post heat-treat processing, many heat treaters, especially commercial shops, now employ vacuum furnaces for tempering and stress relief. These units typically operate in the temperature range of 130 – 675°C (275 – 1250°F), below which radiant energy is an efficient method for heating. As such, heating by convection is utilized. The furnace is normally evacuated to below 0.10 mbar (0.075 torr) then backfilled with an inert gas such as nitrogen, argon or even 97% nitrogen/3% hydrogen mixtures to a pressure slightly above atmospheric, typically in the range of 0.5 – 2 bar. A fan in the furnace recirculates this atmosphere, and parts are heated by both convection and conduction. Temperature uniformity in the range of ±5°C (±10°F) is common with tighter uniformities possible.

Certain horizontal single- and multiple-chamber furnaces have been designed to perform single or multiple tempering treatments after hardening or case hardening without having to remove the workload from the equipment. This process is in common use for tempering high-speed-steel components and a variety of other materials.

The series continues discussing aerospace and other applications in Part Two. **IH**

References

Herring D. H., *A Perspective from the Americas: Advanced Technology Trends in Vacuum*, White Paper, May 2007.

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