The use of doped and alloyed molybdenum in vacuum furnace hot zone design and construction improves furnace performance and efficiency. Lower mass in the hot zone equates to less material to heat and cool, and high performance materials make hot zones last longer because of their enhanced metallurgical properties. Hearth components remain straight and true, heating elements resist sagging and distortion, radiation shielding is less likely to break and gas-cooling nozzles maintain their shape.
Historically, furnace manufacturers improved the quench properties of their vacuum furnace products by increasing the horsepower on the fan/blower assembly (to achieve faster gas velocities), increasing quench gas pressure (to improve heat transfer), and focusing on the process gas and/or gas blend (for improved velocity and/or heat transferability). Although there is little doubt the aforementioned parameters are paramount in the quenching process, the mass of the hot zone is the one element within the overall furnace design that is frequently overlooked. Elegance in hot zone design is not achieved by how much material is incorporated into the hot zone, but rather by how much material is removed. The hot zone itself is a process variable in the quench equation. Lower mass in the hot zone equates to less material to heat and cool and therefore begets improved efficiency and performance.

New high performance, low-mass, all-metal vacuum hot zones designed specifically for rapid gas quench furnaces leverage the superior strength and performance properties of advanced molybdenum alloys. Low-mass designs require less mass of high performance materials, relative to conventional construction materials. These designs maintain the strength and integrity of the hot zone, yet improve performance. Because of their lower mass, new hot zone designs quench faster than standard hot zones (all else being equal). A side benefit is the decrease in cost because materials utilized in hot zone construction are sold by weight (and there are less of them in a low-mass design) so the initial capital outlays are also less.

Additionally, to all-metal, low mass designs, high performance alloyed and doped molybdenum can be successfully incorporated into insulated hot zone designs (i.e., graphite board, graphite foil-board, graphite felt, graphite foil and ceramic fibre types). This allows the creation of a new, advanced, energy efficient hybrid that combines insulated materials with metal to further improve the quench rates (through better heat transfer) and increased durability (elimination of hot-gas erosion) of these type hot zones.

**Background**

For more than two decades, vacuum furnace manufacturers have responded to the market demands of heat treaters to rid themselves of the hazardous heat treating processes of the past. This was done primarily in response to local and national legislation that insisted on environmentally friendly process alternatives. The equipment suppliers’ solution was to develop rapid gas quench vacuum furnaces utilizing a variety of environmentally harmless process gases in place of the environmentally hostile quench oils, salt baths (barium-chloride and cyanide salts), and other hazardous liquids. Rapid gas quenching in a vacuum furnace is really the process of forced convection cooling accomplished by recirculating the quench gas through a high efficiency gas-water heat exchanger and transferring the heat from the workload (and hot zone) to an external cooling water system.

Advances in vacuum furnace quench technology were necessary to heat treat the oil hardened tool steels, high speed tool steels, hardenable stainless steels, and assorted super alloys that were processed by hazardous means in the past. These materials are utilized in diverse applications including aerospace, defense, gas turbine, tool and die, and automotive markets.

**Heat Transfer and Gas Quenching**

Three major factors contribute to heat transfer when rapid gas quenching in a vacuum furnace: temperature difference between the part and the re-circulated quench gas; surface area of the load exposed to the quench gas; and the heat transfer coefficient. The heat transfer coefficient of the work load is a function of the quenching gas employed, the thermal conductivity of the workload material, and the gas temperature and pressure of the furnace. Since both the surface area of the load that is exposed to the quench gas and the thermal conductivity are essentially constant for a given application, the industry focus has been on improving the heat transfer coefficient by choosing an optimal quench gas and/or blend and increasing its pressure and velocity across the workload.

The first generation of rapid gas quench furnaces was introduced about 20 years ago with pressures 0.8 to 1.5 Bar (80 to 1.5 kPa to 20 psi) and relatively low gas velocities. The next generation (15 - 20 years ago) typically cooled at a maximum of 2 Bar (200 kPa to 30 psi), and used gas velocities about twice as fast as earlier units. About 10 years ago, the first commercial 10 Bar (1000 kPa to 150 psi) furnaces were introduced with gas velocities seven to ten times the second generation systems. Some of these units were coupled with the first convection assisted heating options – and the race was on!

Today, quench pressures up to 20 bar or more, with thermocoupled loads for computer controlled directional cooling (to reduce distortion) combined with convection assisted heating (for faster heating rates), are offered by most leading vacuum furnace manufacturers.

Theoretically, there is no limit to cooling rate improvements that can be obtained by increasing gas pressure and velocity. Practically, however, with very high gas pressures (>20 Bar or more) and extreme gas velocities (made possible with 400HP+ fan motors), these furnaces become costly to procure and to operate. Or, put another way, the costs quickly accrue at a faster rate than the benefits.

Typically, the mass of the hot zone is not factored into the gas-quench equation as a target for improvement. Yet, as an example, in a typical standard size single chamber, batch-type vacuum furnace hot zone with an operational temperature of 2,400°F (1,315°C) and a workload size of 36 in. wide x 36 in. high x 48 in. deep (914mm x 914mm x 1219mm) with a maximum load capacity of 3,000 lbs. (1,361 kg.) can weigh 1,600 to 1,750 lbs. (680-793 kg.). The hot zone, along with the work load, must heat and cool to complete a heat treat process cycle. Ideally, the function of the hot
Three major factors contribute to heat transfer when rapid gas quenching in a vacuum furnace: temperature difference between the part and the re-circulated quench gas; surface area of the load exposed to the quench gas; and the heat transfer coefficient.

Standard Types of Rapid Gas Quenching Hot Zones

There are two broad categories of vacuum furnace hot zones based on their materials of construction: radiation designs and insulated types. Both styles strive to minimize heat losses to the cold wall, but they do so by different thermodynamic principals.

The radiation design, or “all-metal,” type works by providing a vacuum between several layers of metal shielding to reduce heat losses to the cold wall. The vacuum itself is a good insulator and the heat transfer from shield to shield only occurs through radiation losses. Heat loss, in turn, with these types of hot zones, is not dependent on relative width of the shield spacing but rather on the type of material and the surface quality of the radiation shielding employed – this is referred to as emissivity. In general, highly polished (reflective) metals have low emissivities. The opposite condition is a “blackbody,” which has high emissivity (or is an absorber of energy).

The insulated design is available in many different configurations and combinations. The usual materials of construction include: rigid graphite board (coated, uncoated, and foil-bonded), non-fibrous board (foil board), graphite felt, graphite foil, carbon fiber composite (CFC), and high purity alumina fiber. These designs attempt isolate the thermal radiation emitted from the innermost part of the hot zone from the cold wall by blocking the heat loss through their insulating properties. These hot zones are more energy efficient than conventional all-metal designs. The trade-off, unfortunately, is that insulated designs have reduced quenching capability because the same properties that allow for their higher energy efficiencies. The heat is simply trapped inside a well-insulated zone and does not readily escape, as it would in an all metal design (this is the heat-transfer coefficient).

In many thermal processes, particularly rapid gas quenching, all-metal vacuum furnaces offer a variety of benefits compared to graphite insulated types. All-metal hot zones enable higher vacuum levels, quicker pump-down, cleaner atmospheres, no carbon contamination, better temperature uniformity, and higher quenching rates.

All Metal Hot Zone Materials of Construction

The refractory metal molybdenum has been used in high temperature furnace construction for many years. Its metallic properties, in combination with a high melting point and good creep resistance, make it an ideal material for high temperature vacuum furnace components like heating elements, radiation shielding, hearth assemblies, and other components.

One of the major problems in incorporating pure molybdenum in furnace construction is its embrittlement (re-crystallization) after exposure to high temperatures. Re-crystallized pure molybdenum cracks easily along its grain boundaries at ambient temperature when impact loaded. This can lead to premature damage (sometimes accidental) or failure of the hot zone. As a side note, re-crystallization has inhibited pure molybdenum’s wider use with furnace racks and high temperature brazing fixtures.
To meet the increasing demands of the thermal processing industry (such as lower operational costs through extended service life and reduced maintenance), it became necessary to develop advanced molybdenum-based alloys with specific, enhanced, high temperature properties. Doped/alloyed molybdenum materials have been available since the 1970s, but Schwarzkopf’s Plansee MLR is the first doped molybdenum sheet material that comes in a re-crystallized condition – by design. Finely dispersed lanthanum oxide particles doped into the material improve its creep resistance and stabilize the microstructure for improved ductility even after re-crystallization. This is because the lanthanum oxide limits/inhibits the grain growth of the molybdenum grain boundaries to an acceptable level, at which ductility and strength are maintained. In contrast, pure molybdenum’s grain boundaries continue to grow and expand to the point of embrittlement and strength is lost compared to the un-crystallized material. The advantages of the material’s special composition and production process are: the material does not become brittle in operation because of its stable microstructure; and it has excellent creep properties up to 3,362°F (1,850°C).

The material is used for service temperatures above 2,372°F (1,200°C) and up to 2,722°F (1,800°C). MLR (molybdenum – lanthanum oxide – re-crystallized) in sheet form is available in .040 in. (1mm) thickness and greater. Another Plansee material, MLS (molybdenum – lanthanum oxide – stress relieved), is used for sheet thickness from .010 in. (.254mm) up to .040 in. (1mm). MLS differs from MLR in that it is processed with a special stress relieving heat treatment to optimize the room temperature ductility and additional properties required for enhancing radiation shielding.

Plansee TZM is a molybdenum-based alloy containing small additions of finely dispersed particles (carbides and oxides from titanium and zirconium). These particles stabilize the microstructure, shift the recrystallization temperature beyond 2,192°F (1,200°C) and inhibit grain growth at high temperatures. Thus, its ductility and high temperature strength are substantially increased in comparison to pure molybdenum. TZM is employed in high loaded/stressed components such as vacuum furnace hearth assemblies preferably in the temperature range between 1,832°F (1,000°C) and 2,372°F (1,300°C).

Field Experience
Pat Wall, president of Stack Metallurgical Services Inc., Portland, OR, was an early adopter of high pressure rapid gas quenching and has operated an all-metal, 10-bar, 400 HP rapid gas quench (48 in. wide x 48 in. high x 48 in. long) batch vacuum furnace for the last 10 years. Regularly processing at high pressures and at peak horsepower, the quench gas moving through Stack’s hot zone can best be described as torrentially brutal. For the last 5 years the hot zone in this furnace has required virtually no replacement spare parts. The heating elements, element supports, hearth assembly and radiation shielding all remain sag and distortion free due to the incorporation of the high performance Plansee ML and TZM materials.

Insulated Hot Zone Materials of Construction
Insulated hot zones, as stated earlier, are available in a variety of materials and combinations each with its own set of advantages and disadvantages. One popular material is a rigid carbon fiber insulation often referred to as “graphite board.” Typically, boards are combined to form a group of interlocking panels for radiation shielding. The material is available uncoated, or it can be coated with a graphite reflective paint, occasionally a CVD (chemical vapor deposition) coating, or with a foil bonded to the hot face (more common). This material is relatively easy to work, install, maintain, and repair.

The weakness of this material is gas erosion along the exposed seams and penetrations of the individual boards when rapid gas quenching. This is particularly a problem in 6-20 bar furnace applications. Erosion is often the reason for the premature demise of a graphite board hot zone. In addition, the gas erosion of the board, in part, explains some of the free carbon in the furnace. The other contributor of free carbon is the binder vaporizing out of the board, leaving only the fine graphite powder from which the board is made. All this “free graphite powder” makes for an unclean working environment that can contaminate the work load (particularly in graphite sensitive processes). Even if workload contamination is not an issue, there is absolute certainty that the free graphite will be carried through the re-circulation system onto the heat exchanger and blower of the gas quench system. There, it deposits on the cooling fins of the plate and fin tube heat exchanger, decreasing efficiency through the...
loss of its ability to transfer heat.

There are, however, design improvements that can extend the life of graphite board insulated zones and prevent the effects of “free graphite.” One way to decrease or eliminate the de-binding problem with conventional graphite board is to switch to a new, non-fibrous foam board composed of the material used in the construction of a typical foil. This material is graphite-free, has low thermal mass, and high resistance to gas erosion. Non-fibrous foam board, however, has its limitations. It has two disadvantages to conventional graphite board: it has very little mechanical strength (extremely fragile to handle and work with) and breaks very easily; and it has poor insulating capabilities relative to regular density graphite board or common graphite felt. In fact, non-fibrous board has only 25 percent the insulating properties of graphite felt, meaning four times the material thickness must be used for equivalent insulation.

An alternative is a lanthanum oxide-doped molybdenum hot face backed by graphite felt insulation. This is a modern version of an old design, which utilized a pure molybdenum hot face backed by high purity alumina fiber. In each case, these combination, or “hybrid,” hot zones provide high emissivity because of the metallic hot face, and good insulation properties for energy efficiency. They don’t quench as well as an all-metal hot zone, but they have superior quench properties than an all-composite hot zone because the molybdenum acts as a “heat exchanger” for the removal of the heat energy stored or contained in the graphite felt insulation.

Low-Mass Vacuum Furnace Hot Zones for Rapid Gas Quenching

The emissivity of its radiation shield package has an enormous effect on the heat losses of a vacuum furnace. Perfect reflectivity would be an emissivity value of “0” (no loss of reflective energy) and an emissivity of “1” would be a complete absorber of energy (a true blackbody). Typical values for the emissivity of molybdenum are 0.10 (20°C/68°F) to 0.22 (1,000°C/1,832°F) and for stainless steel 0.30 (20°C/68°F) to 0.70 (1,000°C/1,832°F).

Traditionally, all metal hot zones are constructed with three layers of molybdenum (.015 in. thick hot face, with two .010 in. thick sub shields) and two layers of stainless steel (typically .010 in. - .018 in. thick each) for a standard radiation shield pack with spacers between each layer. Based on actual test data and theoretical calculations, stainless steel radiation shields at normal vacuum furnace operational temperatures have such high emissivity values that they perform more like a blackbody than a reflective radiation shield! Therefore, stainless radiation shields really do not contribute significantly to the prevention of heat losses to the cold wall. Therefore, the removal of both stain-
less shields and subsequent replacement with a single .005” molybdenum shield dramatically decreases both the mass and emissivity of the hot zone. The simple “two for one” substitution provides for faster quenching (less mass) and better energy efficiencies (less heat loss) – without significantly adding to the costs.

Low Mass Hearth Assemblies
The hearth assembly is another area in which TZM molybdenum alloy can be used in place of pure molybdenum. At typical furnace temperatures, TZM is 3-5 times stronger than pure molybdenum, so a 50 percent reduction in the size of these components is possible. The benefit is that even with their significantly smaller mass, the TZM components are stronger and more resistant to brittle fracture (after recrystallization) than their pure molybdenum counterparts.

Low Mass Plenums/Retorts
The final performance improvement (and subsequent mass reduction) occurs by substituting 304 (or 316) stainless steel for carbon steel in the plenum/retort portion of the hot zone. The main purpose of the plenum/retort is as a support structure to hold the radiation shield packs, heating elements, and element supports. In addition, it helps uniformly distribute the quench gas throughout the hot zone by incorporating strategically located gas nozzles. The plenum/retort also defaults as a radiation-shielding barrier.

Traditionally, MIG welded carbon steel is chosen for these fabrications, but TIG (tungsten inert gas) welding is required for stronger, more heat-resistant materials such as stainless steel. Although stainless has a higher specific heat ratio than carbon steel, it is significantly stronger at elevated temperatures and less of it is required for a given application. Again, the theme is ‘less material equates to lower mass and faster heat-up and cooling rates.’ An added benefit of stainless steel is its greater emissivity than carbon steel, so it can radiate more energy to the center of the hot zone, where it is needed.

Conclusion
In a hot zone of standard size, a 29 percent reduction in mass is possible by using high performance doped and alloyed molybdenum. Actual quench performance is improved by a minimum of 15 percent. Improved performance stems from sound mechanical design and improvements in the properties of the materials of construction. Lower mass in the hot zone equates to less material to heat and cool, for improved thermal efficiency and performance. In addition, high performance materials make hot zones last longer because of their enhanced metallurgical properties. Hearth components tend to remain straight and true, heating elements resist sagging and distortion, radiation shielding is less likely to break and gas-cooling nozzles maintain their shape. Most of these components typically last the life of the hot zone – with little or no need for spare parts.

Thomas P. Farrell, Jr. is vice president of sales and marketing for Schwarzkopf Technologies LLC, Franklin, MA. He can be reached at (508) 553-3800; by e-mail at stc.sales@stc-ma.com. For additional information visit www.stcmetals.com or www.plansee.com