

Case study



Figure 1. Rotary furnace system.

Credit: Harper International

Combining expertise and innovation to design advanced combination furnace systems

By Colleen Marren

Customized engineering and unique dual-function rotary furnaces solve thermal processing challenges.

Thermal processing of advanced and high-purity materials requires equipment that can reliably balance high-quality product output, safety, and economic production. In today's market, companies must balance a variety of product lines, each requiring the same standards. Harper International understands that each product requires its own customized processing solution. Advancements in Harper's rotary furnaces allow for the interchange of process tube materials or shapes to utilize one thermal system for a variety of advanced material product lines and material developments (Figure 1).

An indirectly fired rotary furnace is a continuous thermal system that applies heat to the incoming process material over multiple thermal control zones. The complete system includes feeding, heating, cooling, and gas handling and provides a very efficient method for processing powders. Rotary tube furnaces primarily are used because of their efficiency in heat transfer and mass transfer for powders as well as minimizing material-handling requirements.

A rotary tube furnace transfers heat from the heat chamber to a rotating tube, which contains the process materials and the required special atmosphere. Specifically, the furnace transfers heat from the tube wall to the bed of material being processed. Maintaining the process tube at a small angle and rotating it

causes the material to continuously convey through heating and cooling sections. Rotary furnaces have one of the highest thermal efficiencies and lowest operating costs compared with other types of equipment.

The following are two cases in which Harper designed unique combination furnaces to fit specialized needs for advanced material development.

Case study 1: Alloy/ceramic tube rotary furnace combination system

ECRIM, a leading ceramic micro-electronic materials company, develops advanced ceramic materials for applications requiring high thermal conductivity, such as high-power LED lighting products and other heat-sink substrates.

Beginning production before proper research and development can waste countless hours and raw materials, which often are sparse in any new project, and result in inconsistent and low-quality products. Therefore, ECRIM first develops the necessary processing for its new materials in a process development mode. After optimizing various parameters—including residence time, tube rotation rate, tube inclination angle, temperature profile, atmosphere flow-rate, powder bed depth, and feed rate—the company then uses those parameters to demonstrate its ability to scale up production while maintaining precise product requirements.

ECRIM's development of new processes and products necessitates that its equipment can easily vary processing parameters. The company needed a rotary furnace to obtain excellent gas-solid contact, controlled atmosphere with gas-tight rotary seals, material mixing, and continuous production of its products.

Tube material selection

Rotary furnaces use a variety of tube materials, including alloys, graphite, silicon carbide, and aluminum oxide. Harper carefully selects the tube material in a rotary furnace to ensure that it can withstand the process atmosphere and temperature. Selecting the improper tube material can have many unwanted

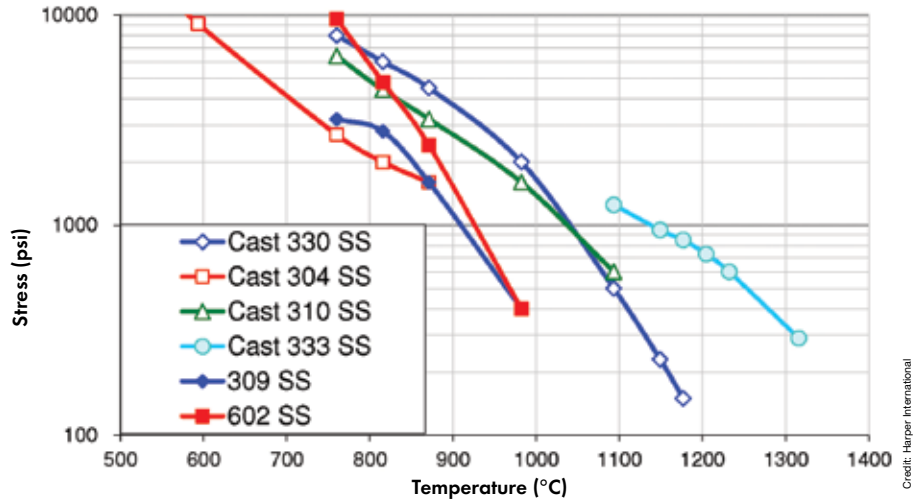


Figure 2. Temperature versus allowable creep stress of stainless steel alloy process tubes.

consequences, the most obvious of which is product contamination. Serious consequences also can occur if hazardous materials and/or hazardous gases are processed inside the tube, because it is the only object separating the process environment and ambient atmosphere.

Ceramic tubes can shatter if they experience too much stress under the improper conditions, which can cause substantial damage inside the furnace. Alloy tubes can crack if they experience high stress or react with the process environment. Cost, thermal shock, and creep stress also are important in tube selection. Figure 2 shows the correlation between process tube material type, temperature, and maximum allowed stress for several stainless steel alloys.

Maximum allowed stress is based on the amount of creep (deformation) a tube can withstand before rupture. Typically, manufacturers design furnace tubes so that after 10,000 hours at maximum process temperature, the tube deforms less than 1% because of creep stress. Stress on the tube varies based on rotational speed and amount of material inside the tube, diameter of the tube, suspended length of the tube, and thickness of the tube.

Manufacturers consider cast and wrought alloys for high-temperature applications. Figure 2 shows that cast alloys often have higher allowable creep stress. Also, some materials are available only in cast form because they lack sufficient ductility to be worked into a wrought tube, such as high chromium content alloys.

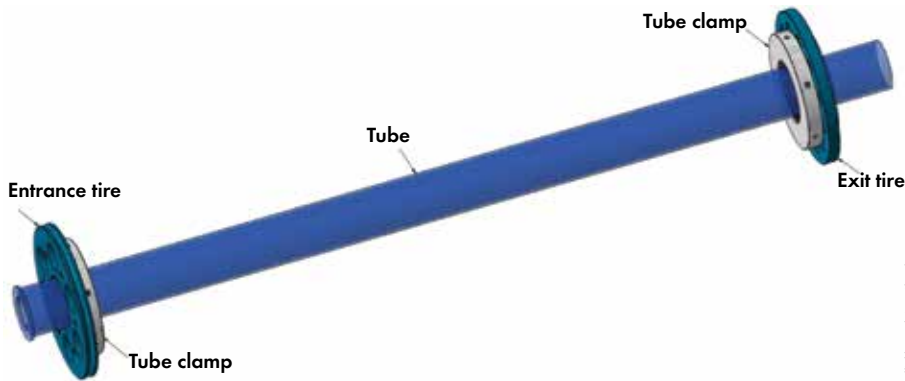
Users strongly prefer alloy tubes in rotary thermal systems requiring diameters greater than ~9–12 in., because they are significantly less expensive, have shorter lead times, and do not suffer catastrophic brittle failure. Thermal stress increases with tube diameter, causing an increased probability of tube failure for brittle ceramics. Large-diameter tubes with high costs and long lead times often are not practical or economical, because ceramic tubes can fail at any time from random defects.

Users should consider ceramic tubes when a product that requires high purity is being treated or the metals in the alloy tube have potential to react with the product or off-gases created during production. Ceramic tubes resist corrosion and are useful in high-temperature applications. Commonly used ceramic tubes include quartz, silicon carbide, aluminum oxide, and, in some cases, graphite.

Manufacturers use graphite tubes and muffles in furnaces requiring high temperatures or high purity. They are best suited for environments between 1,000°C and 3,000°C and under inert atmospheres, such as nitrogen or argon. Graphite tubes are not suitable for oxidizing environments, because carbon will oxidize at these temperatures.

Silicon carbide tubes come in many forms, including nitride-bonded silicon carbide, reaction-bonded silicon carbide, recrystallized silicon carbide, solid-state-sintered silicon carbide, and liquid-phase-sintered silicon carbide. Silicon carbide tubes are best used for processes requiring

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Figure 3. Harper dual-material tube assembly uses tube clamps instead of welded-on flanges.

an air atmosphere at temperatures below 1,400°C, depending on the grade of silicon carbide. Many economical grades of silicon carbide are porous, making a controlled atmosphere difficult.

Aluminum oxide tubes, similar to silicon carbide tubes, come in many grades. Aluminum oxide tubes that can tolerate some thermal stresses are highly porous and, thus, are best for processes that do not require a controlled atmosphere environment. Aluminum oxide tubes are preferred for processes requiring temperatures $\leq 1,600^{\circ}\text{C}$, depending on tube size and mass rate of the process material. However, aluminum oxide tubes cannot easily tolerate thermal shock and must be heated and cooled very slowly.

Quartz tubes are suitable for processing at temperatures $< 1,300^{\circ}\text{C}$ and are suitable for oxidizing, reducing, or inert atmospheres as well as many high-purity applications. These tubes are not permeable and have a very low coefficient of thermal expansion compared with most ceramic tubes. However, quartz tubes are brittle and can break because of random defects.

ECRIM's production demanded a quartz process tube based on strict product purity, temperature requirements that reached 800°C, and need for an atmosphere containing a precise composition of nitrogen and air for a tightly controlled oxidation reaction. With the required tube diameter, a quartz tube is significantly more expensive than an alloy tube and could break during ECRIM's research and development period. However, an alloy tube, even though it can withstand the required temperatures, introduces contamination risks.

Therefore, ECRIM worked with Harper to design a single, continuous thermal rotary processing system that could easily interchange between an alloy and a quartz tube.

System design

Harper's equipment design solution uses an alloy tube for process development, followed by a quartz tube for significant quantity production of high-purity material. In addition to being multifunctional, this strategy eliminates contamination between research and development material and production material.

The single thermal processing system features three individually controlled temperature zones. Users can easily optimize the temperature profile during process development. The furnace also features a clamshell design that allows the top half of the furnace to open, exposing the furnace's internal section. The top half of the clamshell is hinged at one end and opens via pneumatic actuation, making it easy to access the furnace tube, insulation, and heating elements without a crane.

The rotary tube furnace features a completely sealed rotary tube system and enclosed discharge collection to maintain atmosphere integrity. The exit hood, entrance hood, and rotary seals are on rails to allow the seal to move with the expanding tube. This is especially important when using the alloy tube, which has a thermal expansion of $\sim 1.35\%$ —much larger than the quartz tube.

Tube rotation and tube inclination directly affect residence time. The user controls tube rotation by a programmable

logic controller system via a variable frequency drive. Thus, ECRIM can easily change the rate of tube rotation and resulting material residence time during process development. A pneumatic adjustable jack controls tube inclination to easily adjust the system without a crane.

This furnace is unique because of its ability to use a quartz or alloy tube. A Harper rotary tube on this scale often is supported via flanges welded to the exterior of the tube. These flanges are then bolted to tire assemblies, which are set upon casters. The casters are designed to be gentle on the tube and constructed to resist heat. This dual material rotary furnace system uses tube clamps instead of flanges welded to the exterior of the tube (Figure 3). Features designed on the exterior circumference of the tube secure these tube clamps. ECRIM can interchange the same tire easily between quartz and alloy tube assemblies. A variable speed drive connected to the tire through a V-belt to the reactor tube, mounted at the end fixed from expansion, rotates the tube.

ECRIM removes the tube assembly by unlocking and sliding away the feeder, on rails, from the entrance seal assembly. The entrance seal assembly and exit hood assembly also are on rails, allowing them to be unlocked and slid out of the way for tube removal. Users can easily remove and replace the tube after pneumatically opening the furnace clamshell. ECRIM now has flexibility to test and develop new materials utilizing alloy or ceramic tubes for its processes. This furnace will support the company's mission to produce advanced ceramic materials for applications requiring high thermal conductivities. Harper's design of this dual functionality furnace supports requirements to work with multiple types of tubes, saving money, time, and facility space.

Case study 2: Rotary/mesh belt rotary furnace combination system

A national laboratory, which had limited available floor space, contacted Harper to assist design of a single, continuous thermal processing system. To

enable process development for commercial production of carbon materials, the lab needed a continuous thermal processing system to operate at temperatures up to 1,050°C and in a variety of atmospheres. The lab wanted to vary carbon precursor materials in composition, particle size, moisture content, density, and shape. These variations in precursor materials required two distinctive types of furnace systems: a rotary furnace and a mesh belt furnace.

Precursors with good flow properties that require shorter residence time and better gas-solid contact are ideal for rotary systems. However, for fragile precursors, a rotary furnace is not an option because mixing and tumbling throughout the tube would be detrimental to the product. Tube rotation rate and inclination control residence time in a rotary furnace. Materials requiring longer and more controlled residence time also are unsuitable for a rotary furnace, because long residence times require a very small angle of inclination and a slow tube rotation rate, making it extremely difficult to control residence times more than two hours.

A mesh belt furnace is a continuous thermal system that applies heat to the incoming process material via multiple thermal control zones. Either gas or electric heating powers the system. A muffle can control atmospheres in the reaction chamber. Further, if a customer requires an inert atmosphere, the product conveyed on the belt can enter a purge chamber before entering the heated zone of the furnace. Process materials are continuously fed directly onto the belt or placed in crucibles for thermal processing. Because the belt moves through the heated section supported by the alloy muffle, the process material continues to progress through the length of the furnace supported on the belt. The belt conveys the material through each temperature control zone, and belt speed controls the soak time in each zone. Once thermally processed, material travels through a cooling section and then an exit purge chamber before unloading.

To meet all required design aspects, initially it seemed that the laboratory required two separate furnaces. Harper first considered a traditional rotary furnace and a traditional mesh belt



Figure 4. Mesh belt/rotary thermal combination system designed for a national laboratory with space constraints.

furnace. However, this client had very limited space and limited resources, so two stand-alone furnaces were not optimal. The Harper team instead designed a multifunctional thermal processing system that can be transformed from a rotary tube furnace to a mesh belt furnace using a single thermal platform (Figure 4).

System design

The single thermal processing system featured a clamshell design that allows the top half of the furnace to open, exposing the internal section. The rotary tube furnace features a completely sealed rotary tube system and enclosed discharge collection to maintain atmospheric integrity. Four zones of control allow adjustments to heat-up rate, soak period, and cooling rate. The user controls process capacity by adjusting the angle of the system's inclination, along with rotational speed of the tube and feed rate from the screw feeder system.

Akin to autobots in the movie *Transformers*, the user reconfigures the main parts of the system in quick succession to transform the system between mesh belt and rotary tube systems. If starting with the rotary tube system, users remove the screw feeder, rotary tube (via clamshell access), and entrance and exit hoods. Next, users install the alloy muffle, cooling section, purge chambers, and mesh belt. The mesh belt drive stands and the heating chamber remain in place, so minimum number of subassemblies are reconfigured. The mesh belt thermal system also is atmospherically controlled, with purge chambers located at entrance and exit ends of

the furnace system. Four zones of control allow for adjustments to the thermal profile. Harper also designed the internal alloy muffle with several exhaust ports at key locations for removal of corrosive off-gases.

Both furnace systems are gas-tight and operate under a variety of atmospheres, including reactive and corrosive gases. The systems can operate in the 1,000°C range, with thermal processing cycle variations from 30 minutes to 10 hours.

This leading national laboratory now has the flexibility to test and develop new carbon materials from a variety of sources, including renewable materials, to support its mission to advance the future of materials research. Harper's design of this dual functionality furnace supports requirements to work within limited laboratory space and the need for flexibility in processing a wide variety of materials in rotary and mesh belt furnace systems.

About Harper International

Harper International (Buffalo, N.Y.) develops complete thermal processing solutions and technical services for the production of advanced materials. The world of advanced ceramic materials continues to change at an accelerated pace. Design of the above custom furnace systems demonstrates Harper's commitment to provide solutions, investments, and new concepts to achieve the needs of its valued customers.

About the author

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