

The development of an innovative continuous belt furnace for the high temperature sintering of MIM and CIM products

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Centorr Vacuum Industries, based in Nashua, New Hampshire, USA, has specialised in the development, engineering, design and manufacture of vacuum and controlled atmosphere furnaces since 1954. In the following paper the company introduces a unique low mass, fast throughput belt furnace design that operates in ultra clean environments of inert or process hydrogen gas that can be used over a wide temperature range from 1000°C up to 2800°C. While this continuous furnace design is not suitable for all applications, if considered for the right process, it can provide the combined benefits of fast cycle times in an oxygen free atmosphere.

Processing metals and ceramics can require very special sintering conditions and operating environments in order to achieve optimal physical properties. While conventional vacuum, atmosphere batch, and continuous belt or pusher furnace designs work well for a majority of these applications, there are some unique materials (or processes) that could benefit from an ultra clean, oxygen free environment and a fast ramping speed with minimal soaking times.

Although vacuum and atmosphere batch furnaces are commonly used in industry, cycle times can be long and there can be variances present throughout the load, especially when processing a large quantity of small parts in a large furnace chamber. This is especially the case where fixturing and setter furniture like hearth plates can cause thermal scattering of the heat or act as a heat sink for the small components.

Conventional belt furnaces utilise Inconel mesh belts, have refractory ceramic (brick or fibre) insulation, and can process loads of many kilogrammes per m² of belt area. They are typically rated for maximum continuous use at temperatures to 1150°C or up to 1288°C for units with ceramic belts. While large belt and pusher furnaces are ideally suited for high throughput applications such as those found in the Powder Metallurgy industry, they are not as economical for lower volume, shorter furnace runs of small parts under 6-12 mm in size that need to be run in ultra clean environments. These conventional belt furnaces require expensive nickel alloy or ceramic muffles inside the hot zone to help control, protect and direct the reducing gas away from the insulation and heating elements, and maintain the desired furnace atmosphere. These muffles can have a short lifespan if cycled frequently and for this reason, once the units are heated up, they are seldom turned off and instead idled over weekends or periods of low production rather than turned off completely. Large belt furnaces

are typically shut down only one to two times over several years of use, and not every application can justify these production capacity requirements. They must also be cycled with dummy loads during periods of non use to prevent overheating of the main chamber. For improved cleanliness, some designs also include a “humpback” feature which lifts the belt and load into a higher area of the hot zone where cleaner, lower dewpoint hydrogen gas is present.

To address the above concerns and accommodate these niche small volume and fast throughput applications a new style of furnace design was conceived which was born of the benefits of vacuum batch furnaces, with the processing speed and process uniformity found in continuous designs. This novel high



Fig. 1 Conventional vacuum furnace hot zone



Fig. 2 2000°C rated belt furnace for sintering refractory metals in hydrogen gas



Fig. 3 Refractory metal belt furnace hot zones



Fig. 4 Refractory metal belt furnace hot zones

temperature continuous belt furnace is shown in Fig. 2.

While this unique belt furnace design was originally introduced in the early 1990s its popularity has recently begun to rise in the past five years as a number of new applications have opened up for its use. The furnace is built similar to vacuum furnace construction with a water cooled stainless steel chamber and an interior hot zone designed using refractory metals for the heating elements and shielding (such as molybdenum or tungsten), or rigid graphite insulation and graphite elements for ceramics applications. These unique continuous furnaces are rated for maximum temperatures of 2000°C in either inert or process hydrogen gas with a refractory metal hot zone, or up to 2800°C in inert gas when using graphite hot zones. To provide fast heat up and cool downs, double wall water jacketed entrance and cooling tunnels are located on the ends of the chamber. For material transport, the furnace is fitted with a range of belt materials depending on use temperature and load weights. This includes a proprietary patented molybdenum or tungsten mesh belt design rated to 1550°C or 2000°C. For higher load applications, silicon carbide link belts are used to 1800°C, and for temperatures over 2200°C flexible graphite cloth is the belt material of choice. The furnaces are available in a range of sizes and throughputs, with a nominal belt width of between 50 - 200 mm, a height of 25 - 100 mm, and a length dependent on the desired throughput, but typically 200 - 1525 mm. They are configured in either one, two, three, four or five zones of control and achieve excellent temperature uniformities of +/- 3 to 5°C across the belt in inert or hydrogen process gas.

The throughput and process repeatability have made this furnace a successful tool for dependable production in applications such as Metal Injection Moulding, the high temperatures sintering of refractory metals and ceramics, precision brazing, metallization and the ceramic to metal joining of components. Recent installations to date have centred around applications in Micro-MIM parts, silicon nitride components, high temperature sintering and hydrogen brazing. Four application case histories are presented below.

Metal Injection Moulding

A MIM parts producer was looking to manufacture large quantities of a relatively small part with the flexibility to start and stop runs several times per week. These parts were made of stainless grades including 17-4PH or 316L and applications included orthodontic braces and brackets, cell phone cam hinge pieces and miscellaneous small parts weighing under 5 g each.

Unlike the previous applications, this one added the complexity of removing up to 1-3 wt% of a difficult polymer based binder system commonly used in Metal Injection Moulded parts during a low temperature binder burnout step. In this case the belt furnace's hydrogen flowthrough gas provided an excellent vehicle to breakdown the binder's hydrocarbon chains so that offgassing could be safely removed from the parts, and reduced to gaseous form with little solid binder residue left in the entrance tunnel. In certain applications a gas moisturising system can be added for processes requiring higher hydrogen gas dewpoints (typically from -20°C to over + 20°C), such as used in metallising applications, or instances where moisture assists in the binder removal phase.

The small cross section and fast throughput of the belt furnace was advantageous in providing a thorough and uniform debinding environment which is considered a bottleneck in batch sintering applications where larger loads must be debound slowly with several holds at intermittent temperatures. By keeping the load weights light and the furnace cross section small the parts are intimately and quickly debound before entering the sintering

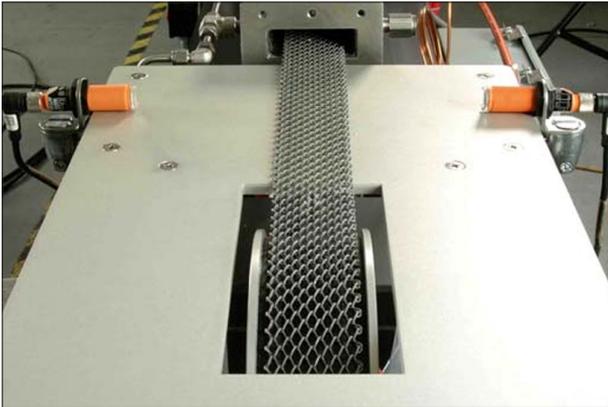


Fig. 4 Proximity sensors at the exit tunnels signal to operators when a work tray is ready for unloading



Fig. 6 Centorr Vacuum Industries belt furnace for the processing of advanced metals and ceramics

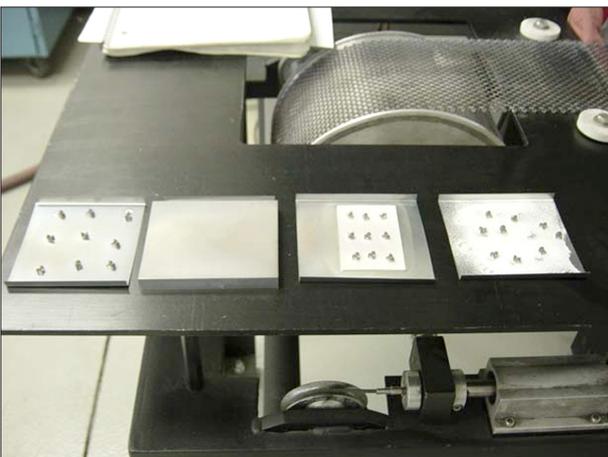


Fig. 7 Trays of small hinges on varied ceramic setter materials including sapphire, ceramic paper and alumina powder



Fig. 8 Photograph of stainless steel component



Fig. 9 Microstructure of part with 97% theoretical density

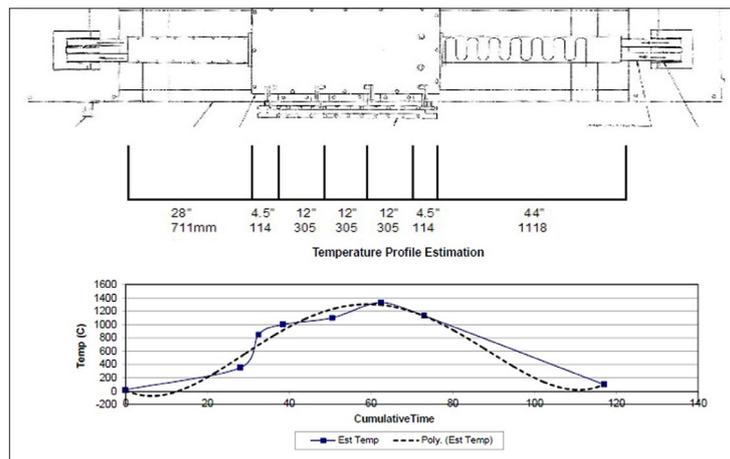


Fig. 10 Belt furnace time/temperature modelling curve for a MIM process run at 25 mm/min, yielding fully sintered parts in 120 minute cycle time

section. By using a specially designed gas labyrinth curtain at the entrance and exit zones, the customer is able to maintain the clean environment inside the furnace even at the tunnel ends, and ensure that the hydrogen process gas is directed up the burnoff chimneys located at both ends.

Final sintering of the parts takes place in the main furnace chamber and the three zones of control are individually adjusted to get the desired heatup, soak time, and cool down profile, which results in theoretical densities of 97% for the 316L components in

cycle times of 120 minutes. Various trays and setting material were also evaluated to minimise sticking of the parts to the metal trays.

Future work in the MIM field is being performed to determine the effect of cycle times on carbon control and investigating blended gas atmospheres in an effort to reduce operating costs. Future development work will additionally include adding debinding capabilities in order to provide a two stage debind and sinter design for MIM parts.



Fig. 11 Graphite hot zone and SiC belt in ceramic belt furnace

Ceramic processing

In 1993, Centorr Vacuum Industries cooperated in a study by Prof. Dale Wittmer, Southern Illinois University (SIU), USA, to develop a cost effective means of sintering selected Si_3N_4 compositions in large volumes required by the automotive industry. While these parts could be reliably produced in a batch process, the major barrier to extensive use of Si_3N_4 components was cost. The primary areas of research had to do with evaluating low cost raw material powders and the sintering process. Previously, Wittmer and Miller showed that it was feasible to sinter large Si_3N_4 disks of 100 - 150 g to full density in a continuous belt furnace in flowing nitrogen gas. A comparison was made between continuous sintering in a commercial belt furnace and sintering in a batch furnace for similar times and temperatures [1]. For the Si_3N_4 sintered in the belt furnace, the average four point flexural strength was found to be 35% higher with a fracture toughness over 22% greater. Remarkably, the overall cycle times for the continuous process were in the range of 3.5 - 4.5 hours compared with 18 - 24 hours cycles in batch sintering furnaces, and this translated to a reduction in time at sintering temperature of 30-60 minutes for continuous compared with more than three hours in the batch process. It was determined that this reduced time in the presence of flowing nitrogen gas, was one of the main reasons the continuous furnace provided enhanced material physical properties. Another explanation why the continuous furnace made parts with improved properties when compared with pressureless sintered furnaces, or even overpressure furnaces is that the belt furnace provides very fast uniform heating and cooling (as the effective hot zone is essentially a 100 mm x 100 mm square), which positively affects the structure of the material.

Depending on formulation, the parts could be processed in graphite, BN, or ALN workboxes with or without the use of packing powders. Early work was done in a tungsten hot zone but

the SiO vapour attack that dissociated off the load was degrading to the refractory metal. Later designs at SIU used an all graphite hot zone with rigid graphite board insulation and graphite resistance heating elements.

And while SiC formation was a concern with the graphite hot zone, the new belt furnace hot zone lasts significantly longer than what is achieved in graphite batch furnaces or tungsten refractory metal designs. Fears about a graphite hot zone contributing to a carbon reaction layer have also been alleviated as there was no difference in the material properties produced in tungsten versus Graphite hot zones.

In recent years, this work has transferred over to other ceramic formulations traditionally processed in batch sintering furnaces and has led to more work in oxide sintering with sapphire formulations for applications in wafer annealing and LED processing, and polycrystalline alumina for metal halide lighting. Benefits of improved densification, minimised weight loss due to dissociation, and preferred crystallinity are possible with this unique belt furnace design.

High temperature sintering

When faced with high temperatures and low dewpoint hydrogen gas requirements for the sintering of refractory metal components used in consumer electronic applications, a customer had a choice of using either high temperature ceramic lined pusher furnaces, or expensive refractory metal batch furnaces. However, because the customer's parts were small and the volumes were not large in terms of mass, large pusher or batch furnaces were not an economical solution.

Due to the fact that refractory metals are very sensitive to oxygen contamination, which is very damaging at high temperatures, the cleanliness of the furnace interior is of paramount importance. The small belt furnace design utilises refractory metal insulation shielding and heating elements which generate no significant offgassing inside the furnace as can be found with traditional porous refractory brick or fibre insulation materials. In fact no 'conditioning' (running the furnace for one to three days upon initial startup to drive off moisture from the insulation brickwork), is necessary with this belt furnace design, and it is simply purged with inert gas for 30 - 45 minutes at the start of the cycle to ensure an oxygen content of under 10 ppm, as interlocked by an integrally mounted oxygen monitor. These belt furnace designs routinely achieve the same dewpoint of hydrogen gas inside the hot zone during operation as is measured at the gas bottle inlet (typically in the range of -50 to -60°C).

The customer in question ended up with multiple belt furnaces with 5 cm and 10 cm wide tungsten mesh belt and 20 - 30 cm long hot zones rated for operation at 2000°C. The units are set up in

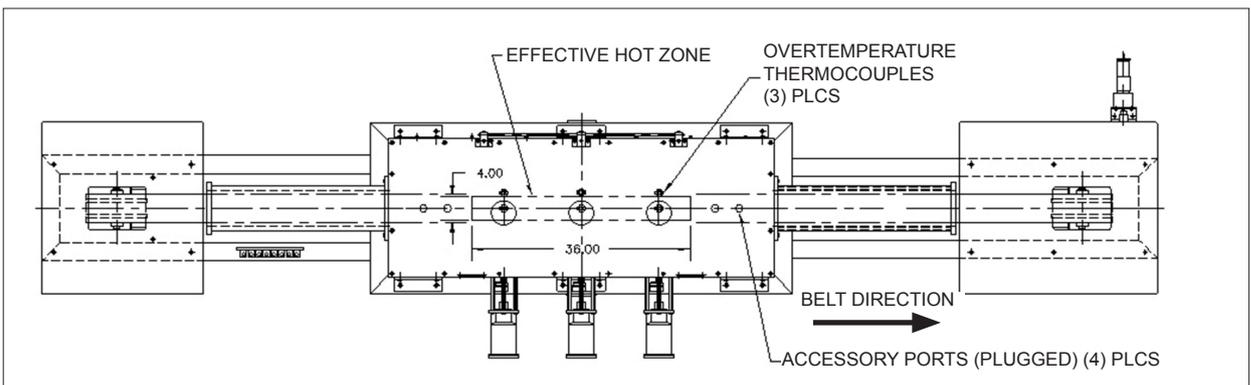


Fig. 12 Hot Zone layout of three zone Ceramic belt furnace

small independent manufacturing cells where a pick and place robot automatically loads the parts onto small trays that are continuously queued up in front of the belt furnace entrance. Proximity switch sensors located at the exit of the belt furnace signal when a boat is ready for unloading, and can be interlocked to put the belt furnace drive motor into hold, if a tray is not removed from the unloading area.

Because the small belt furnaces can be heated up to temperature from a cold start and operational in under 90 minutes, the belt furnaces are run either weekly (Monday through Friday), or can be run daily similar to a batch furnace and cycled on an 8:00 am to 5:00 pm schedule depending on production volumes and holiday schedules. There is no longer the requirement to run the units at lower idling temperatures over the weekend if production demands don't require the throughput, which allows for significant savings in energy costs, as well as process and inert gas costs. At the end of each process campaign the belt furnace is powered down and purged with inert gas for approximately 30 - 60 minutes before it reaches room temperature. Compare this to conventional brick lined units that take two to five days to cool down depending on maximum operating temperatures, and the operating cost savings at this customer were further multiplied. Another benefit is that problems such as tunnel blockages, stuck trays, or minor hot zone or belt repairs can be readily accessed and corrected in under a day including time for cool down and reheating due to the novel hot zone design. The overall length foot print of this customer's small belt furnace fits in a 2.8 m long space, which was less than half the length of a conventional brick lined continuous furnace.

It is important to note that these high temperature continuous furnaces are generally limited to applications with light part weights up to 1.3 kg/linear 300 mm of belt length for molybdenum and tungsten mesh belts, and up to 4 kg/linear 300 mm of belt width for SiC link belts, so thought needs to go into the choice of fixturing for these furnace designs. Operators should consider the use of low mass plates or trays, made of thin molybdenum or tungsten sheet material, with lightweight ceramic separator plates if required by the process. Dense ceramic plates cannot be used in most instances, as the fast cooling that occurs in the cooling tunnel results in thermal shock and cracking of the ceramics.

Brazing

Conventional brazing encompasses a wide variety of techniques and materials, and can be performed in a variety of furnace equipment; however, for some specialised braze materials either vacuum batch processing or hydrogen belt or pusher furnaces are used. One such niche process would be for the precision brazing of very small, lightweight parts such as those found in the medical industry. The secret to excellent brazing in this case is summarised by the mantra of "get to temperature quickly, stay there a short period of time, and get down in temperature quickly". The primary reason for this sentiment is the desire to minimise high temperature reactions between the braze filler metal and base metal which can occur if the two materials are in contact at temperatures above the liquidus point for long periods of time. A second corollary reason is that the shorter the overall cycle time, the less time there is available for contamination of the base metal to occur which can result in brazing defects such as poor adhesion between base metal and filler metal, and insufficient bonding properties.

In order to achieve this goal a very clean "vacuum like" atmosphere is desired to ensure little to no oxidation of the base metal. This means conventional belt or pusher furnaces could not be used because these designs cannot offer the fast cycle times and level of cleanliness and low dew point required for some braze processes, such as those

involving active metal or Ti-Cu-Ag/Ti-Cu-Ni formulations.

A customer had a difficult brazing request for a small medical part that used a tiny amount of braze at temperatures under 1100°C. After testing in various vacuum batch furnaces and batch hydrogen units it was decided to try the parts in the belt furnace using hydrogen process gas to maintain a clean, oxygen free environment. While the parts processed in the batch cycles were acceptable, there were braze blush variances throughout the load due to the different thermal conditions each part was subjected to, and each cycle took on the order of three to four hours, whether the unit had a full load or not.

After completing two days of test runs at varying belt speeds and zone temperatures, the customer achieved parts that exited the 15.25 cm wide by 91.5 cm long hydrogen belt with excellent surface finish, a bright shiny exterior, and excellent braze joints. Furnace speeds of 75 mm/min were used during development, which provided a door to door time of just under 40 minutes. For budget reasons and total capacity requirements, this customer opted for a 10 cm wide belt furnace with an 20 cm long hot zone. While a belt speed of approximately 13 mm/min would have been appropriate to provide a similar time at temp inside the furnace hot zone, compared with the lab testing in the 91.5 cm long unit, this customer made their initial runs at the same 75 mm/min speeds and found that the parts came out every bit as shiny and clean as the lab testing done in the longer belt furnace design. While not true for all applications, because the mass of these parts were so small this customer found that their parts would almost instantaneously heat up inside the hot zone, being bathed by the low dewpoint H₂ gas, and due to the small amount of braze material present, result in contamination free joining in a matter of seconds. This application in a way simulated the speeds of batch induction brazing, except for the ability to continuously and safely maintain the clean hydrogen gas environment.

The only operational issues onsite have been related to environmental conditions on the shop floor. Due to the small size of the furnace openings, if the entrance or exit tunnels are close to a loading dock or large window, the gas curtains and process gas chimneys can have their flow patterns disturbed resulting in frequent flow adjustments to the gas panel. It was also learned that the furnace preferred to be run with a full load of workboats to get consistent gas flow dynamics, or else the process gas that is introduced into the main chamber and entrance/exit tunnels will preferentially exit out the furnace end with the smallest blockage. Happily, the customer has noticed that the water cooled shell of the furnace results in almost no heat radiation to the surrounding plant environment (as opposed to the significant heat generated from conventional brick lined furnaces), and the furnace operation has not impacted facility heating or air conditioning utilities

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