Vacuum furnaces were initially developed to process reactive and refractory materials under protective conditions at extremely high temperature. The technique of heating materials in vacuum, albeit with rather limited apparatus, was being used earlier than one might think. An Englishman in the first decade of this century, for example, prepared tantalum powder in vacuum in order to produce pure tantalum wire.

Vacuum furnace evolution and metallurgy developments over the intervening years have followed parallel and frequently interrelated paths. Today's sophisticated vacuum furnaces make use of high performance materials while practically all superalloys and many "tailor-made" composite materials have been exposed to vacuum furnace processing sometime during production.

Vacuum furnaces are now used for a number of commercial metallurgical processes including melting and casting, heat treating, sintering of powdered metals, hot pressing, diffusion bonding and degassing. In this article the technology of heat transfer in vacuum furnaces will be reviewed briefly and the state of the art of vacuum sintering furnaces will be examined in some detail.

HEAT TRANSFER

In vacuum furnaces heated by resistance elements or induction coils with susceptors, radiation is the dominant heat transfer mode. In the absence of air or gas, convection or conduction modes are not effective. Loads are heated from their outer surfaces which are exposed to the radiant heaters. The "ideal" radiative enclosure is the ultimate goal of vacuum furnace designers. A sphere surrounding the object to be heated would be ideal. The sphere's inner surface would be at uniform temperature and the outside perfectly insulated. The perfect radiator or "black body" must have an absorbivity of unity and, therefore, a reflectivity of zero.

In practice, black body conditions are not attained, but vacuum furnaces can be designed to produce remarkably uniform temperatures with load geometries which are often challenging. Vacuum furnaces have been built which heat ten ton loads, ninety feet long, within ±18°F uniformity. Smaller furnaces with heat zone shapes approaching cubes can achieve ±10°F uniformity.

FURNACE DESIGN

Two basic design approaches have been used to provide hot radiating enclosures in which to heat work. The "hot-wall" approach is based on evacuating a muffle which is heated externally with a conventional electric or fossil fuel-fired furnace. The main limitation of this design is the strength of the muffle. Both external pressure of the atmosphere and sagging limit operating temperatures and determine useful life. Temperatures above 1800°F can be achieved if the external electric furnace is, in turn, evacuated. The use of a muffle for these higher temperatures, however, is usually redundant. By introducing true "cold-wall" design principles, both the refractory brick insulation and the muffle can be eliminated. The resulting internal heating element thus makes possible a fast-response furnace capable of high temperatures, reliable performance and versatile operating cycles.

A cold-wall vacuum furnace consists of a work space surrounded in succession by radiant heating elements of various shapes, arrangements and materials; thermal insulation of either metallic sheets or fibrous materials; a supporting structure; and a water-cooled steel enclosure which can be evacuated and back-filled with any desired atmosphere (Figure 1). Typically, internal resistance heating elements can be raised to close to maximum operating temperature almost instantaneously. Metallic heating elements, alloy, molybdenum, or tungsten depending upon temperature requirements, exhibit a positive temperature coefficient of resistance which dictates selection of controls for and rates of applying power. Graphite heating elements

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LOAD HEATING

Furnace loads can vary from a single solid piece of material such as a die or roll to trays containing large numbers of small pieces. From the standpoint of energy applied for heating and removed during cooling, the weight and specific heat of the load are governing factors. To the extent that spacing and shadowing exist in the load, however, heat transfer by radiation will favor the outer portions of such a load and internal sections which are shadowed will take longer to heat up.

A 600 lb. load of low carbon steel structural angles with 1/2" section thickness was heated in a typical cold-wall furnace to demonstrate furnace and load response. Temperatures were measured with the furnace control thermocouple located in the hot zone near the heating elements, a thermocouple inserted 1" inside the edge of the load and a thermocouple in the center of the load (Figure 2). Power to the heating elements was reduced at about 1000°F to maintain the desired load heating rate. The sequential heating effect resulting from self shielding in the load is evident. This was purposely an extreme case for illustrative purposes, since most applications require slower heating rates for process reasons. Automatic controls, which will be described later, provide for very even and uniform heating over the entire temperature range.

Difficult geometries (i.e.: stacks of plates, tubes or coils) benefit from furnace conditions approximating the ideal black body enclosure. Furnaces are available with heating elements on two sides, on two sides plus top and bottom, or on all sides of the enclosure. For both maximized radiant transfer during heating and uniform heating conditions during soak in larger furnaces, multi-sided heating arrangements are preferred (Figure 3).

VACUUM SINTERING FURNACES
EARLY DEVELOPMENTS

Vacuum furnaces were first used in the production of cemented carbides in 1931 to sinter multiple carbide alloys based on mixtures of WC-TiC with additions of (Ta, Nb)C. Conventional atmosphere furnaces could not be used for these materials because hydrides and nitrides would be formed.

Pioneering cemented carbide producers
used induction heating with a graphite susceptor inside quartz and the induction coil outside, or, alternatively, a completely enclosed induction coil within a stainless steel vacuum chamber. Induction was considered the simplest approach to achieve the temperatures required to liquid phase sinter the complex carbides. The number of vacuum furnaces used for this process grew during the 20 years following 1931 until, the late 1950's and early 1960's increasing production requirements dictated the development of semi-continuous vacuum furnaces. These furnaces had vertical load locks and separate vertical dewax-presinter and sinter furnaces with resistance heaters. They were mounted on top of a large cylindrical vacuum chamber in which a multi-station rotary table indexed loads through the entire cycle and allowed them to cool before unloading. The electric resistance heating elements used in these furnaces began to be used in vertical single chamber batch furnaces of the same size. Cylindrical, top-loading units, they were primarily used for mass production of small parts such as indexable tips. It was also in the 1960's that vacuum sintering of the straight WC-Co alloys began to become a more widespread practice.

**FRONT LOADING BATCH FURNACES**

The sharp climb in energy costs during the past decade has given added impetus to the trend to vacuum in preference to atmosphere furnaces which consume lots of hydrogen and use large amounts of electric power to heat it.

In addition to offering the ideal protective atmosphere at lowest operating cost, vacuum furnaces can be precisely controlled and equipped with process gas attachments to provide for a close adjustment of carbon balance. The close temperature uniformity achieved in today's resistance furnace helps maximize yield for all types of parts being processed.

The combination of these features plus the need for higher production capacity in terms of both size of parts and tonnage throughput led to development of horizontal batch vacuum furnaces which could be installed at floor level and loaded with conveniently-operated fork lifts. Although dewax cycles were most often conducted in separate atmosphere or vacuum furnaces, the new front-loading vacuum furnaces included provisions for wax handling and fast cooling by recirculated gas thus making them capable of complete cycle processing with a high production throughput.

Furnaces are now available with work spaces ranging from 8 in. by 8 in. to 20 in. long to 24 in. wide by 18 in. high by 72 in. long with 1600°C capability and 10^-3 torr ultimate pressure achieved with Roots-type mechanical booster pump systems. Load capacities range from 40 kg to 750 kg (Figure 4). Parts can be dewaxed (paraffin wax), heated to presinter stage and quickly cooled prior to removal for final sizing or they can be completely sintered in one cycle.

**HEATING RATE CONTROL**

The success of the dewax operation for large batches or large parts in a resistance furnace of these dimensions depends to a large extent upon the precision of low temperature control during initial heating. Power supplies with the high kVA rating required to heat the vacuum furnace for final sintering temperatures are difficult to control automatically in the dewax temperature range. The power supply may tend to cycle on and off, causing the furnace to exceed the desired temperature. Heating too fast can lead to hairline cracking in the compacts and to other problems caused by too rapid wax evolution, or overheating and breakdown of the wax.

To overcome this drawback, a solid state electronic circuit (Parametric Power Control) has been devised to smooth out temperature ramps at the very lowest levels in vacuum sintering furnaces. All vacuum sintering furnaces use a temperature sensor such as a thermocouple or pyrometer to provide a signal for proportional control so that temperature can be automatically programmed through various rise/hold patterns. For a vacuum sintering furnace, the requirements of the temperature control system are very different between the low and high temperature ranges. At sintering temperatures, radiant heat transfer between the heating elements and the load is the dominant factor. Very sensitive control is achieved through the entire control loop at high temperatures.

When heating from ambient through the dewaxing range, problems start with the initial application of power to heat the resistance elements. As soon as the programmed temperature set point exceeds the actual temperature, the controller will call for full available power. When full furnace power is applied, the temperature will rise so fast that it will overshoot the set point by a substantial margin, even though the power will be reduced to zero when the measured temperature reaches the set point. The furnace will eventually cool below the programmed set point temperature. Power will then be applied and a cyclical pattern established which will continue until a higher temperature in a more responsive radiant range is reached.

Parametric Power Control makes smooth start-up from ambient conditions possible by modifying the temperature control signal to apply a constant low power level, thus causing the furnace temperature to rise at the desired preprogrammed rate. With proper adjustments the Parametric Power Control can provide extremely smooth heating ramps at all desired temperature levels for any type of combination dewax, presinter, and sinter cycle. (Figure 5) The precise heating required to uniformly bring large loads of thick section carbide products up to and through the dewax point can be controlled accurately.

![Figure 4. Dual vacuum sintering furnace has two 24 in. x 18 in. x 36 in. long hot zones for 1600°C in separate vacuum chambers operating from a common pumping system, power supply, cooling gas recirculation system and control console.](image-url)
NEW FURNACE DEVELOPMENTS

A 300 kg vacuum sintering furnace has been developed with graphite tube heating elements above, below and on both sides of the charge area. This four-sided heating provides excellent thermal response and permits uniform heating, maintaining the heat zone within ±5°C (Figure 6). Longitudinal orientation of the resistance heating elements from front to rear also provides flexibility for two alternative hearth arrangements. The furnace can be supplied with an 18 in. wide by 36 in. long hearth, 12 in. load height, or, with the entire heat zone assembly rotated 90° within the vacuum chamber, a load space 12 in. wide by 36 in. long and 18 in. high. Because the heating element geometries are not changed, thermal response and temperature uniformity are the same for both positions. This flexibility in design of the hot zone permits the user to select the orientation best suited for the particular work being planned for the furnace.

Microprocessor-based programmers with controllers provide additional operational flexibility in that many different programs can be stored and additional events in the furnace operating cycle can be tied into the program, making possible completely automatic furnace operation.

In addition to improvements in vacuum furnaces designed for carbide sintering, vacuum furnaces have been adapted to produce new powder metal materials being developed. Powders which have special properties and which reduce or completely eliminate the usage of scarce or expensive materials are being prepared in vacuum furnaces. Vacuum-tight, pre-exhausted chambers maintain the purity of process gases introduced to react with the metallic powders to develop desired characteristics. Several medium size horizontal vacuum furnaces are operating reliably at temperatures in the 2000°C range on a day after day production basis.

New sintering furnaces also provide metallic hot zones for other materials for which a graphite furnace environment is not suitable. One example is a tungsten heating element furnace with extremely close temperature uniformity (Figure 7). This furnace provides capacity for development programs. Larger sizes have been constructed for production with provisions for operation in inert or reducing gas.

SUMMARY

Progress in the design of horizontal-loading batch combination cycle vacuum sintering furnaces continues to provide cemented carbide and other powder metal users with increasingly easy to use, more efficient, repeatable and reliable sintering equipment. Vacuum chambers provide an ideal way to use process gas economically and with accurate control. Automatic operation has advanced to a degree unprecedented in high temperature furnace technology.

As new materials are developed and put into production for hard metal applications, vacuum sintering furnaces will find increasing use in preference to atmosphere furnaces because of their ability to provide consistent process environments, extremely close temperature uniformity, various atmospheres, precise heating ramps and hold time cycles, repeatability, and economy in operation.