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Vacuum furnaces for metallurgical processing

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Abstract: Large scale vacuum furnaces are becoming crucial in metallurgical processes like melting, casting, heat treatment, degassing, annealing and brazing. The vacuum conditions help in reducing contamination (like oxidation) and improve the micro-structure to get favourable metallurgical properties for industrial applications. The vacuum also reduces the melting and boiling points of metals and alloys, thereby reducing the electrical power requirement in high temperature furnaces for clean metallurgical processing. The present paper describes the design criteria of vacuum metallurgical furnaces involving vacuum equipment, heating elements and process instrumentation.

Keywords: *Vacuum furnace, Pumps, Hot zone, heating elements, thermocouples, safety interlocks.*

INTRODUCTION

Vacuum furnaces are finding increasing application in the metallurgical processing of high temperature materials, where pick up of Oxygen and Nitrogen during melting, casting and subsequent heat treatment is minimum[1]. The vacuum environment is a pure protective atmosphere than any inert gas. For most of the metallurgical processes, such as sintering, brazing, annealing, and melting & casting, a vacuum of the order of 1×10^{-5} Torr is adequate [2]. Hence, the vacuum system consists of a combination of a mechanical vane pump/piston pump and oil diffusion pump. In case of furnaces which involve heavy degassing, a roots pump (increasing the effective pumping speed of roots-rotary combination) is essential for maintaining the required backing pressure of the diffusion pump for its efficiency.

The pumping system includes other sub-assemblies such as chevron baffles, valves and pipe lines.

The various types of valves used in the pumping line are angle valves, ball valves, butterfly valves and gate valves. Gate valves are the best suited for vacuum pumping line, as they offer minimum impedance to gas flow. For high vacuum line, angle valves fabricated out of stainless steel are used. The pipelines in the high vacuum side (near the furnace chamber) should be designed to have high flow conductance (C), so that the effective pumping speed (S_{eff}) of the HV pump at the chamber is sufficient [3]. S_{eff} can be estimated from the manufacturer's specified maximum speed (S_{max}) and the conductance (C) by the following equation:

$$\frac{1}{S_{eff}} = \frac{1}{S_{\max}} + \frac{1}{C}$$

The throughput, (Q) representing the amount of pumped gases from the furnace chamber at any pressure (P) is given by $Q = P*S_{eff.}$

CLASSIFICATION OF VACUUM FURNACES

Vacuum furnaces are broadly classified into two types, i.e cold wall and hot wall types

Cold wall type: The vacuum chamber is water cooled and kept at room temperature. The job is kept in a hot zone (resistance heating/induction heating) supported by thermally insulating structures. Here the size of the hot zone is small and energy input is less. But degassing from the walls of the vacuum shell remains as a problem to be solved.

***Hot wall type**:* The vacuum chamber/muffle is externally heated (by resistive heating) and job located inside receives heat mainly by radiation. Degassing/water vapour problems are absent. It can handle large quantities of

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jobs to be processed. However, these are not popular, as they require special vacuum seals which can withstand high chamber temperature and thermal insulation outside to reduce electrical heating power requirement and reduce room heating. Hence the rest of the paper deals with cold wall type furnace.

DESIGN OF VACUUM CHAMBER CONSIDERATIONS FOR COLD WALL TYPE FURNACE

Fig 1 shows the schematic of a typical cold wall type vacuum furnace for high quality brazing application. It consists of a double walled cylindrical chamber, matching diffusion/rotary vacuum pumping system, hot zone with heating elements and shields, several control valves and sensors (for pressure and temperature measurement).

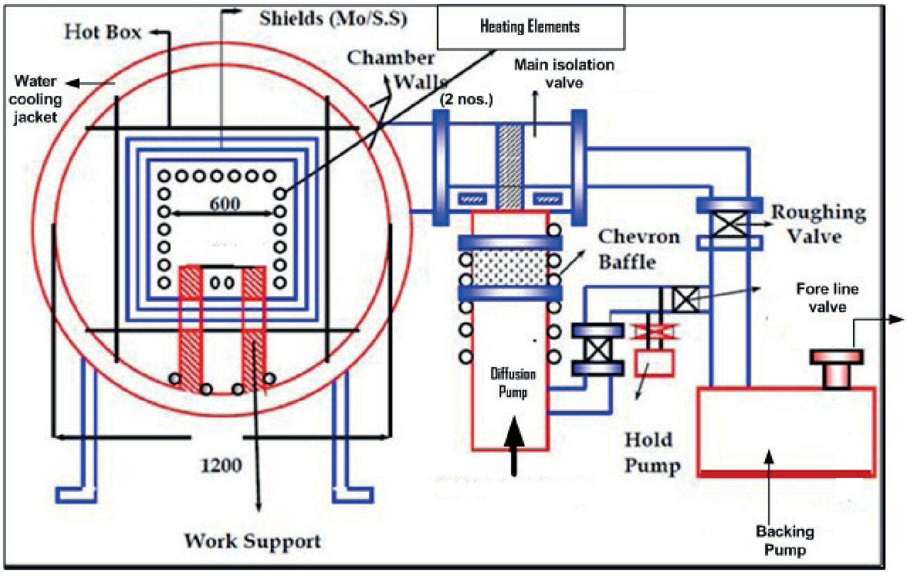

Fig 1: Schematic of typical double walled vacuum brazing furnace (dimensions in mm)

For the fabrication of chamber, either hot rolled mild steel or stainless steels of grade 304, 304L and 316 are used. The chamber should not collapse under vacuum. The thickness of the chamber should be optimised to stand the pressure difference (1 Kg/cm^2 + cooling water pressure in the outer jacket) and not to increase the mass and out gassing rate. Further it should be able to prevent leakages from outside atmosphere, when it is pumped down to a low pressure and to permit the material to be processed without any damage to the chamber as well as to the product. However, thinner sheets (to create lower weight) can also be used by reinforcing the strength of the chamber by welding stiffeners. For vacuum applications, argon arc welding is preferred as the contamination from flux is totally avoided. If complete argon arc welding is not possible, we can weld few tags and then remaining by brazing with compatible metals/flux.

It is essential to have good surface finish in all the grooves meant for 'O' rings,Wilson seals for rotating devices and door seals. Regarding shape, cylindrical and rectangular chambers are very often used. The best method of cylindrical chamber fabrication is from a sheet cylindrically rolled up and dished heads on both ends welded throughout. Although this type of construction offers good mechanical strength, some designers select a rectangular cross section for efficient space utilization and use of low capacity vacuum pumps. However, rectangular chambers have to be designed for higher stresses and obviously require higher shell thickness.

With respect to orientation/operation, the furnace chambers are classified as:

i. Vertical type, bottom loaded for heavy jobs loaded from bottom opening
ii. Vertical type, top loaded where top dished end can be lifted by an over head hoist and the job can be loaded by a crane.

iii. Horizontal type, front loaded (by opening the front door with suitable seals and clamps) for long light weight components/flat pieces etc.

For the purpose of external water cooling, most of the manufacturers prefer double walled configuration as the cooling efficiency is maximum and so the furnace can be designed for very high temperatures (1400°C). The basic disadvantage in this design is the repairing of any water leak developed in the inner vacuum chamber. This is dangerous, as one cc of water leaking in a vacuum chamber at 10^{-5} Torr produces nearly 108 litres (i.e. 100 million litres) of vapour, contributing heavy unwanted load on high vacuum pumps. The contamination due to water vapour and other species can be monitored using a residual gas analyser (RGA) connected with a valve to the vacuum furnace chamber.

HOT ZONE FOR COLD WALLED FURNACES

The primary criterion for the hot zone of vacuum furnace (See Fig. 1) is that the entire volume of the work piece should experience the same temperature. The important parameters on which the thermal uniformity of the hot zone depends are size, shape and location of the heating elements, rate of heating, the type of insulation/shields used etc. In general, the hot zone of the furnace consists of suitable heating elements, thermal insulation/radiation shields to conserve the heat and ceramics for electrical/thermal insulation.

Heating Elements: Kanthal is not recommended for temperatures above 900°C in a vacuum, as its electrical characteristics change in course of time due to chromium losses. Molybdenum provides the high power density required for vacuum furnaces. However above 1400°C, the discharge voltage is 40V, and so the cross-sectional area of the rod/strip has to be increased which in turn imposes constraints on the formability. Hence, for operations above 1400°C molybdenum is not preferred. Besides, after a few heats, grain growth occurs which will be sufficient to cause the material to become very brittle, thereby seriously limiting its ability to withstand any thermal shock due to which frequent replacement is required.

For temperatures between 1400-1800°C, tungsten/tantalum is preferred due to its capability to withstand thermal shock. However, fabrication difficulties and higher initial costs are significant points to be considered. With the availability of high-quality graphite, graphite elements have been widely used up to 2000°C. The advantages of graphite heating elements are Low initial cost, no change in resistance at high temperatures, high hot strength, ability to withstand reasonable mechanical shocks and accidental high-temperature air breaks and ease of fabrication of the hot zone due to its availability in the forms of thin sheets, strips and cloth. However, the user has to ensure that the heating elements do not come in contact with the job, as carbon can diffuse into base metal interstitially. If carbon contamination is not tolerable even in ppm level, these elements are not recommended.

Depending on the uniformity of temperature required the heating elements are located on either on two sides, four sides or six sides. For zone trimming, the heating elements are located on all six sides. However, power coupling is adjusted depending on the size, shape and uniformly of temperature required, generally, this configuration is followed in brazing furnaces.

Insulation/Shields: The efficiency of the furnace depends on the way in which the heat is conserved. The main mode of heat loss in any furnace is through radiation and hence designing radiation shields/insulation plays an important role in the overall design of the furnace. The essential requirement is the use of highly polished metallic sheets (with low emissivity) as shields. Depending on the operating temperature and the shield materials, the number of shields needed may vary. The common materials widely used for shields are stainless steel, molybdenum and tantalum. Of these molybdenum and tantalum have very low emissivities in comparison to stainless steel and hence minimize heat losses. Thus, for high temperature applications above 800°C, four polished stainless steel sheets are used. For temperatures up to 1600°C, six shields, consisting of a combination of SS + Mo, are used. Graphite felt can also be used as the insulating material in place of shields where carbon contamination is not critical.

Induction heating: Induction heating is characterised by excellent control over alloy composition because of the stirring action generated by eddy currents. Hence it is preferred for alloy melting and refining processes. Induction stirring homogenises the melt as well as brings the reactants to the melt vacuum interface so that the reactions can proceed rapidly. Induction is also used for applications like sintering, brazing, annealing etc, where temperature accuracy on the component need not be so accurate. With the development of medium frequency generators using thyristor techniques, it has become possible to design vacuum furnaces having induction coils located inside the cold walled furnace. The frequency of these generators is in the range of 0.5 to 10 KHz. The induction coils are fabricated out of conventional copper tubes which are water cooled using softened water. If needed, magnetic shunts can be used to prevent induction leakage.

PROCESS INSTRUMENTATION FOR VACUUM FURNACES

Today, microprocessor based temperature programmers/controllers, wherein it is possible to preset heating rates, holding time, cooling rates etc., are available. Temperature Recorder/Over temperature protection devices are used when multiple thermocouples are used for recording the temperature at different locations of the job. Indicating lights corresponding to different on conditions display the status of the furnace operation. In addition, alarms and fault indication lights are also to be provided. It is advisable to keep a helium- Leak detector as an essential accessory for trouble shooting and leak rate measurement.

Safety interlocks linking furnace operation with water flow, overheating and valve closing are essential to ensure that proper sequential operations are followed. This helps in increasing the life and preventing the frequent breakdown of the furnace and its subsystems.

Thermocouples for temperature measurement in vacuum furnaces are to be chosen on the basis of (a) useful temperature range, (b) sensitivity, and (c) speed of response. Metals and alloys with high vapor pressures should be avoided where elevated temperatures and bake-out are scheduled. The range of emfs produced by most conventional couples in use is up to about 50µv/°C. The speed of response is largely determined by the size of wire used for the two metals, and is greater with finer wires than with larger sizes. Platinum and platinum-rhodium alloy couples are expensive but can be used at temperature up to 1500°C in air. Chromel-Alumel thermocouples are widely used in industrial and laboratory equipment because of their uniformity, corrosion resistance, sensitivity, and relatively low cost (compared to platinum). Chromel-Alumel thermocouples can be used between -200 and +1260°C in the air or non-reducing environments. Copper-Constantan junctions can be used in the range -260 to +350°C, the upper value being set by the rapid oxidation of copper at higher temperatures.

HEAT TRANSFER IN VACUUM FURNACE SYSTEMS

Above approximately 10 Torr, the heat transfer through gas inside a relatively small chamber is dominated by convection (bulk motion of gas). Between 1 to 10^{-3} torr the heat transfer is through gas conduction which is a linear function of pressure (hence applied in thermal conductivity gauges). In high vacuum conditions (Below 10^{-4} Torr) the heat transfer is mainly due to radiation and to some extent by solid conduction through support structures. It is to be mentioned here that if two solid pieces are in just apparent contact in a vacuum chamber the heat transfer is very poor, as the real area of contact between two rigid bodies is usually only 0.1% of the apparent area. Heat is transferred only through a few touching high points between the plates. To increase the heat flow, one (or both) of the plates should be soft and high clamping forces must be used. Even then, the heat transfer by conduction will be limited to the small areas of contact created near the clamping bolts.

In view of the above facts, we can calculate the temperature rise of the job in a vacuum furnace due to resistive heating from surrounding heating elements, based on radiation formula.

$$Q_{12} = \delta_{12} A_1 (T_1^4 - T_2^4)$$

$$\text{where } \delta_{12} = C_b / 1 / E_1 + A_1 / A_2 (1/E_2 - 1)$$

where C_b = *radiation* coefficient of the black body i.e. 5.7 x 10^{-11} KW/(m^2T^4)

$E_1 E_2$ = emissivities of 1st and 2nd shields.

A_1, A_2 = Areas of the 1st and 2nd shields

T_1, T_2 = Temperatures of the 1st and 2nd shields.

As the radiation between two surfaces is proportional to the difference in fourth power of temperature, the heat flux in the initial period to the job from a heating element (reached fast to the equilibrium, say 800°C, due to resistive heating) is very high. Hence if we want more or less linear slow increase in job temperature, it is very important to raise the temperature of the heating elements in steps and allow the job to reach that set limit. For heating in several steps using suitable temperature controllers, we can apply radiation formula piecewise taking the necessary temperature limits. Once heat flux is known, the temperature rise can be calculated using the physical properties of the job namely specific heat (more or less a constant value in high temperature range) and mass. For calculating heat flux due to support solid structures, one can use fourier equation as given below, However, in high temperature region for most of the metals thermal conductivity does not change much and can be taken as constant.

$$Q = A/L\, K(T)dT$$

CONCLUSIONS

The importance of using vacuum environment in large vacuum furnaces is increasing to process metals and alloys at high temperatures. The technical details are explained in this paper, highlighting vacuum and heat transfer aspects.

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