Review Article

Vacuum Heat Treatment of Engineering Materials; The Technical Aspect

S Khan*, M A Khan and A ul-Haq

Metallurgy Division, Dr. A. Q. Khan Research Laboratories, P.O. Box No.502, Rawalpindi, Pakistan

In view of the rapid technological development taking place around the globe, advanced materials processing comes into sharp focus. Heat treatment in vaccum offers a plausible alternative over conventional heat treatment process as the former allows to heat treat finished parts and components with minimum surface degradation. The paper reviews various vacuum heat treating processes and their advantages over the conventional processes with specific reference to nuclear, missile and aerospace materials.

Key words: Vacuum technology, Heat treatment, Atmosphere.

Introduction

The space and nuclear program world over has given considerable impetus to the growth and development of vacuum technology. Today, even in the third world countries, the development of vacuum technology has been initiated to meet the need of their nuclear research programs and later to utilize it in other applications including the space program (Raja Rao 1987).

Until the advent of vacuum furnace, the non-vacuum application of heat treatment was generally accomplished either in air or in specially prepared atmosphere, such as CO, CO_2, H_2O and H_2 which was oxidizing, reducing, carburising or inert. The manufacture and chemical supervision of such protective atmosphere with regard to the furnace construction, the material and the techniques involved were costly and difficult to maintain the respectable results (Diman 1975). Many equipment manufactures have soon realized the advantages of "no atmosphere". Consequently vacuum furnace technology has been developed which is discussed in this article (Van Atta 1965; Diman 1975).

Vacuum Heat Treatment

Vacuum heat treatment is relatively a new development in metallurgical processing; it was started commercially in 1966. Vacuum heat treatment can be employed for steel, non ferrous and super alloys in place of conventional methods such as controlled atmosphere or salt bath processes. Vacuum furnaces provide the necessary condition without which the heat treatment of various sophisticated components/parts for aircraft engines, nuclear centrifuges and missiles would not be possible. Heat treatment under vacuum is gaining more appli-

cations since it ensures consistent metallurgical characteristics on the component's materials together with less scaling and brilliant surfaces finish (Prabhvdev 1995).

Vacuum heat treatment of metals is carried out in an enclosure that is evacuated to partial pressure compatible with the particular metals and processes. Vacuum furnaces are used for annealing, carburising, hardening and tempering and for stress relieving. Better results can be obtained with assurance of quality and the processing can be completely automated minimizing the need for skilled operators and technicians (Matal Hand Book 1981).

A Typical Vacuum System

Vacuum is defined as the absence of any atmosphere in a given volume. The minimum requirements for vacuum system are simply a container, which will not leak under outside gas or vapour pressure, from which a pump is capable of removing most of the air. In practical vacuum heat treatment, however, it is usually necessary to provide for control and measurement of the vacuum, rapid attainment of vacuum. The most common units for measuring vacuum is torr or milibar. The order of vacuum required depends upon the process to be used and may be classified as: (Thompson and Finn 1988; Fabian 1993).

1. 10^0 to 10^{-1} mbar-commonly used for brazing and some sintering operation.
2. 10^{-1} to 10^{-4} mbar-used for brazing, low vacuum heat treatment and most of the hard metal and steel sintering.
3. 10^{-3} to 10^{-5} mbar-used for diffusion bonding, heat treating and brazing.
4. 10^{-5} to 10^{-6} mbar-used for aluminium brazing, reactive metal sintering and heat treatment.
5. 10^{-7} to 10^{-9} mbar-used for semi-conductor applications.

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*Author for correspondence

In general, the vacuum range between 10^{-3} to 10^{-6} mbar is referred to as high vacuum, which is normal operating range for heat treatment. Figure 1 provides a comparison in relation to the atmosphere which shows that as we move towards high vacuum side the pressure becomes low. Vapour pressure, which is the gas pressure exerted when a substance is in equilibrium with its own vapour, increases rapidly with temperature, because the molecules in the outer surface of the solid material have higher energies than others. Consequently they escape as free molecules or vapour. For example, if brass is heated in a vacuum at a temperature of 540°C and a vacuum level of the order of 10^{-3} mbar, the zinc will start to vaporize and brass component will be soon converted to copper sponges. Vapour pressure considerations are therefore most significant in vacuum furnace operation (Metal Hand Book 1981).

DESCRIPTION OF VACUUM FURNACE

Vacuum furnaces, simply described, are the low-pressure vessels which have valves and pumping system and that are used in conjunction with a shielded and heated volume that will accept a work charge. The typical cycle for heat treatment application is to load the work charge, cool the work charge, back filled the heating chamber to atmospheric pressure and remove the work charge from the furnace.

A great deal of advancement has been made in vacuum furnace technology incorporating controls of pressure, time and temperature regime and recording systems. The design and functional capabilities can be categorized into three main groups (Source Book on Heat Treatment, Material & Process 1977; Source Book on Heat Treatment, Production & Engineering Practice 1977; Metal Hand Book 1981).

1. Batch furnace, under this
    a. Hot wall furnace
    b. Cold wall furnace
    c. Top loading
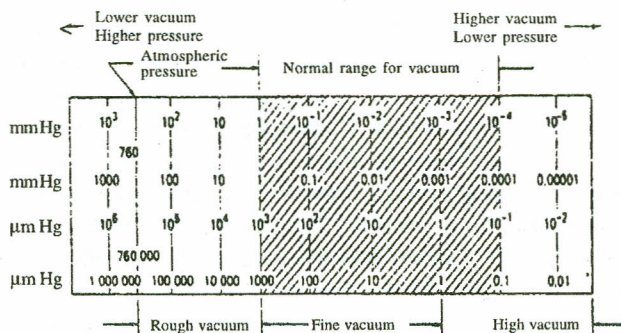   d. Bottom loading
    e. Horizontal loading with or without liquid quenching facility.
2. Semi-continuous furnace with quenching facilities.
3. Continuous furnace with quenching facilities.

It must be mentioned that continuous vacuum furnace offers great versatility of design, large production volumes and high thermal efficiency.

VACUUM PUMPING SYSTEM

Pumping system are usually categorized as (Leybold Data Book 1987; Hameed *et al* 1991).

a. Roughing pump, consisting of piston or rotary positive displacement mechanical pump which operates on fluid flow principle used as single unit or with a back up pump to achieve a pressure level up to 10^{-3} mbar. A mechanical pump compresses gas or air at its inlet and discharges it to the atmosphere.

b. Diffusion pump, to achieve vacuum level below 10^{-3} mbar; a vapour diffusion pump is generally used. Molecules of air or gas pumped out of the chamber are diffused with heavy molecules (of pump fluid, silicon based oil) directed downward at high velocity towards outlet and removed by fore pump. The fluid should have low vaporization and high flash point 330-350°C. Figure 2 shows vacuum furnace system for heat treatment.

HEAT TRANSFER IN VACUUM FURNACE

Resistance heating and induction heating are the two most common heating sources within the cold wall vacuum furnace. Resistance heating uses molybdenum and induction heating uses graphite elements. In the absence of air or gas atmosphere heat transfer by convection and conduction can
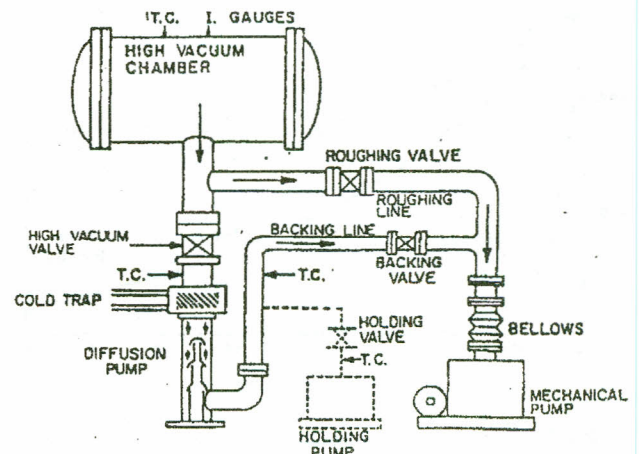
Fig 1. Range of pressure and their relation to atmosphere.

Fig 2. A typical high vacuum system.

not occur. Heat transfer from the hot resistance element to the cooler workload occurs only by radiation. Heat transfer from hot body to cold body occurs in accordance with the fourth power of the relative absolute temperature. Heat transfer per unit time is equal to $T_1^4 - T_2^4$, where T_1 and T_2 are the absolute temperature (Brown and Marco 1958; Fabin 1993). As the heating rate under vacuum is governed almost entirely by radiant energy, this can greatly affect the manner in which the workload should be placed. Figure 3 shows the mechanism by which the heat is transferred to the work piece under vacuum for static and distributed load.
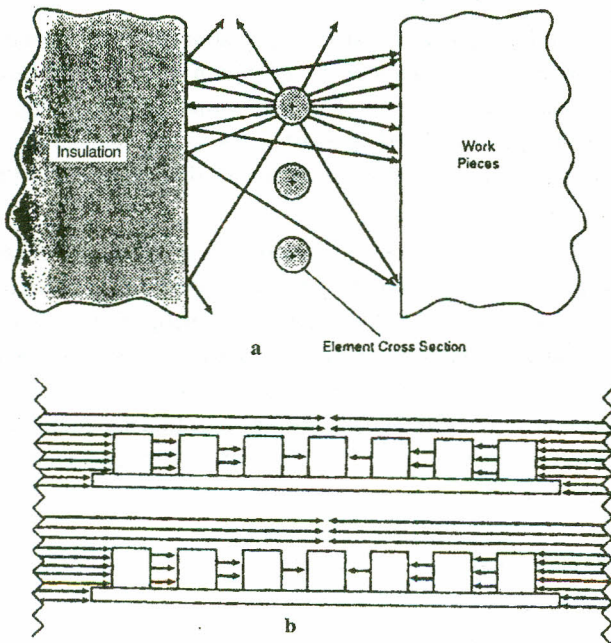

Fig 3. Showing the way heat is transferred to the workload., (a). Static load. (b). Distributed load.
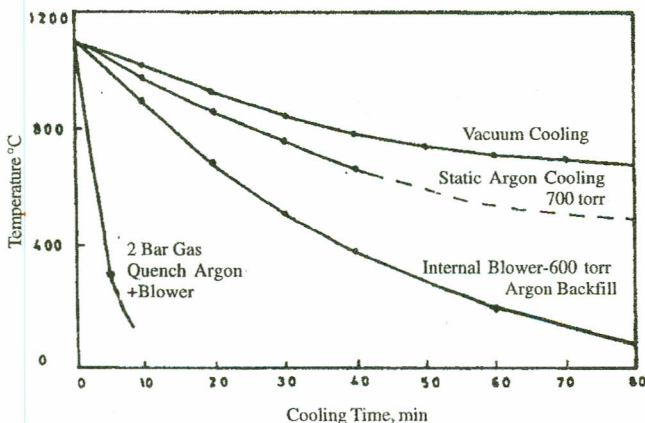

Fig 4. Showing the cooling characteristics of the workload in vacuum furnace.

QUENCHING UNDER VACUUM

Cooling the workload in a furnace under vacuum is a slow process. The heat on the load must pass by radiation through the heat insulation to cold chamber wall. Faster cooling is accomplished by recirculating the gas between the workload and the heat exchanger at a speed of a few hundred m^3 min^{-1}. A motorized blower installed inside the furnace chamber but outside the furnace hot-zone performs this forced convection. A cooling jet directed perpendicular to the work face has been shown to be more efficient than parallel flow along the surface. Cooling gas jets are arranged, therefore, to direct the gas against work. They are kept as close to the work as practically possible; to avoid diffusion in a large volume before impingement on the work.

A basic equation governing gas quenching is (Van Atta 1965).

$$\flat T = WC_p/AH_1 \; In \; T_1 - T_f/T_2 - T_f$$

Where, $\flat T$ is the time of cooling in minutes from T_2 to T_1, W = Weight of load in kgs., C_p = Specific heat of workload, $calkg^{-1} \, °C^{-1}$., H_1 = heat transfer coefficient, $calhr^{-1} \, m^{-2} \, °C^{-1}$., A= Surface area of workload, sq.m., T_f = Average fluid (gas) temperature, °C., T_1 = Initial load temperature, °C., T_2 = Final load temperature, °C.

Figure 4 is showing the cooling characteristics of the workload in vacuum furnace. When even more faster cooling rates are required, quenching in oil is best choice under vacuum. The quenching liquid is agitated vigorously by propeller and heat absorbed by the quenching medium is removed by heat exchanger similar to that used in many atmosphere heat treating furnaces. Figure 5 whows the vacuum furnaces with the added facility of oil quenching.

PREPARATION OF WORKLOAD

In preparation and handling of work piece for vacuum heat treating, several factors have to be considered which will influence the end result of the processing. Cleanliness of the job is very important. The pieces must be free of dirt, oil, grease, machining coolants and forming compounds before they are loaded into the furnace. Some lubricants and cutting oil containing sulphur compounds can adversely affect the material being treated. Inadequate cleaning can produce unsatisfactory surface finish. Contaminants with high vapour pressure such as drawing and stamping lubricants will be vapourized during heating causing unacceptable increase in pressure and loss of vacuum. The vapour will condense on the colder surface of the furnace, presenting a potential for gassing off during subsequent higher temperature cycle. Table 1 provides the methods commonly used for cleaning of work piece for vacuum heat treatment (Pritchard 1993).
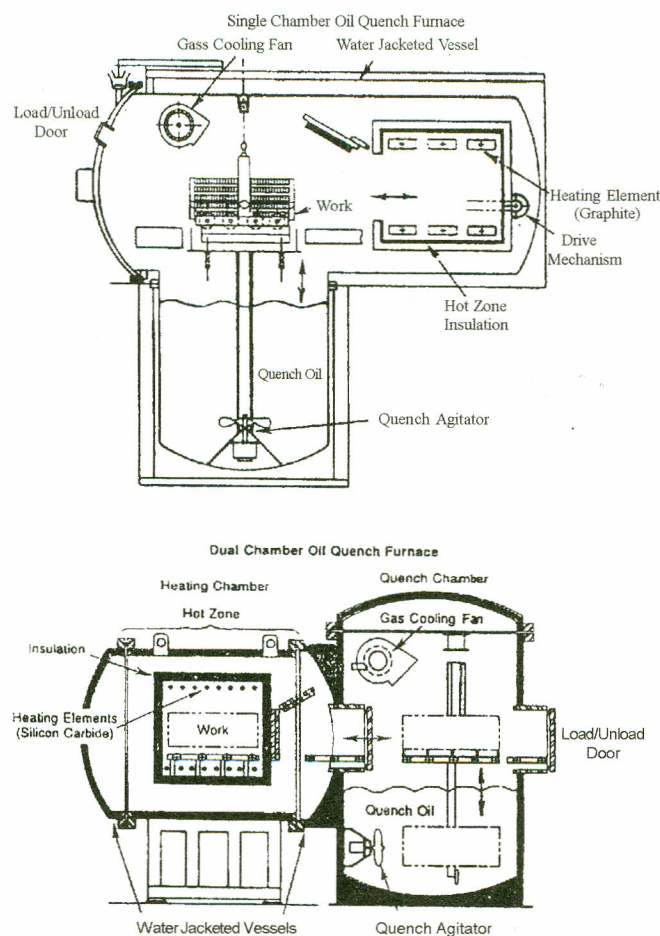
Fig 5. Section of the vacuum furnace with an added facility of oil quenching.

Table 1
Cleaning methods for vacuum heat treatment

Method	Bath Composition	Suitability for Removing	
		Mineral Oil Cutting Fluids	Water-Soluble Oils
Emulsion Cleaning	A mixture of insoluble hydrocarbons and water	Good	Good
Alkaline Cleaning	Water-base containing alkaline cleaners and other additives	Good	Good
Solvent Cleaning	-Mineral spirits -Alcohol -Acetone -Toluol -Chlorinated hydrocarbons	Good	Poor
Vapour Degassing	Chlorinated hydrocarbons -Methylchloride -Perchlorethylene -Trichlorethylene -Trichloroethane	Excellent	Poor

Note: Do not use chlorinated hydrocarbons for cleaning titanium and zirconium alloys.

A few processing cycles of some materials which are being heat treated in vacuum furnace are displayed in Fig. 6.

Summary

This technology is advancing rapidly and new material and engineering developments have allowed the industry to break away from conventional methods to more advanced methods of vacuum heat treatment. Vacuum is now a partner of the shop floor. The future of vacuum processing of heat treatment will be its integration into the manufacturing cell. The vacuum furnace inline, processing parts within the time constraints that allow the parts to be produced as needed. This means no inventory, and very short lead times. The industries will have to meet this challenge in the coming future.
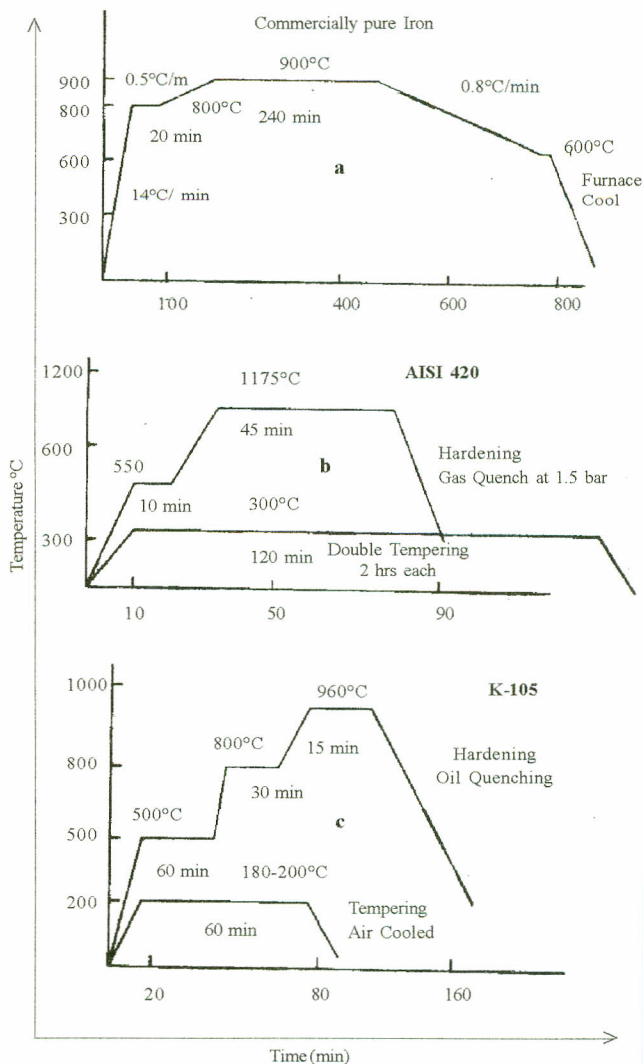

Fig 6. Processing cycle of various materials heat treated under vacuum., a. Commercially pure iron, vacuum annealing., b. AISI 420, gas quenching., c. K-105, oil quenching.

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