INTRODUCTION

For heat treatment of metal parts wide variety of furnaces are employed. Protective atmosphere furnaces like sealed quench furnace, vacuum furnaces, pit type retort, rotary retort furnaces and salt bath furnaces are used for heat treatment processes. In recent years fluidised bed furnaces are gaining popularity due to certain inherent advantages associated with them. Almost all the processes such as neutral hardening, carburising, carbonitriding, annealing, nitriding nitrocarburising etc. can be carried out with a single fluidised bed furnace installation. Added to this flexibility bed temperature uniformity, quick change over of atmospheres, fast rate of heat transfer and bed buoyancy results in predictable and consistent results in properties and dimensions after heat treatment.

THE PRINCIPLE

Fluidisation is the method of making a bed of small particles behave like a liquid. This is achieved by passing a stream of air or gas upward through the particle bed. As the velocity of the air/gas stream is raised, it meets with resistance due to the mass of the particles. However, at a critical point (where the frictional resistance equals the particle mass) (fig.1) the bed suddenly expands. Particles that were touching each other move apart and float on a cushion of air. The particles are now said to be “fluidised”.

Q-1
The velocity when this occurs is called minimum fluidisation velocity and it is a function of the particle diameter and its mass generally as per

$$U_{mf} d^2 p$$

an increase in velocity causes bubbles to form which displace the particles as they rise to the surface and cause turbulent particle movement. The fluidized bed now looks and behaves remarkably like a liquid. Light objects like wood float and dense objects like steel sink. The bed finds its own level and flows out if the container is punctured. The bed surface remains on a horizontal plane even if the container is titled.

CONSTRUCTION

The construction of the fluidised bed is shown in the Fig.2.

It contains a alloy retort with a specially designed diffuser assembly in its bottom. This retort is heated by heating elements and insulated by technical fibre. There are facilities to send different gas combinations into the retort. The retort contains fine alumina particles. The furnace has a lid and provision for the gases to be passed out.

HEAT TRANSFER

When heated the fluidised bed becomes an excellent media for heat transfer and consequently an excellent heat treating furnace. Its heat transfer rate is comparable with lead and salt baths and is very much faster than convention radiation furnaces (Fig.3).
This high rate of heat transfer is dependent on four main factors:

* the particle size
* The bed material
* The fluidising velocity, and
* the bed design

The lower the particle size, the higher is the heat transfer. Very small particles however generate undesirable electrostatic effects, get carried out with the components and get blown all around.

The nature of the bed material, in terms of its density is another factor that contributes, to the heat transfer capacity. The lighter the particle, the lower is the power and gas flow required for fluidisation. Also the heat transfer is higher. Beyond a certain point however, operational problems occur. The thermal conductivity and the specific heat of the particles do not have a significant effect on heat transfer.

**TEMPERATURE UNIFORMITY**

The turbulent motion of the particles in all parts of the container and the high gas-solid interface area causes extremely uniform temperature conditions in any part of the fluidised bed. As a matter of fact, fluidised beds are used for calibrating thermocouples.

These units exhibit a temperature uniformity of +/- 0.14 deg C fluidised bed heat treating furnaces average +/- 3 deg C, throughout the bed with greater uniformity in the actual work zone.
HEAT TREATING PROCESSES IN FLUIDISED BEDS

NEUTRAL HARDENING

Hardening, the most common heat treating process involves heating the metal up to its austenitic state and cooling rapidly so as to achieve the desired structure. The rate of heating to austenitising temperature is unimportant for most of metals. What is important is that the temperature is attained throughout the section, the time taken to do so and the temperature uniformity. The fluidised bed furnaces presents an excellent as well as a low cost method for austenitising. The heat transfer rate is extremely high and the temperature uniformity is at +/- 3 deg. C, and in addition, the bed is buoyant. These factors combine to produce a high throughput of distortion free components from a comparatively small fluidised bed furnace.

Using a fluidised bed for hardening is very similar to using a salt bath without the pollution hazard and danger presented by salt baths. Precleaning to remove oil or moisture is not required in fluidised beds. As in salt baths, partial, 'tip only' treatments can be performed. Unlike salt baths, the fluidised bed furnace can be switched off when not in use without solidification.

The fluidised bed can be operated at any temperature from ambient up to a possible 1200 deg C and hence the same furnace can be used to austenitise a variety of steels including tools steels.

Neutral hardening is a term that is used when no surface reaction such as oxidation, decarburisation etc. occurs to the components when being heated. This is achieved by fluidising with pure nitrogen or commercial nitrogen spiked with a small percentage of LPG. In special cases where nitrogen cannot be used argon is used. Major economies are gained by the ability to switch atmospheres
within seconds. When not actually processing loads, the fluidised bed furnace can be fluidised with air, with nitrogen only being used during actual processing. The furnace need not be "conditioned".

**TEMPERING**

Tempering involves reheating of previously hardened steel components so as to obtain specified mechanical properties, to release stresses caused by the thermal shock of quenching and to ensure dimensional stability.

The quality of tempered components is mainly influenced by temperature uniformity and in turn, the precise controllability of the furnace with respect to tempering time. The fluidised bed assures the required temperature uniformity and thus makes precise process control a simple matter. The same furnace can be used for tempering of various steels at tempering temperatures ranging from 150 deg C up to 600 deg. C.

**CARBURISING**

Carburising can be done in fluidised bed furnaces by employing a variety of atmospheres, all gases become fully reactive immediately upon entry into the bed and hence separate gas generators are not required.

This is done in mixtures of LPG/air, nitrogen/LPG or nitrogen/methanol (with LPG enrichment). When using LPG/air mixtures, carburising proceeds rapidly by using the boost/diffuse technique where the boost is done at a high carbon potential, which promotes rapid carbon transfer. Such high potentials cannot generally be maintained in conventional atmosphere furnace for fear of soot damage or in salt baths on grounds of safety. The desired surface carbon level is then obtained by diffusing either in a linear ratio or
in a neutral nitrogen atmosphere. This technique is especially useful for rapid attainment of deep case depths. Controlled potential carburising for finish machined components or where surface finish is critical is obtained in the nitrogen/methanol system where again the boost/diffuse technique can be gainfully employed. User experience has established both the predictability and the reproducibility of either method. Cycle times are generally about 40% quicker than standard endogas atmosphere furnaces (Fig.4).

The speed of case formation for shallow depths, up to 0.5mm, in salt baths is similar to fluidised bed furnaces but is significantly slower for deeper cases.

CARBONITRIDING

Carbonitriding is done, at slightly lower temperatures with the addition of ammonia to the carburising atmosphere.

NITROCARBURISING

This is a new development over nitriding processes and it should not be confused with carbonitriding process. Traditionally nitriding is carried out at 520 to 560 Deg C with the use of Ammonia and Nitrogen for 40 to 80 hours. Nitriding process forms complex nitrides in the surface essentially a mixture of epsilon and gamma prime nitrides.

This combination though hardened wear resistant has harmful brittleness and hence are removed by subsequent lapping operations. Costly special alloys are used for nitriding processes.

In the case of nitrocarburising process a small amount of carbon is ahead alongwith the Nitrogen. This ensures formation of epsilon nitrides in the surface which is hard and wear resistant and also less
brittle. This layer is acceptable for engineering applications and need not be removed by troublesome matching operations. This process gives all the advantages of nitriding but with a sharp reduction of processing time. Less expensive plain carbon steels could also be subjected to this process with advantage.

The mechanism of this process is explained below:

Fig. 5 shows an isothermal section of the Fe C N phase diagram. The nitrogen content in a nitrocarburised case is 7% and the carbon content is between 1-2%. This region represents a stable epsilon carbonitride phase. The component zone being non-metallic has high hardness, anti-seizure properties (galling and scuffing resistance) and a low coefficient of friction.

Nitrocarburising has traditionally been done in salt baths, earlier techniques were poisonous and polluting. Later versions (e.g. tufftriding drastically reduce (but not completely remove) this hazard.

Fluidised bed furnaces, with their extreme flexibility have contributed significantly to the nitrocarburising process and have enable many variations of the basic process to enhance various properties for diverse applications.

Fluidtherm Technology, has developed a family of new generation nitrocarburising processes which are described below:

**THE FERRINIDE PROCESS**

This is ferritic (as it is done below transformation temperature) nitrocarburising process as done in fluidtherm fluidised bed furnaces, the atmosphere used is a mixture of ammonia, nitrogen and a small amount of a carbon source gas.
A significant feature of the compound zone is its microporosity. Pores form both in salt and gas based processes, however, the quantum is much greater in salt bath processes. Whereas pores generally have a beneficial effect in terms of lubricant retaining ability, large pores cause cracks due to shear stresses. It is therefore sometimes necessary to remove the skin even in some salt bath nitrocarburised components as done in the case of classical nitriding. The formation and extent of porosity depends on the uncontrolled richness of the nitrogen content compared to the carbon content. It is a feature of the ferrinide process that the gas mixture can easily be modified to control the porosity to suit different applications.

The growth of the compound zone depends on time, temperature and the steel (or iron) composition. The rate of growth is the least in highly alloyed steel and increased in low alloy steels.

The hardness of the compound zone also depends largely on the alloying content with a higher hardness being exhibited by highly alloyed steels.

The compound zone is backed by a diffusion zone rich in nitrogen which increases its hardness. When the components, especially low alloy steels are cooled rapidly after nitrocarburising, the nitrogen is retained in supersaturated solid solution which contributes to a significant increase in the fatigue strength of the component by as much as 120% and also the yield strength of thin section components. This is due to the positive inherent compressive stresses due to nitrogen "locking" in solid solution. Slow cooling or reheating contributes to precipitation of nitrogen which reduces the fatigue/yield strength.
The hardness of the diffusion zone is lower than the compound zone and depends on the alloy content in positive correlation. The thickness of the diffusion zone increases with treatment time though the rate of growth is higher in low alloy steels than in higher alloy steels. The slowest growth is exhibited by high alloy steels.

THE FERRINOX PROCESS

Ferrinided components exhibit an enhanced degree of corrosion resistance (except stainless steels) depending on the density of the compound zone. This corrosion resistance can be increased significantly by an oxidation step as done in the ferrinox process. By introducing an oxygen releasing gas for a short period immediately after the nitrocarburising process, a thin surface layer of oxide generally 1-2 microns is formed. The increase in corrosion resistance is comparable to that obtained by electroplating. This oxidation is obtained in salt bath processes by quenching in a nitrate bath (e.g. ab 1) which is a necessary step to neutralise the cyanide drag out. As this bath is at around 350 deg.C, it "unlocks" the nitrogen in diffusion zone. Consequently the yield/fatigue strength of low alloy components processed in salt baths is less than what is obtained in the ferrinox process.

The major advantage in the ferrinox process is that the oxidation phase is carried out after nitrocarburising and before quenching and this allows enhanced corrosion resistance without a loss of strength.

For much enhanced corrosion resistance the ferrinox b process can be used. Here the components are dipped into a synthetic rust preventive which is absorbed into the micropores of the compound zone. The resulting corrosion resistance is extremely high. Around 1000 hours in the ASTM b 117 salt spray test, rivalling the performance of corrosion resisting superalloy.
THE AUSTINIDE PROCESS

This is primarily a process to replace carburising/cyaniding on certain components in some applications. Austinide process is an austenitic nitrocarburising process. As already explained in ferritic nitrocarburising which is done below 580 deg C there is no formation of austenite with the diffusion of carbon and nitrogen. But when the temperature exceeds 580 deg C there is partial transformation of the matrix to austenite due to the enrichment with carbon & nitrogen at the surface.

This process imparts:

- a compound layer, primarily of carbonitrides/higher hardness
- a back up layer of higher hardness
- improved core strength
- low distortion (compared to carburising/carbonitriding)
- improved indentation resistance.

A two phase case is obtained with the outermost layer consisting of epsilon nitrides similar to ferrinide upto 50 microns backed by a deeper carbon rich martensitic layer typically 0.15 mm and up to 1.5 mm (fig.10). Core strength can be increased by the formation of a duplex structure or left unchanged depending on the processing sequence and the time/temperature employed.

This process enables the use of cheaper medium carbon/manganese steels in place of more expensive alloy case hardening steels for certain applications. In the case of thin section sheet metal pressings and small diameter shaft like components, this process allows a significant reduction in the section thickness required for the same duty thus reducing raw material and fabrication costs considerably.
In addition to the beneficial properties imparted by ferrinide which briefly are:

- excellent wear resistance,
- improved fatigue strength,
- lubrication retention,
- galling/scuffing resistance, etc.

THE AUSTINOX PROCESS

Like ferrinox, components subjected to the austinide processes can be subjected to an oxidation phase as well as impregnation by rush preventive for much enhanced corrosion resistance equally and exceeding that provided by plating.

It is to be noted that by austinox process if you form effectively 4 different distinct less namely surface oxide layer, compound nitride layer. Martensite/banite layer, Nitrogen diffusion layer. Here each layer has a useful role to play for engineering applications. This constitutes fabrication of an ideal composite material using an innovative thermochemical process.
Fluidizing flow rate

Schematic diagram of the fluidization process follows the response of the particle bed as gas flow rate increases. The bubbling seen toward the end of the fluidized regime is responsible for advantageous heat transfer properties.

Fig. 1

CROSS SECTION OF A TYPICAL FLUIDISED BED FURNACE SHOWING

1. RETORT
2. THE PARTICLES CONTAINED THEREIN
3. THE GAS DISTRIBUTOR ASSEMBLY
4. THE LOADED WORK BASKET
5. ELECTRIC (OR)
6. GAS HEATERS
7. GAS PANEL

Fig. 2