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Description

This invention relates to a furnace configuration for high temperature processes which may require high thermal energies such as melting and/or chemical reaction and starts from GB-A-891263.

Many high temperature processes are not suited to a scale-up in production from laboratory bench to industrial scale because of their heat transfer requirements and in such cases scale up results in a marked deterioration in furnace performance.

Processes which are limited in their scale-up because of heat transfer requirements are generally those in which the charged material or products formed have a high thermal resistance, undergo endothermic reactions, one component of the reaction is a significantly volatile species at the temperature of the reaction. Upon scale-up, such heat transfer limited processes suffer from the problems of greatly increased reaction times, increased specific energy consumption or incomplete reaction leading to non-uniform product.

Examples of reactions which exhibit such properties are the carbothermic reduction of transition metal or rare earth oxides to transition metal or rare earth borides, carbides and nitrides production of silicon carbide and boron carbide, silicon smelting, remelting of oxidised metal fines, and ferroalloy production.

Furnaces usually used for production of transition metal borides, carbides and nitrides and similar refractory materials include arc furnaces, resistance furnaces, rotary furnaces, pusher furnaces, induction heated crucible or induction heated shaft furnaces.

Rotary kilns, and pusher furnaces suffer from a low occupancy of charge in the hot zone, so for a given scale, the surface area and heat losses are high. Rotary kilns which can operate under reducing conditions are expensive to engineer for high temperature operation and are limited in their ability to be scaled up.

Induction heated crucibles and shafts suffer from rapid loss of thermal efficiency and long reaction times as soon as the scale exceeds about 3 kg batch.

Various attempts have been made to improve the performance and scale-up of processes limited by heat transfer requirements.

A) Choosing a reactor design which reduces the heat transfer distance.

Methods to reduce the heat transfer distance include the use of multiple small reactors or crucibles in a pusher furnace, the use of a long slender reactor in which one or two dimensions are small, and tumbling the charge as in a rotary kiln. These attempts suffer from the disadvantages of increased surface area to volume ratio and hence high heat losses, and an increase in the size of the furnace hot zone with no corresponding increase in production capacity or efficiency.

B) Driving the heat transfer with high temperature source such as an arc or a plasma.

Arc and plasma furnaces have been designed to place a high intensity heat source at the core of the furnace, and temperatures in the zone of the arc at all stages of reaction are in excess of 2000 - 2500°C. Whilst high heat transfer rates can be achieved with the high arc temperature, any components which are volatile may consequentially be lost to the gas phase before they have an opportunity to react to completion. In addition, such furnaces are restricted in their ability to achieve reaction in a narrow temperature range which may be regarded as optimum from a thermodynamic/ processing point of view and arc furnaces usually produce a sintered product.

Use of three electrodes enables some reduction of the heat transfer distance, in the horizontal plane, but there is little scope to increase the reaction zone in the vertical direction as this greatly increases heat transfer distances.

Arc furnaces usually use for example, the starting materials $TiO_2/B_4C/C$ for synthesis of TiB_2 because the boron is present in a less volatile form than B_2O_3 at reaction temperature, and the bulk density of the charge is higher. The high temperature of the arc can then be used to thermally drive the endothermic reaction. The synthesis of B_4C also made in an arc furnace, however suffers from the same constraints and costs of B_2O_3 volatility.

B_2O_3 can be used in the arc furnace charge as a boron source but losses of B_2O_3 to the gas phase are high and consequently B_2O_3 is the major cost in raw materials.

C) Using a heat source which generates heat within the charge.

Microwave and resistance heating, and reaction synthesis techniques would at first glance, appear to be the ideal solution to heat transfer limited processes.

Microwave techniques can be used to achieve through heating and high temperatures. However, the containment of microwave energy, the measurement of temperature, the selection of microwave transparent refractories, the low efficiency of conversion of electrical energy to microwaves (50-70%), and the limitation of magnetron sizes to 10 - 40 KW each, all represent significant problems in engineering microwave based processes. A more fundamental limitation which may occur in a process which undergoes chemical reaction is the uneven power distribution between reactants and products which can lead to thermal runaway in the product.

Resistance techniques have traditionally been used in the Atcheson type furnace, but the technique suffers from hot spots and the cost of high current engineering.

The use of self-propagating high temperature synthesis techniques (SHS) has attracted a lot of attention for synthesis of refractory hard materials, because reactions are chosen so that the reaction is exothermic, instead of strongly endothermic. The method however transfers the problems and costs of endothermic processes to the synthesis of the reactants used. For example Ti and B metal powders can be used to synthesise TiB_2 but the costs of the powders are high. This is justified only when

> a) there is no other viable method,
> b) there are advantages in obtaining high value-added sintered or fused product, or
> c) convenience for small quantities.
>
> In another variation, the reductant for SHS may be Al or Mg powder, which in addition to the above mentioned limitations, leaves an oxide in the product for later separation.

The price of transition metal or rare earth borides on the world market is high because a furnace configuration which is capable of operating at a reasonable thermal efficiency on a large scale and produce quality product with low boron loss has not previously been used.

It is an object of the invention to provide a simple, durable high temperature furnace configuration which allows increased production with a high temperature process, especially those processes whose production is limited by heat transfer characteristics and/or the volatility of one or more of its components.

To achieve the objectives it has been found that by placing the heat source within the charge, the heat transfer distance is reduced. The heat source is therefore away from the insulation hot face and high heat losses are avoided, particularly when driving the reactors at higher power densities. In addition higher power can be used with given process constraints because heat flow is both inward and outward.

GB-A-1430382 describes an induction furnace for melting glass. The furnace is assembled by arranging, in a vessel containing the glass, electrically conductive susceptor elements of a material having no significant chemical effect on the glass and having an electrical conductivity higher than that of molten glass, the susceptor elements being positioned to define an electrical current path as a closed loop partly through the glass and partly through the susceptor elements. An alternating current is passed through a conductor coil around the vessel to induce an electric current in the current path through the glass and the susceptor elements.

GB-A-891263 describes a process and apparatus for the regulatable, continuous melting down of metal chips in an induction-heated crucible. The crucible contains one or more susceptor bodies, such as graphite bodies, arranged in the zone of the chips. In one embodiment, a single susceptor ring of pure graphite coaxial with the longitudinal axis of the vessel is immersed in the metal chips.

The present invention provides a high temperature furnace configuration for use in preparing products by heating a charge of reactants to a high temperature, said charge and/or products having a low thermal conductivity, said reaction having a high heat of reaction, said furnace configuration comprising: an outer shell; an electromagnetic induction means adjacent said outer shell; a thermal and electrical insulating layer for insulating said induction means from the charge; a charge receiving space within the insulating layer to receive said charge; and an induction susceptor ring positioned within a charge in said charge receiving space such that a cross-sectional centre of area of the susceptor ring in a radial direction is within the range of 30% to 90% of a radial distance from the centre or inner limit of the charge to the outer limit of the charge, characterised in that the induction susceptor ring comprises a coupling portion and a conducting portion extending radially from the coupling portion to conduct heat generated by the coupling portion into the charge, whereby a progressing reaction front for completely reacted product reaches the centre or inner limit of the charge before it reaches the outer surface of the charge.

The present invention also provides a method of controlling a furnace configuration having an outer shell, an electromagnetic induction means adjacent said shell, a thermal and electrical insulating layer insulating said induction means, a charge receiving space within the insulation layer, a charge positioned in said charge receiving space, and an induction susceptor ring positioned within said charge in said charge receiving space such that a cross-sectional centre of area of the susceptor ting in a radial direction is within the range of 30 to 90% of a radial distance from the inner limit of the charge to the outer limit of the charge (1), said method characterised in that it comprises the steps of:

> (i) heating said susceptor ring up to a predetermined temperature,
> (ii) increasing a temperature of the susceptor ring after a progressing zone front for completely reacted product has progressed away from a surface of said susceptor ring such that the temperature of the charge in the advance of the progressing zone front remains below said predetermined temperature, and
> (ii) continuing to increase the temperature of the susceptor ring while maintaining the temperature in advance of the progressing zone front below said predetermined temperature until a maximum temperature of the susceptor ring is reached or the progressing zone front reaches the outer limit of the charge.

The positioning of the susceptor means is important to the invention because if the susceptor means is placed closer to the center, fusion of product occurs at the core and the time for the processing zone front to reach the outside increases. In some cases, as a consequence of the high energy input for the processes suitable for the furnace configuration of the invention, a steady state may be achieved before the reaction zone front reaches the outside of the charge and so the yield of product is reduced.

If the susceptor means is placed closer to the outside, additional time is required for the reaction zone front to reach the core. During this time, heat losses are high and insulation damage occurs. The external limits of the charge are the internal surfaces of the furnace which are insulated and include the sides and ends of the furnace.

The furnace may be provided with spacers to maintain the position of the susceptor means within the furnace.

The body of the charge may be a solid cylindrical or annular cylindrical configuration. When the body of the charge is annular the inner limits of the charge becomes the inner surface of the furnace confining the annular charge. However, it is also possible that furnaces which have non-cylindrical external surfaces may also be used in accordance with the invention. In such cases the external surface would be an averaging of the distances from the core.

The electromagnetic induction means preferably comprises one or more heating coils operating at a medium frequency which may be water-cooled and have at least one layer of thermal and electrical insulation separating them from the charge.

The induction susceptor means preferably consists of at least one ring shaped susceptor of suitable material such as graphite.

The susceptor means is preferably shaped as a solid of revolution of its radial cross-sectional configuration.

For a susceptor of constant density, the center of area of a radial cross-section is the centroid of the radial cross-section.

The power rating of the susceptor relates to the power transfered per unit surface area of susceptor and is within the range of 2 to 200 kW/m^2 and preferably 30 to 60 kW/m^2.

If the susceptor means is a ring, it preferably is coaxial with the charge.

Preferably, the susceptor means comprises at least two susceptors positioned in the charge such that a reaction front progressing from the susceptor reaches the centre or inner limits of the charge or the progressing reaction front of an adjacent processing front prior to reaching the external limits of the charge. Preferably the progressing reaction front reaches the centre or inner limits of charge at about the same time as it reaches the progressing reaction front of an adjacent processing front.

During the reaction, the reaction zone front spreads in every direction from the surface of the susceptor means. As discussed earlier to avoid unnecessary heat losses and insulation damage, it is preferable that the processing is completed when the processing zone front reaches the external surface of the charge. To maximise yield and quality, when two or more susceptors are used, it is preferable that the susceptors are placed so that the reaction of reactants is complete when the processing zone front reaches the external surfaces of the charge.

Graphite is described as the preferred susceptor material, but many structural forms of carbon can be used as well as refractory hard material (RHM) and composites thereof.

A refractory hard material is a carbide, boride or nitride of a group comprising transitional and rare earth elements in groups IV, V and VI of the period table, boron and silicon.

The RHM must be able to act as a susceptor at or up to the reaction temperature and beyond.

As would be appreciated the requirements of the susceptor are that it is electrically conductive so that a current can be induced and thermal energy generated by resistive losses and the material is capable of withstanding the reaction temperature and chemical environment.

While the furnace configuration is particularly suited to a low thermally conducting charge and/or product and the charge can be in physical forms such as powder pellets, brequettes and granules.

The susceptor means further comprises a conducting portion extending radially from said coupling portion to conduct heat generated in said coupling portion into a charge within the charge receiving space.

As the susceptor represents the path of least thermal resistance, the conducting portion of the susceptor means allows heat to be more uniformly delivered to the reactants. This is particularly useful when the furnace for the reaction is scaled-up so that heat can be delivered to charge in the extremities of the furnace without increasing the temperature of the product nearer the susceptor beyond acceptable limits.

The configuration of the present invention changes the sequence of insulation/susceptor/charge typically used in an induction heated furnace to insulation/charge/susceptor/ charge. This enables the susceptor temperature to be raised to drive the heat transfer to the charge without also driving the heat losses to the walls. Also the charge actually provides additional thermal resistance to the losses to the surroundings. Energy efficiency is then better than external heating and is better than that achieved with through heating.

The positioning of the susceptor in the charge allows a more uniform transfer of heat to the charge, which enables a high temperature process to be scaled up to produce a high purity product at a high yield.

The configuration of the invention enables minimization of maximum heat transfer distance which enables maximization of reactor scale at a given maximum heat transfer distance.

The foregoing and other features objects advantages of the present invention will become more apparent from the following description of the preferred embodiments in which:

FIGURE 1 is a schematic cross-sectional elevation of a comparative furnace configuration where the charge is annular and the centre is hollow.

FIGURE 2 is a similar cross-sectional elevation of a second comparative furnace configuration with a solid cylinder of charge.

FIGURE 3 is a schematic view illustrating the progression of the processing zone from the susceptor.

FIGURE 4 are further schematic views illustrating the progression of the processing zone through the charge.

FIGURE 5(a) is a charge profile illustrating the progression of the processing front with time.

FIGURE 5(b) is a temperature profile of FIGURE 5 (a) showing heat being driven to the charge of a ring heated furnace,

FIGURE 6 is a schematic temperature profile of an externally heated furnace of the prior art,

FIGURE 7 is a schematic view illustrating various configurations of reactors for the same maximum heat transfer dimension "a",

FIGURE 8 is a graph illustrating the scale-up benefits of the invention,

FIGURES 9(a) - (d) are graphs illustrating the effect of radial placement of susceptor on reactor performance.

FIGURES 10(a) and (b) are graphs illustrating the effect of maximum heat transfer distance on scale-up, and

FIGURE 11 is a radial cross-section of alternative and comparative embodiments of the susceptor.

Referring to FIGURES 1 and 2, a furnace comprises a body of charge 1 within which are suspended one or more spaced graphite susceptor rings 2. When two or more susceptors are used, the susceptors are held in physical separation by supports (not shown) preferably of graphite. Thus as the reaction proceeds and the bulk density of the charge changes, the orientation of the susceptors in the furnace is maintained. The susceptors are heated by an electromagnetic induction coil 3 operating at a frequency of 50 -100,000 Hz. The susceptors and the coil are generally co-axial. One or more insulation layers 4 separate the charge from water cooling coils (not shown), the electromagnetic induction coils and the outer shell 6.

For a solid charge as in FIGURE 2 the susceptors are positioned so that the center of area of the radial cross-section is 30 - 90% of the distance from the centre to outer surface of the charge and preferably within the range of about 46 - 85% and most preferably 64 - 76%. The maximum heat transfer distance for the invention is therefore much less than the maximum heat transfer distance of 50% of the diameter for externally or core heated furnaces currently in use.

For an annular charge as in FIGURE 1 the susceptors are positioned so that the center of area of a radial cross-section is 30 - 90% of the distance from the inner surface of the charge to the outer surface of the charge and preferably within the range of about 46 - 85% and most preferably 64 - 76%. The space 5 in the centre of the annulus can be filled with insulating material or it can be left vacant to act as a chimney for gases produced in the reactor. The center of area of a radial cross-section of the susceptor is the area center of gravity of the radial cross-section.

To establish the optimum placement of the susceptor in the charge, a furnace of a required scale is run a number of times. From observations and measurements of the furnace product as it is removed from the furnace after each run, the extent of the processing zone is determined by chemical analysis or x-ray diffraction testing. FIGURE 3 illustrates the progression of a processing zone through a charge. In the case of a chemical reaction, the processing zone front is considered to be the point where the reaction is 98% complete or such extent of reaction as defines acceptable product grade.

The observations and measurements can be used to assess optimum total energy input to the furnace and in practice, the preferred endpoint is when a thin crust of incompletely reacted material is adjacent the external surfaces of the charge as the external surfaces of the charge represent the external heat loss surfaces. By leaving a thin crust, high yield of product is obtained with minimal damage to the refractories.

From these observations and measurements, the optimal placement of the susceptors in the charge is determined and the position of the processing front with time as shown in Figure 3 can be calculated. The yield, energy consumption, processing time and susceptor temperature can also be determined. A control model can then be developed to determine a time profile of power input for maximum productivity within the processing limits of charge volatility, susceptor temperature and product sintering and fusion.

The reactor performance upon scale-up, in general terms, is strongly dependent on power input and total energy input. Power input is maximised within the limits of charge stability, boron loss, susceptor temperature, product sintering and avoidance of product fusion. That power limit may be expressed in terms of uniaxial heat flow from a single planar source, which generates a reaction zone which progresses through the charge as shown in Figures 5(a) and 5(b).

Placement of the susceptor affects the performance of the reactor and the product obtained from it. The placement should be such that the heat transfer conditions in the direction of heat flow from the susceptor should be similar for all parts of the charge. By placing the susceptor in this way it is possible for the optimum power input profile to be used. With less than optimum placement, the limitations placed on power input for some parts of the charge, apply to all parts of the charge.

If the susceptor is placed outside the range of radial position of the invention, then the processing zone reaches the insulation before reaction is complete within the bulk of the charge. Insulation damage results and heat losses are high if more energy is supplied to complete the processing. If the reaction is stopped earlier, then incompletely processed material within the charge degrades the product.

A similar situation exists if the susceptors are placed too far apart.

If the susceptor is placed too far in, then poor electromagnetic coupling may result. With the susceptor too far in, the susceptor temperature can rise to the point where the product is sintered or fused. At the same time a large unprocessed layer may be left on the outside of the product, which can represent a substantial loss in yield. At large scale it is possible that the processing zone will reach steady state, because the temperature is below that at which significant reaction occurs and yield will not increase, no matter how long the reaction time.

If the susceptors are placed too close to each other then they will run hotter. The main disadvantage is that more susceptors are required and the volume of charge is reduced.

The benefits of the method of the present invention will now be demonstrated with reference to a high temperature process which is heat transfer limited. Such a process is the carbothermic reduction of titanium dioxide to titanium diboride.

The reaction is carried out according to the following formula at temperatures above 1448°C at atmospheric pressure.

$$TiO_2 + B_2O_3 + 5C \rightarrow TiB_2 + 5CO(g)$$

The B_2O_3 has a melting point of 450°C and a boiling point of 1860°C so is highly volatile at process temperature.

Both the product and the reactants have low thermal conductivity values and the chemical reaction is highly endothermic. Low thermal conductivity values are considered to be less than 20 W/m°C.

The effect of changing the radial placement of a cylindrical susceptor for a 300kg reactor, 1.0m diameter charge, 1.5m high with 150kW power input is shown in Figure 9(b) and 9(c). The susceptor temperature is controlled to a maximum of 2600°C while maintaining the temperature in advance of the processing zone front below the temperature constraint on the reactants which is about 1700°C.

With susceptor inside about 0.55 radius ratio, both the reaction time and energy consumption rise rapidly. Yield also drops because an outer layer of unreacted charge is left. The radius ratio at which this occurs is dependent on the amount of insulation used, and the conductivity of the charge and product. In a better insulated furnace the rise will be less steep, and the yield better.

With the susceptor outside the preferred radius ratio of about 0.7, the reaction time and energy consumption increase as heat losses through the insulation increase. The potential for damage to the insulation is greater as the energy input is increased to ensure complete reaction at the core.

A similar example is shown in FIGURE 9(a) and 9 (b), but in that case the power input is constant through to 95% reaction.

With a susceptor placed with a radius ratio of less than 0.6, fusion occurs at the susceptor. With the susceptor placed outside the 0.7 radius ratio, fusion also occurs around the susceptor. With the susceptor placed in the range 0.6 to 0.7 fusion does not occur. It should be noted that if power input is reduced, the range of positions, where fusion does not occur, increases. At lower power inputs, reaction times are longer and energy consumptions higher.

The optimum position for the susceptor placement is dependent on scale, heat losses, charge and product conductivities, arrangement in 3 dimensions, and power input in any particular application. It is considered that for all practical purposes, over a wide range of the above variables, the optimum position will lie in the range 0.3 to 0.9 times the distance from the center or inner limits of the charge.

The preferred range is 0.6 to 0.85 times the distance from the center or inner limits of the charge to the outer limits of the charge. This is based on experience with L, E, F, and M series coils, and on model results such as is shown in FIGURES 11(b) and 11(c). In that example the heat losses are relatively high, and in a better insulated furnace the optimum would move closer to 0.85.

The gap between the coil and the susceptor is important in determining the Q factor of the coil and load.

A working formula is that

KVAR/KW=1.4(GAP+0.5*PENETRATION DEPTHS)/0.5*
PENETRATION DEPTHS
wherein
Q = KVAR/KW = reactive power per kW input
GAP is distance from the coil to susceptor.

At 300Hz, a 200mm gap to a graphite susceptor will give a Q factor about 10. The choice of frequency, susceptor material, refractory and placement of the susceptor is crucial to the satisfactory operation of the furnace. Optimum placement which takes into account coupling requirements may place the susceptor further out than the optimum position based on heat transfer requirements alone.

In low thermal conductivity product or charge, the susceptor represents the path of least thermal resistance and so can be shaped so that it can conduct heat through the charge.

As would be appreciated by those skilled in the art it is desirable to provide as large a heat transfer area from the heat source as possible.

Therefore, it may be advantageous to shape the rings so that the cross-sectional configuration is elongated in the radial direction as shown in FIGURES 11 (b), (d) and (h).

A portion of the susceptor must be capable of coupling with the induction coils. This coupling position can be part of the conducting portion of the susceptor as in FIGURE 11(b) but to reduce the mass of susceptor in the furnace a configuration as in FIGURE 11 (d) and (h) is preferred. The susceptor is formed as a single article to maximise the heat transferred from the coupling portion to the conducting portion.

It is preferable that the total volume of susceptor material in the furnace is less than 20% of the total charge volume.

This limit is due to the additional thermal load of the susceptor on the process during both heating and cooling.

The susceptor also represents the path of least thermal resistance and so it can be shaped so that it can conduct heat through the charge. Therefore, it may be advantageous to shape the rings so that the cross-sectional configuration is elongated in the radial direction for example as in FIGURES 3(b) and 3(c).

As discussed earlier, the susceptor shape is formed as a solid of revolution of the radial cross-sectional area about the axis of the susceptor.

The benefits of the method of the present invention are shown in FIGURE 10 when scaling up the reactor.

For any furnace arrangement at a given scale, the maximum heat transfer distance can be determined. The time taken and the thermal gradient required to progress a processing zone through to that distance is dependent on the thermal conductivity of the charge and product the heat loss from the furnace and the allowable power input. Any scale-up involves a substantial increase in the maximum heat transfer distance unless it is performed in accordance with the invention.

The graph shows that for a ring heated furnace with the susceptors positioned in the optimum position the scale of the reactor increases as the maximum heat transfer distance increases.

A comparison between an externally heated furnace, a ring heated furnace and an extended ring furnace for the production of titanium diboride with a maximum heat transfer distance of 250 mm shows that the maximum furnace capacity is 30 kg, 540 kg and 4136 kg. Thus the enormous benefits of a ring heated furnace and the benefits of the extended ring in accordance with the invention are clearly demonstrated.

The benefits of the method of the present invention are further illustrated in FIGURES 6, 7 and 8 by comparing the size of the furnace which are possible for a given maximum heat transfer distance.

The furnaces shown in FIGURE 7 all have the same maximum heat transfer distance and from left to right represent a solid and annular externally heated furnace, a solid and annular ring heated furnace and an annular extended ring heated furnace.

As shown in FIGURE 8, the variation in configuration from a solid charge externally heated furnace to an extended ring heated furnace represents an increase in scale of over 100 times for the same maximum heat transfer distance.

As the susceptor is placed within the charge, the power input from the susceptor can be controlled to drive the temperature within the physical limits of charge and susceptor and the later stages of the processing on the physical limits of the insulation.

FIGURE 6 illustrates the temperature profile of an externally heated reactor constrained by the thermal limit of the insulation (1700°C). The power to the furnace actually has to be decreased with time to remain within the constraints.

FIGURE 5(b) is a temperature profile further demonstrating the benefits of the invention. The susceptor is heated up to the thermal constraints of the charge (1700°C) until the processing zone front moves from the susceptor surface. At this time the reaction at the susceptor surface is at least 98% complete. FIGURE 5(a) shows how the processing front progresses through the charge when a temperature profile such as shown in FIGURE 5(b) is employed.

As the processing front progresses through the charge as shown in FIGURE 4, the temperature of the susceptor is increased so that the temperature of the charge in advance of the front is below the thermal constraints of the charge.

The temperature of the susceptor can thus be taken up to the temperature limit of a graphite susceptor (about 2600°C), allowing heat to be driven to the charge resulting in faster processing times and higher yield. The processing is stopped when the processing front reaches the outer surfaces of the charge to minimise insulation damage.

The ring heating shown in FIGURE 5 is conducted at constant power input enabling greater ease of control.

Although the invention has been described in respect of one specific TiB_2 reaction route, other TiB_2 production process which are strongly endothermic and which rely on the reduction of oxides either by carbon or mixtures of carbon with Mg, Al, B or Ti may be equally improved by the use of this reactor.

Other high temperature reactions which are highly endothermic such as silicon smelting and ferroalloy production may also derive benefit from being carried out in the furnace configuration of the invention.

The invention is also suitable for the production of other ceramic materials such as silicon carbide, boroncarbide, titanium carbide and various other carbides, borides and nitrides.

Claims

1. A high temperature furnace configuration for use in preparing products by heating a charge (1) of reactants to a high temperature, said charge (1) and/or products having a low thermal conductivity, said reaction having a high heat of reaction, said furnace configuration comprising:

   an outer shell (6);
   an electromagnetic induction means (3) adjacent said outer shell;
   a thermal and electrical insulating layer (4) for insulating said induction means (3) from the charge (1);
   a charge receiving space within the insulating layer to receive said charge; and
   an induction susceptor ring (2) positioned within a charge in said charge receiving space such that a cross-sectional centre of area of the susceptor ring (2) in a radial direction is within the range of 30% to 90% of a radial distance from the centre or inner limit (5) of the charge to the outer limit of the charge, characterised in that the induction susceptor ring (2) comprises a coupling portion (10) and a conducting portion (11) extending radially from said coupling portion (10) to conduct heat generated in said coupling portion (10) into the charge (1), whereby a progressing reaction front for completely reacted product reaches the centre or inner limit of the charge before it reaches the outer surface of the charge.

2. A high temperature furnace configuration according to claim 1, comprising at least two induction susceptor rings, each positioned in the charge such that a progressing reaction zone front for completely reacted product emanating away from one of the susceptor rings reaches the inner limit (5) of the charge (1) or a progressing reaction zone front for completely reacted product emanating away from an adjacent susceptor ring prior to reaching the outer limit of the charge (10.

3. A high temperature furnace configuration according to claim 1 or 2, wherein the cross-sectional centre of area of the susceptor ring (2) in a radial direction is within the range of 60% to 85% of the radial distance from the inner limit (5) of the charge to the outer limit of the charge.

4. A high temperature furnace configuration according to claim 1, 2 or 3, wherein the induction susceptor ring (2) is formed as a solid of revolution of its radial cross-sectional configuration.

5. A high temperature furnace configuration according to any preceding claim, wherein the conducting portion (11) of said susceptor ring (2) has a higher thermal conductivity than the charge (1) or product.

6. A high temperature furnace configuration according to any preceding claim, wherein the conducting portion (11) extends radially inwardly from said coupling portion (10).

7. A high temperature furnace configuration according to any preceding claim, wherein the conducting portion (11) and coupling portion (10) of the susceptor ring (2) are formed as a single component.

8. A high temperature furnace configuration according to any preceding claim, wherein the induction susceptor ring or rings occupy a total volume of less than 20% of the total volume of the charge.

9. A high temperature furnace configuration according to any preceding claim, wherein the charge receiving space is annular shaped.

10. A high temperature furnace configuration according to claim 9, wherein the space in the centre of the annular charge receiving space is left vacant to act as an exit means to allow gases produced during operation of said furnace configuration to escape from said furnace configuration.

11. A method of controlling a furnace configuration having an outer shell (6), an electromagnetic induction means (3) adjacent said shell (6), a thermal and electrical insulating layer (4) insulating said induction means (3), a charge receiving space within the insulating layer, a charge (1) positioned in said charge receiving space, and an induction susceptor ring (2) positioned within said charge in said charge receiving space such that a cross-sectional centre of area of the susceptor ring in a radial direction is within the range of 30 to 90% of a radial distance from the inner limit (5) of the charge (1) to the outer limit of the charge (1), said method characterised in that it comprises the steps of:

    (i) heating said susceptor ring (2) up to a predetermined temperature,
    (ii) increasing a temperature of the susceptor ring (2) after a progressing zone front for completely reacted product has progressed away from a surface of said susceptor ring (2) such that the temperature of the charge (1) in advance of the progressing zone front remains below said predetermined temperature, and
    (ii) continuing to increase the temperature of the susceptor ring (2) while maintaining the temperature in advance of the progressing zone front below said predetermined tempera-

ture until a maximum temperature of the susceptor ring (2) is reached or the progressing zone front reaches the outer limit of the charge (1).

12. A method according to claim 11, wherein the susceptor ring (2) is positioned such that the progressing zone front reaches the inner limit (5) of the charge (1) prior to reaching the outer limit of the charge (1).

13. A method according to claim 11 or 12, wherein the induction susceptor ring (2) is formed as a solid of revolution of its radial cross-sectional configuration.

14. A method according to claim 11, 12 or 13, wherein the cross-section of area of the susceptor ring (2) in a radial direction is positioned within the range of 60 - 85% of the radial distance from the inner limit (5) of the charge to the outer limit of the charge (1).

15. A method according to any of claims 11 to 14, wherein said susceptor ring (2) comprises an inductive coupling portion (10) and a conducting portion (11) extending radially from said coupling portion (10) to conduct heat generated in said coupling portion (10) into the charge (1).

16. A method according to any of claims 11 to 15, wherein the reaction product is a transition metal boride.

Patentansprüche

1. Hochtemperturofen-Anordnung zur Verwendung für die Herstellung von Produkten durch Aufheizen einer Charge (1) von Reaktionspartnern auf eine hohe Temperatur, wobei die Charge (1) und/oder die Produkte eine geringe thermische Leitfähigkeit aufweisen, die Reaktion eine hohe Reaktionstemperatur besitzt, und die Ofen-Anordnung aufweist:

- eine Außenwandung (6),
- angrenzend zur Außenwandung ein elektromagnetisches Induktionsmittel (3),
- eine thermische und elektrische Isolationsschicht (4) zur Isolierung der Induktionsmittel (3) von der Charge (1),
- einen Chargenaufnahmeraum innerhalb der Isolationsschicht zur Aufnahme der Charge, und
- einen induktiven Suszeptorring (2), der innerhalb einer Charge im Chargenaufnahmeraum so angeordnet ist, daß die Mitte eines Querschnitts durch den Bereich des Suszeptorrings (2) in einer radialen Richtung innerhalb des Bereichs von 30% bis 90% der radialen Entfernung von der Mitte oder des inneren Randes (5) der Charge zum äußeren Rand der Charge liegt,

dadurch gekennzeichnet, daß
der induktive Suszeptorring (2) einen Kupplungsbereich (10) und einen sich radial vom Kupplungsbereich (10) erstreckenden Leitungsbereich (11) zur Leitung der durch den Kupplungsbereich (10) erzeugten Wärme in die Charge (1) aufweist, wobei eine fortschreitende Reaktionsfront von vollständig reagiertem Produkt die Mitte oder den inneren Rand der Charge erreicht, bevor sie die äußere Oberfläche der Charge erreicht.

2. Hochtemperturofen-Anordnung nach Anspruch 1, dadurch gekennzeichnet, daß sie zumindest zwei induktive Suszeptorringe aufweist, von denen jeder so in der Charge angeordnet ist, daß eine von einem der Suszeptorringe ausgehende fortschreitende Reaktionsfront von vollständig reagiertem Produkt oder eine von einem angrenzenden Suszeptorring ausgehende fortschreitende Reaktionsfront von vollständig reagiertem Produkt den inneren Rand (5) der Charge (1) eher erreicht, als die äußere Grenze der Charge (1) erreicht wird.

3. Hochtemperturofen-Anordnung nach Anspruch 1 oder 2, dadurch gekennzeichnet, daß die Mitte eines Querschnitts durch den Bereich des Suszeptorrings (2) in einer radialen Richtung innerhalb des Bereichs von 60% bis 85% der radialen Entfernung von der Mitte oder des inneren Randes (5) der Charge zum äußeren Rand der Charge liegt.

4. Hochtemperturofen-Anordnung nach Anspruch 1, 2 oder 3, dadurch gekennzeichnet, daß der induktive Suszeptorring (2) als Körper geformt ist, der durch Rotation seiner radialen Querschnittsform bestimmt ist.

5. Hochtemperturofen-Anordnung nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß der Leitungsbereich (11) des Suszeptorrings (2) eine höhere thermische Leitfähigkeit aufweist als die Charge (1) oder das Produkt.

6. Hochtemperturofen-Anordnung nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß sich der Leitungsbereich (11) radial nach innen vom Kupplungsbereich (10) ausgehend erstreckt.

7. Hochtemperturofen-Anordnung nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß der Leitungsbereich (11) und der Kupplungsbereich (10) des Suszeptorrings als einstückiges Bauteil ausgebildet sind.

8. Hochtemperturofen-Anordnung nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß der induktive Syszeptorring oder die -ringe ein Gesamtvolumen von weniger als 20% des Gesamtvolumens der Charge einnehmen.

9. Hochtemperturofen-Anordnung nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß der Chargenaufnahmeraum kreisförmig ausgebildet ist.

10. Hochtemperturofen-Anordnung nach Anspruch 9, dadurch gekennzeichnet, daß der Raum in der Mitte des kreisförmigen Chargenaufnahmeraums zur Bildung eines Auslasses unbelegt ist, um Gasen, die während des Betriebs der Ofen-Anordnung entstehen, das Entweichen von der Ofen-Anordnung zu ermöglichen.

11. Verfahren zur Steuerung einer Ofen-Anordnung, mit einer Außenwandung (6), angrenzend zu der Außenwandung einem elektromagnetischen Induktionsmittel (3), einer thermischen und elektrischen Isolationsschicht zur Isolierung der Induktionsmittel, einem Chargenaufnahmeraum innerhalb der Isolationsschicht, einer in dem Chargenaufnahmeraum positionierten Charge (1), und einem induktiven Suszeptorring (2), der innerhalb einer Charge in dem Chargenaufnahmeraum so angeordnet ist, daß die Mitte eines Querschnitts durch den Bereich des Suszeptorrings in einer radialen Richtung innerhalb des Bereichs von 30% bis 90% der radialen Entfernung vom inneren Rand (5) der Charge (1) zum äußeren Rand der Charge (1) liegt, wobei das Verfahren dadurch gekennzeichnet ist, daß es die folgenden Schritte aufweist:

(i) Erhitzen des induktiven Suszeptorrings (2) auf eine vorbestimmte Temperatur,
(ii) Erhöhen der Temperatur des induktiven Suszeptorrings nachdem eine fortschreitende Front eine Zone vollständig reagierten Produkts sich von der Oberfläche des induktiven Suszeptorrings (2) fortbewegt hat, so daß die Temperatur der Charge (1) vor der Front der fortschreitenden Zone unterhalb der vorbestimmten Temperatur bleibt, und
(iii) Beibehalten der Temperaturerhöhung des induktiven Suszeptorrings (2), bei Halten der Temperatur vor der Front der fortschreitenden Zone unterhalb der vorbestimmten Temperatur, bis eine Maximaltemperatur des induktiven Suszeptorrings (2) erreicht ist, oder die Front der fortschreitenden Zone den äußeren Rand der Charge (1) erreicht hat.

12. Verfahren nach Anspruch 11, dadurch gekennzeichnet, daß der Suszeptorring (2) so positioniert ist, daß die Front der fortschreitenden Zone den inneren Rand (5) der Charge (1) erreicht, bevor der äußere Rand der Charge (1) erreicht wird.

13. Verfahren nach Anspruch 11 oder 12, dadurch gekennzeichnet, daß der induktive Suszeptorring (2) als Körper geformt ist, der durch Rotation seiner radialen Querschnittsform bestimmt ist.

14. Verfahren nach Anspruch 11, 12 oder 13, dadurch gekennzeichnet, daß der Querschnitt durch den Bereich des Suszeptorrings (2) in einer radialen Richtung innerhalb des Bereichs von 60% bis 85% der radialen Entfernung des inneren Randes (5) der Charge zum äußeren Rand der Charge (1) angeordnet ist.

15. Verfahren nach einem der Ansprüche 11 bis 14, dadurch gekennzeichnet, daß der Suszeptorring (2) einen induktiven Kupplungsbereich (10) und einen sich radial vom Kupplungsbereich (10) erstreckenden Leitungsbereich (11) zur Leitung der durch den Kupplungsbereich (10) erzeugten Wärme in die Charge (1) aufweist.

16. Verfahren nach einem der Ansprüche 11 bis 15, dadurch gekennzeichnet, daß das Reaktionsprodukt ein Übergangsmetall-Borid ist.

Revendications

1. Configuration de four à haute température destinée à être utilisée pour la préparation de produits par chauffage à une température élevée d'une charge (1) de réactants, ladite charge (1) et / ou lesdits produits ayant une conductivité thermique faible, et la réaction dégageant une chaleur importante, ladite configuration de four comportant:

une enceinte externe (6);
un moyen d'induction électromagnétique (3) adjacent à ladite enceinte externe;
une couche thermiquement et électriquement isolante (4) destinée à isoler ledit moyen d'induction (3) de la charge (1);
un espace récepteur de charge, à l'intérieur de la couche isolante, pour recevoir ladite charge; et
un anneau de susceptance inductive (2) positionné à l'intérieur d'une charge dans ledit espace récepteur de charge de telle façon que le centre de la section de l'anneau de susceptance (2) dans le sens radial se trouve entre 30% et 90% de la distance radiale entre le centre ou limite interne (5) de la charge et la limite externe de la charge (1), caractérisée en ce que l'anneau de susceptance inductive (2) comporte

une partie de couplage (10), ainsi qu'une partie de conduction (11) s'étendant radialement à partir de ladite partie de couplage (10) pour conduire jusqu'à la charge (1) la chaleur générée dans ladite partie de couplage (10), ce qui permet à un front progressif de réaction aboutissant à une réaction complète du produit d'atteindre le centre ou limite interne de la charge avant qu'il n'atteigne la surface externe de la charge.

2. Configuration de four à haute température selon la revendication 1, comportant au moins deux anneaux de susceptance inductive, chacun étant positionné dans la charge de façon à ce qu'un front progressif de réaction aboutissant à une réaction complète du produit déclenchée par un des anneaux de susceptance atteigne le centre ou limite interne (5) de la charge (1) ou un front progressif de réaction aboutissant à une réaction complète du produit déclenchée par l'anneau de susceptance adjacent avant d'atteindre la limite externe de la charge (1).

3. Configuration de four à haute température selon la revendication 1 ou 2, dans laquelle le centre de la section de l'anneau de susceptance (2) dans le sens radial se trouve entre 60% et 85% de la distance radiale entre le centre ou limite interne (5) de la charge et la limite externe de la charge (1).

4. Configuration de four à haute température selon la revendication 1, 2 ou 3, dans laquelle l'anneau de susceptance inductive (2) est constitué d'un solide de révolution dont la coupe a sa configuration radiale.

5. Configuration de four à haute température selon l'une quelconque des revendications précédentes, dans laquelle la partie de conduction (11) de l'anneau de susceptance (2) a une conductivité thermique supérieure à celle de la charge (1) ou du produit.

6. Configuration de four à haute température selon l'une quelconque des revendications précédentes, dans laquelle la partie de conduction (11) s'étend radialement vers l'intérieur à partir de la partie de couplage (10).

7. Configuration de four à haute température selon l'une quelconque des revendications précédentes, dans laquelle la partie de conduction (11) et la partie de couplage (10) de l'anneau de susceptance (2) constituent un composant unique.

8. Configuration de four à haute température selon l'une quelconque des revendications précédentes, dans laquelle le ou les anneau(x) de susceptance inductive occupe(nt) un volume total inférieur à 20% du volume total de la charge.

9. Configuration de four à haute température selon l'une quelconque des revendications précédentes, dans laquelle l'espace récepteur de charge est de forme annulaire.

10. Configuration de four à haute température selon la revendication 9, dans laquelle l'espace qui est au centre de l'espace récepteur de charge annulaire est laissé vide, pour servir de moyen d'évacuation permettant aux gaz produits pendant le fonctionnement de ladite configuration de four de s'en échapper.

11. Procédé de commande d'une configuration de four à haute température comportant une enceinte externe (6), un moyen d'induction électromagnétique (3) adjacent à ladite enceinte externe (6), une couche thermiquement et électriquement isolante (4) destinée à isoler ledit moyen d'induction (3), un espace récepteur de charge, à l'intérieur de la couche isolante, une charge (1) placée dans ledit espace récepteur de charge, et un anneau de susceptance inductive (2) positionné à l'intérieur d'une charge dans ledit espace récepteur de charge de telle façon que le centre de la section de l'anneau de susceptance dans le sens radial se trouve entre 30% et 90% de la distance radiale entre la limite interne (5) de la charge (1) et la limite externe de la charge (1), ledit procédé étant caractérisé en ce qu'il comporte les étapes:

(i) de chauffage de l'anneau de susceptance (2) jusqu'à une température prédéterminée,
(ii) d'élévation de la température de l'anneau de susceptance (2) après qu'un front progressif de réaction aboutissant à un réaction complète du produit a progressé à partir de la surface dudit anneau de susceptance (2) de telle sorte que la température de la charge (1) en avant du front progressif reste inférieure à ladite température prédéterminée, et
(iii) de poursuite de l'élévation de température de l'anneau de susceptance (2) tout en maintenant la température en avant du front progressif inférieure à ladite température prédéterminée, jusqu'à ce que la température maximale de l'anneau de susceptance (2) soit atteinte ou que le front progressif atteigne la limite externe de la charge (1).

12. Procédé selon la revendication 11, dans lequel l'anneau de susceptance (2) est positionné de telle façon que le front progressif atteigne la limite interne (5) de la charge (1) avant d'atteindre la limite externe de la charge (1).

13. Procédé selon la revendication 11 ou 12, dans lequel l'anneau de susceptance inductive (2) est constitué d'un solide de révolution dont la coupe a sa configuration radiale.

14. Procédé selon la revendication 11, 12 ou 13, dans lequel le centre de la section de l'anneau de susceptance (2) dans le sens radial se trouve entre 60% et 85% de la distance radiale entre la limite interne (5) de la charge et la limite externe de la charge (1).

15. Procédé selon l'une quelconque des revendications 11 à 14, dans lequel l'anneau de susceptance (2) comporte une partie de couplage inductif (10), ainsi qu'une partie de conduction (11) s'étendant radialement à partir de ladite partie de couplage (10) pour conduire jusqu'à la charge (1) la chaleur générée dans ladite partie de couplage (10).

16. Procédé selon l'une des revendications 11 à 15, dans lequel le produit de réaction est un borure de métal de transition.

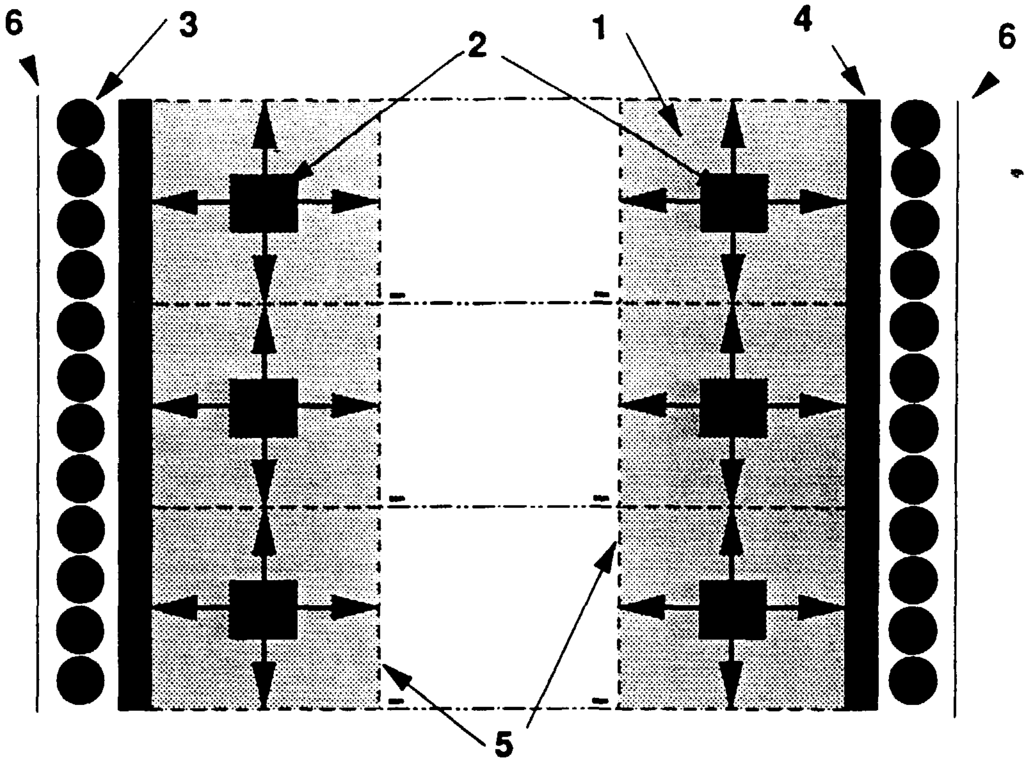

Figure 1

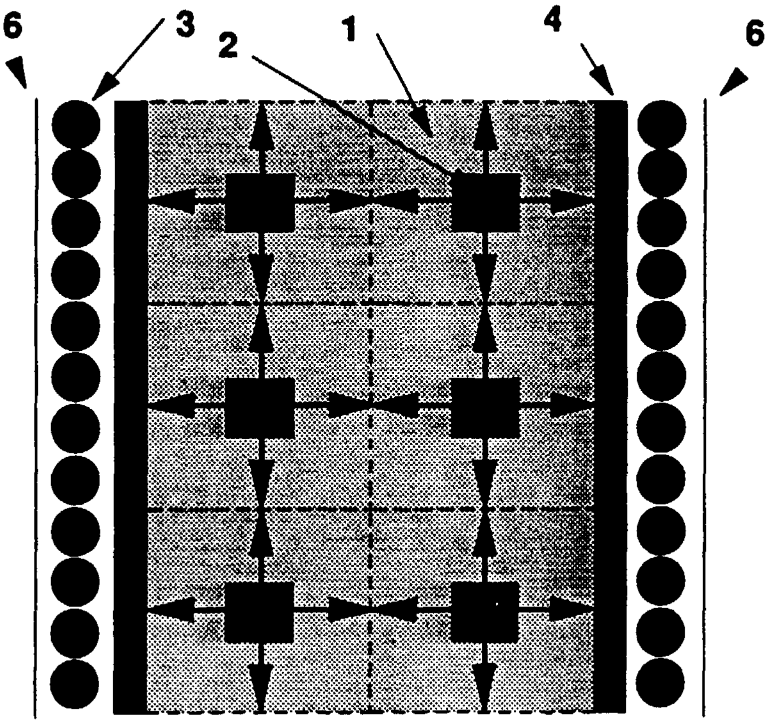

Figure 2

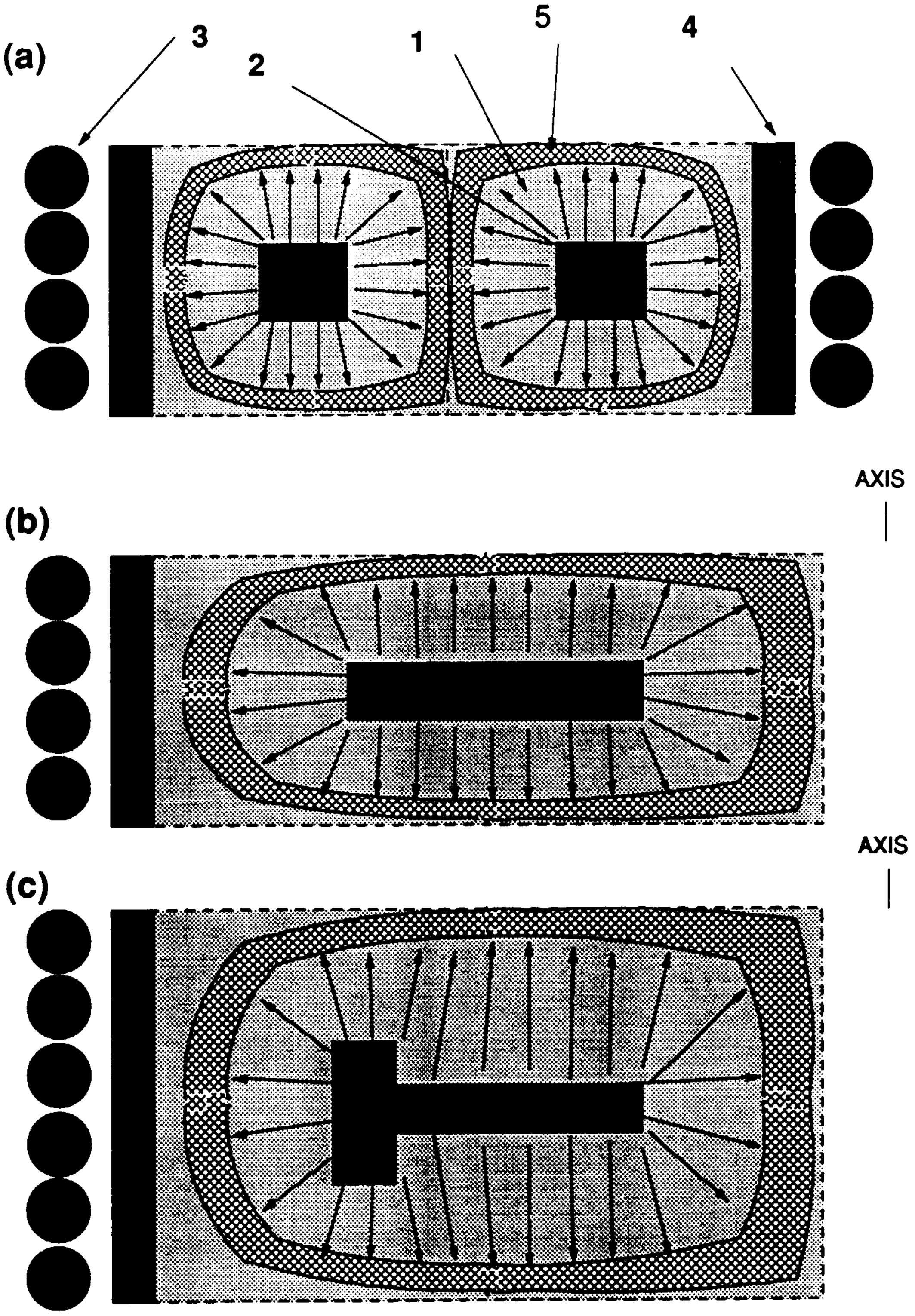

Figure 3

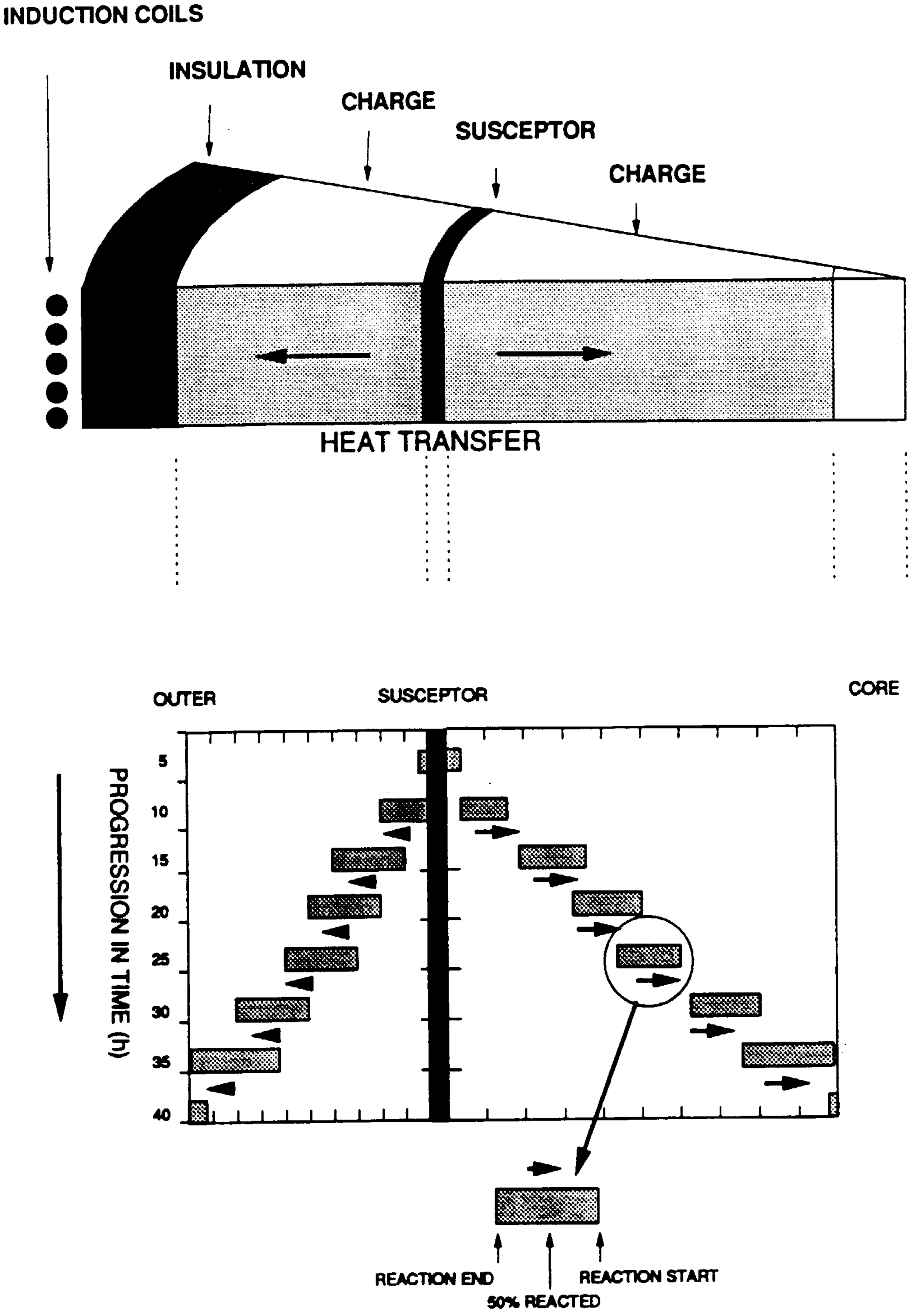
INDUCTION COILS
INSULATION
CHARGE
SUSCEPTOR
CHARGE
HEAT TRANSFER
OUTER
SUSCEPTOR
CORE
5
10
15
20
25
30
35
40
PROGRESSION IN TIME (h)
REACTION END
50% REACTED
REACTION START

Figure 4

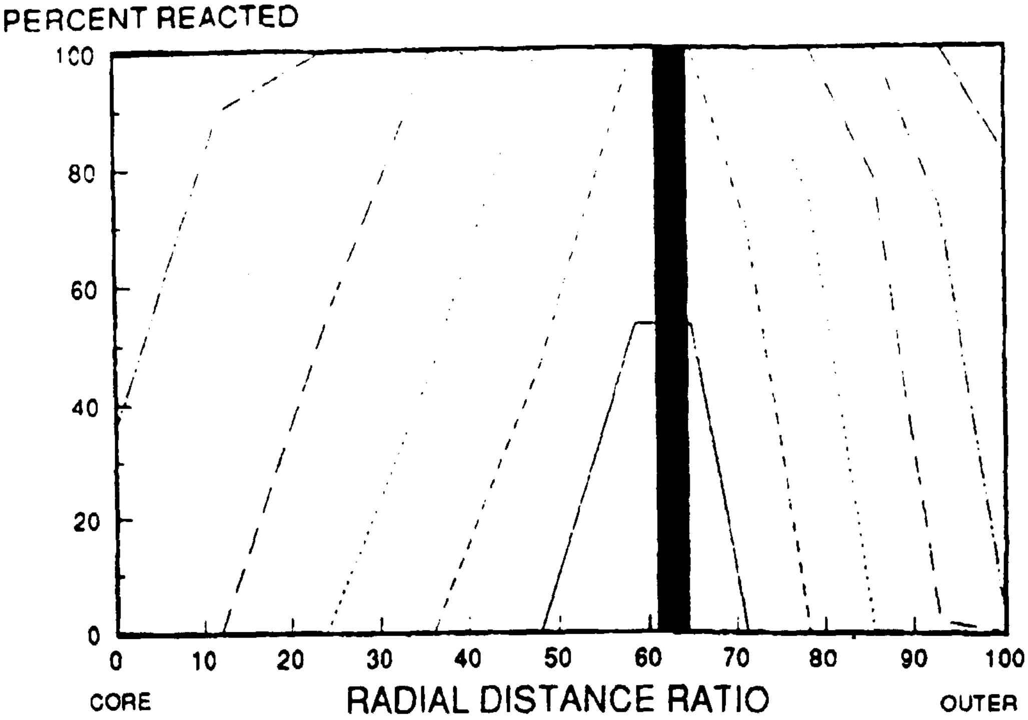

Figure 5a

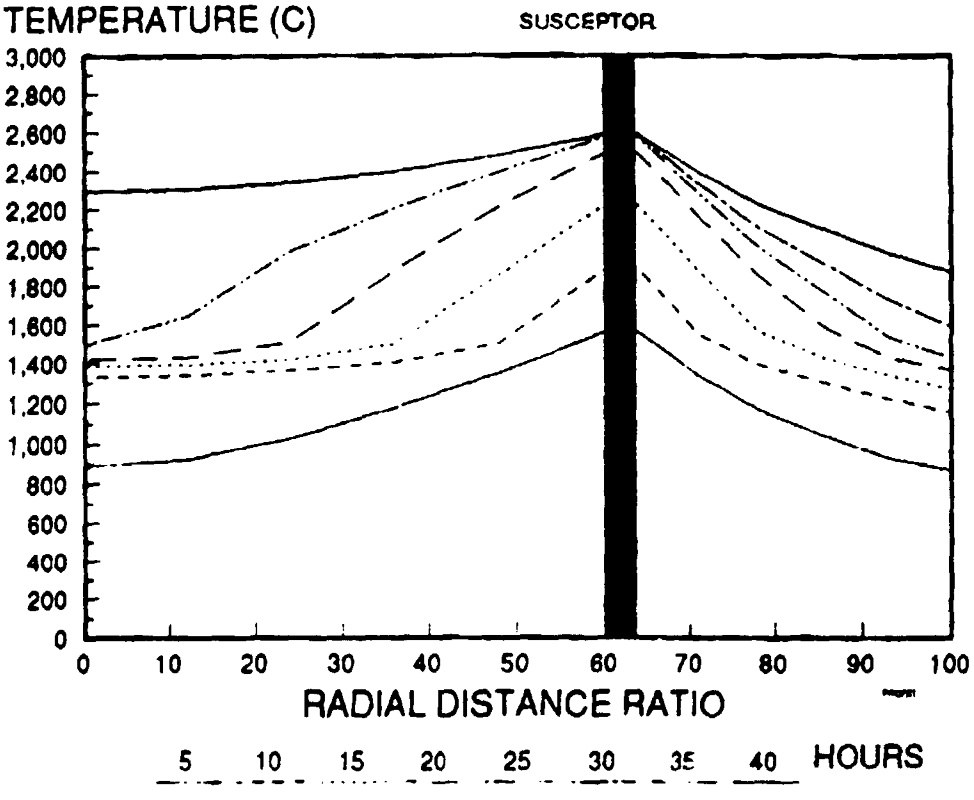

Figure 5b

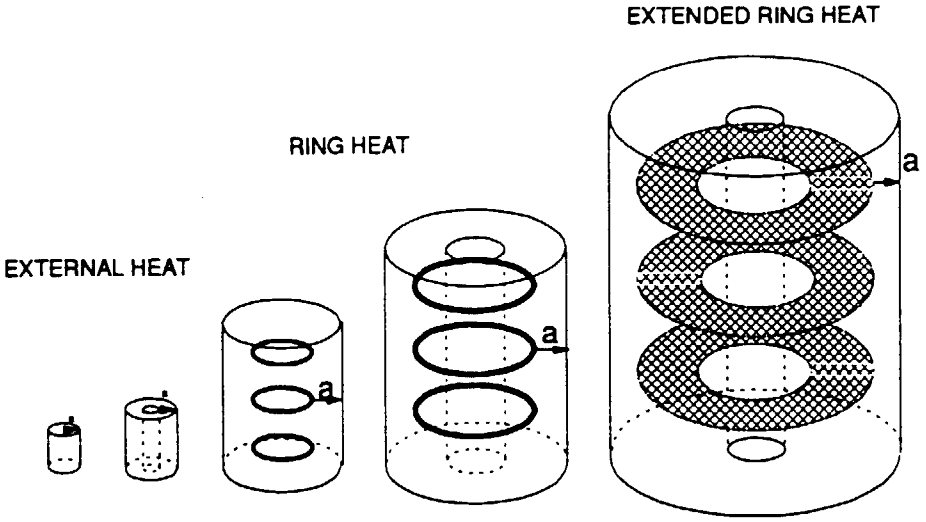

Figure 7

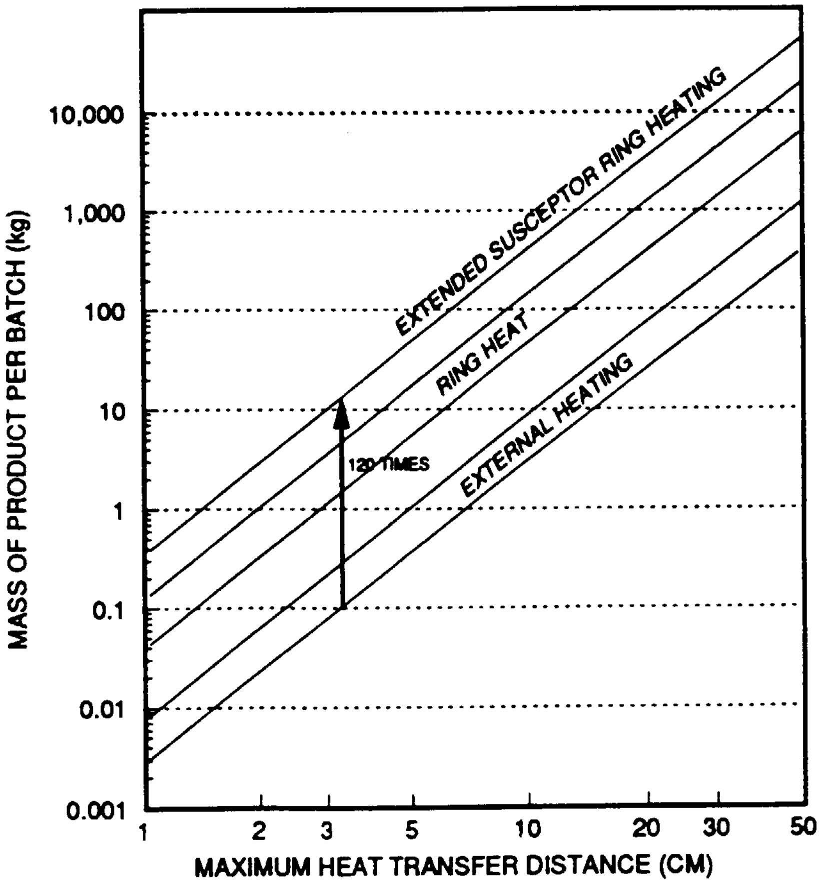

Figure 8

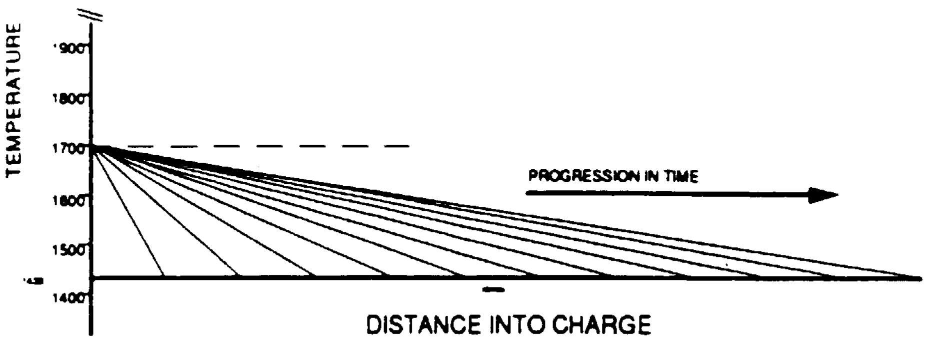

Figure 6

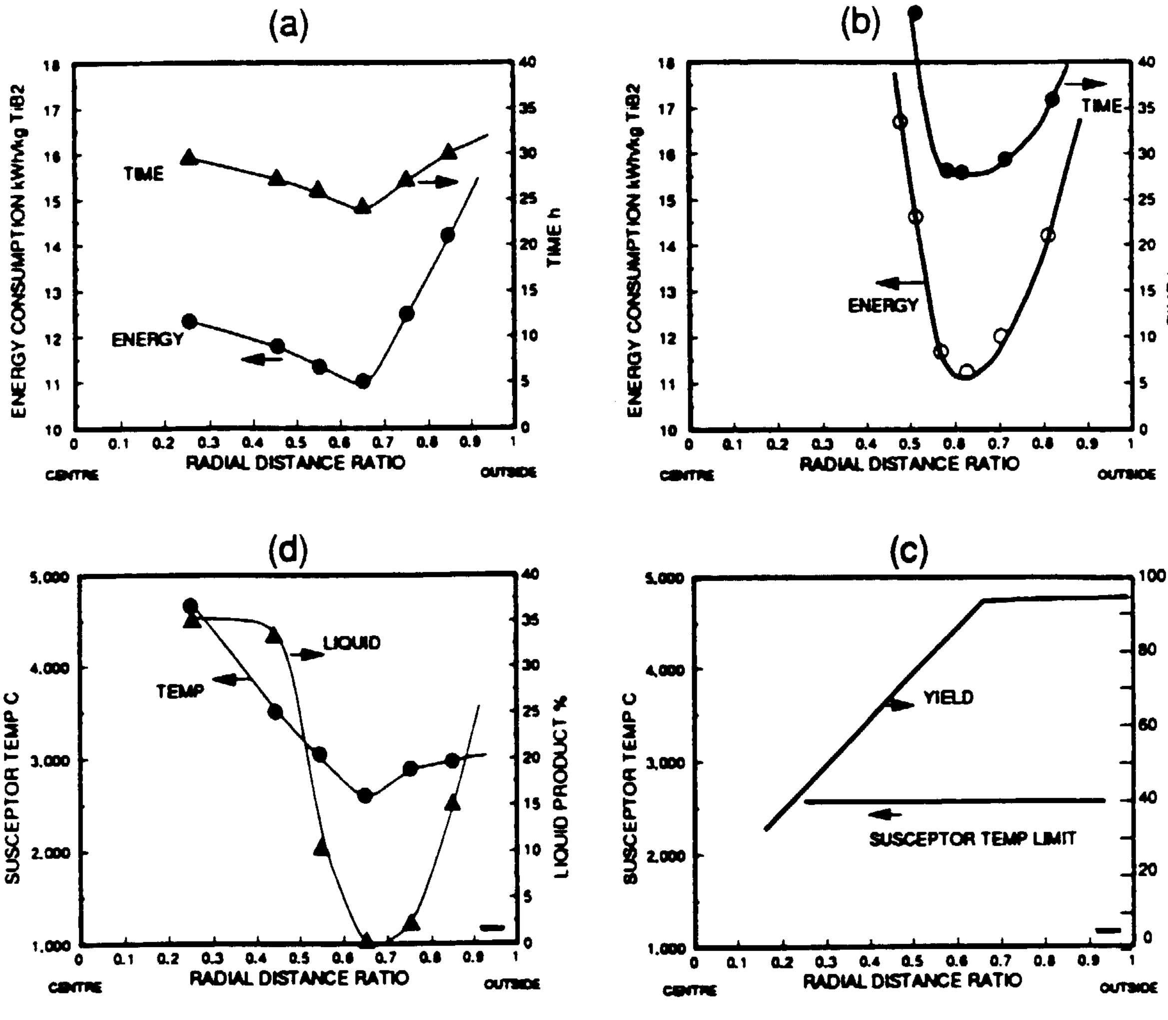

Figure 9

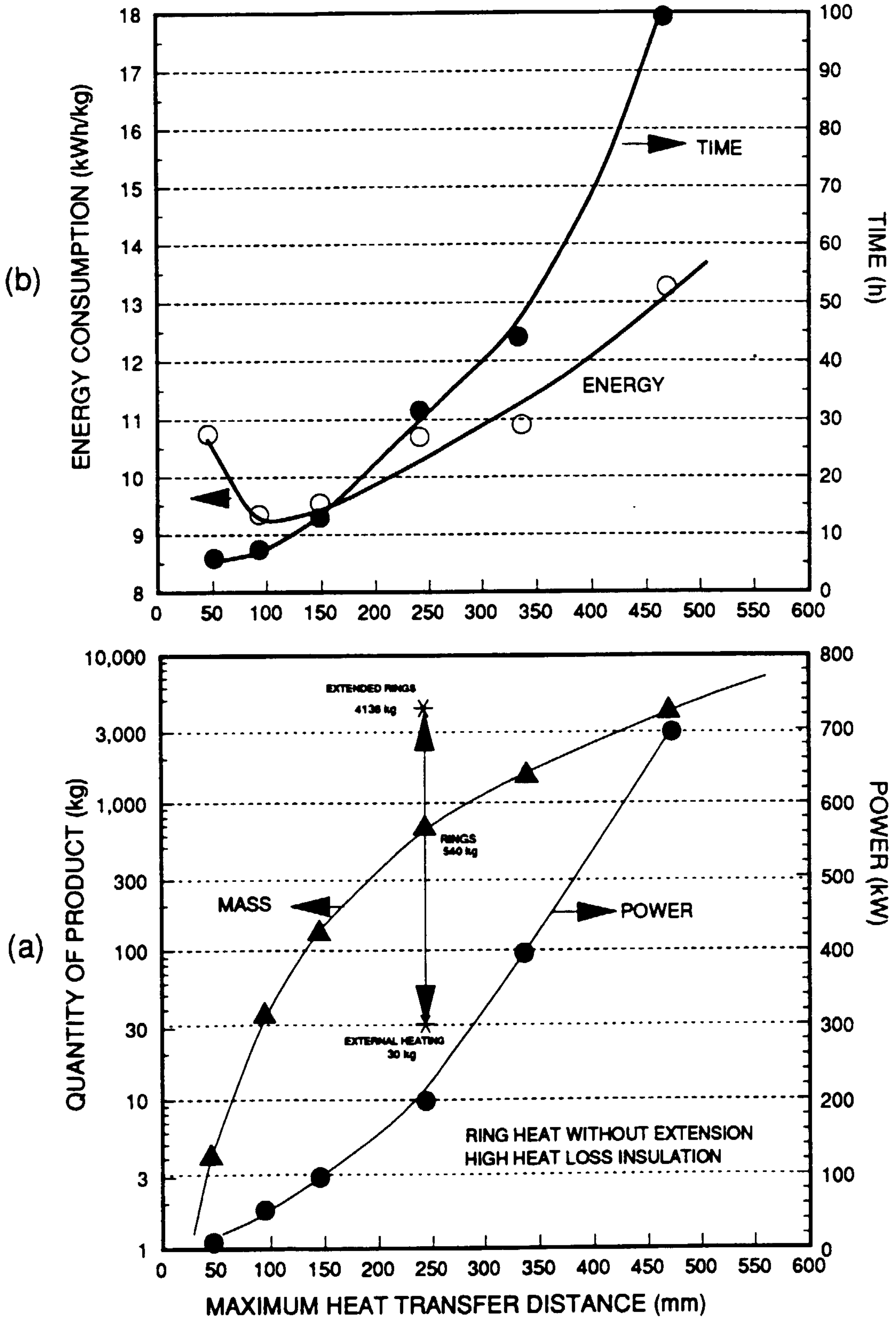
(b)
ENERGY CONSUMPTION (kWh/kg)
18
17
16
15
14
13
12
11
10
9
8
TIME (h)
100
90
80
70
60
50
40
30
20
10
0
TIME
ENERGY
0
50
100
150
200
250
300
350
400
450
500
550
600
(a)
QUANTITY OF PRODUCT (kg)
10,000
3,000
1,000
300
100
30
10
3
1
POWER (kW)
800
700
600
500
400
300
200
100
0
EXTENDED RINGS
4136 kg
RINGS
540 kg
EXTERNAL HEATING
30 kg
MASS
POWER
RING HEAT WITHOUT EXTENSION
HIGH HEAT LOSS INSULATION
0
50
100
150
200
250
300
350
400
450
500
550
600
MAXIMUM HEAT TRANSFER DISTANCE (mm)

Figure 10

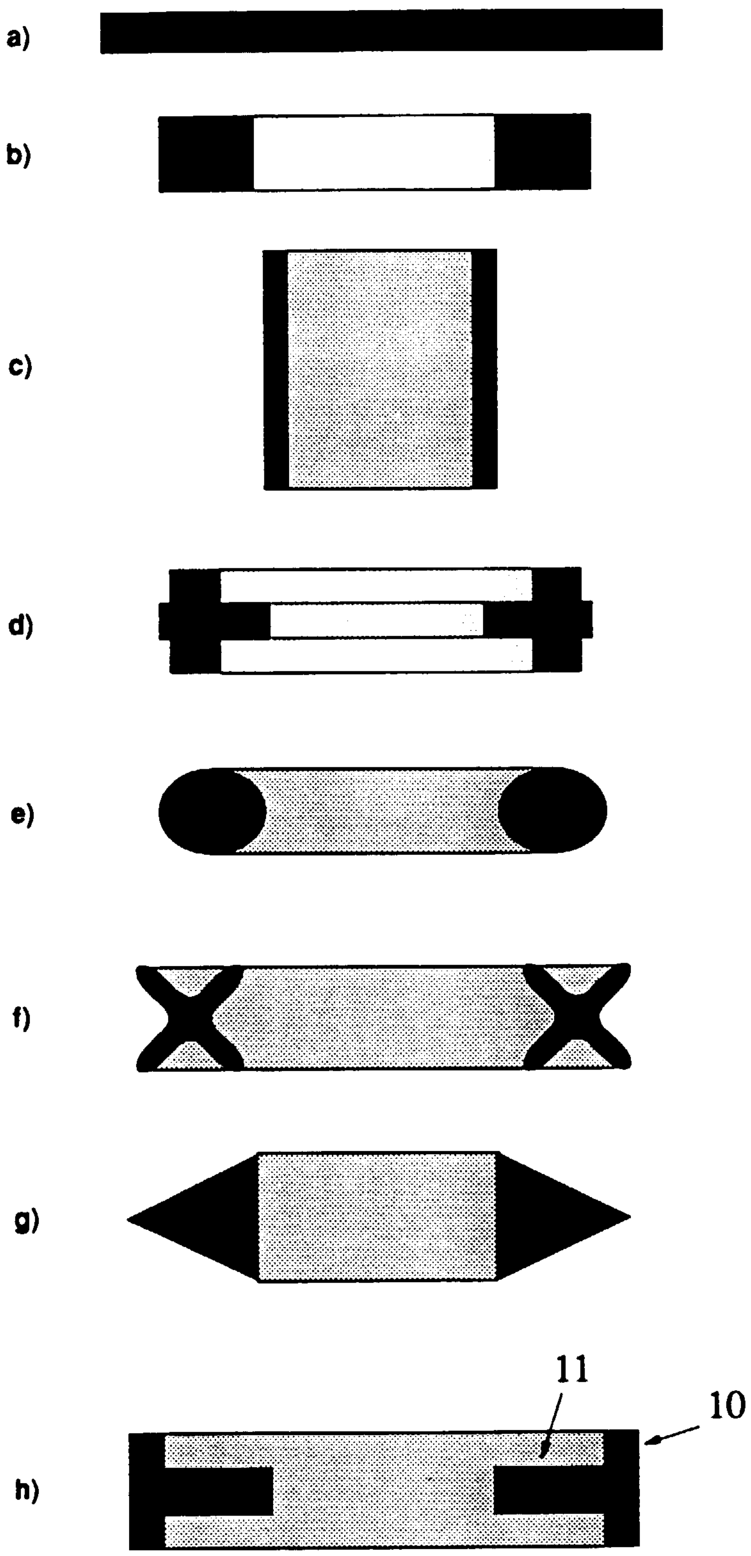

Figure 11