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DOE-ENERGY RELATED INVENTIONS PROGRAM GRANT #DE-FG01-96EE15666

FINAL REPORT

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INTRODUCTION

About five years ago development work began on a new concept for processing metal powders at high temperature under various special atmospheres. The process was called mechanical fluidization. The machine which performs the process is known as a Mechanical Fluidized Vacuum (MFV) machine because it is possible to fluidize material in a vacuum, something that heretofore was impossible.

In an MFV machine, a horizontally disposed retort is two-thirds filled with material and rotated at a speed that keeps the material in a fluffed up or fluidized state. It's turning a lot faster than a kiln, but not fast enough to cause the material to centrifuge outward and stick to the walls. In this mechanically fluidized state it was discovered that the thermal transfer rate between powders and amongst parts immersed in those powders is extremely fast, faster even than a gas fluidized bed despite the total lack of gas in the retort.

Figure 1 compares the heat transfer rate in air, in a vibratory bed, in a gas fluidized bed and a rotary fluidized bed. As shown, the rotary fluidized bed heat transfer is much faster than all the others. Fluidization is entirely mechanical so no gas at all is required for fluidization; only the gas required for processing need be directed to the retort. It is possible to fluidize material in a vacuum -- a feat heretofore impossible.

Figure 1. Comparative heat transfer

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Figure 2. Mechanical fluidization concept

Figure 3. Production MFV machine

Mechanical fluidization is shown conceptually in Figure 2. The retort is heated from the outside -- usually by electric resistance panels mounted within an insulated chamber. The retort is rotated on a horizontal axis so that the material is constantly moving. It takes time for material to settle down and by the time it gets settled it starts to move again. Even with relatively coarse materials, about 80% of material is moving at one time. Nothing is still for longer than a half second or so. Inside the retort the gas composition is controlled by bringing gas in and out of the axle through a special commutator at the end of the axle.

Figure 3 shows the present design of the machine as it is applied to the treating of metal powders. The retort rotates on large ball bearings. Gas is brought in through the axle, passes through the internal filter, exits through a similar filter and then passes through an external filter before passing through the commutator. A reversing valve mechanism periodically back flushes gas through the filters in the retort. 0.5 micron average filtration is maintained inside the retort. 300 nanometer absolute filtration is provided in the exterior room-temperature filters. The exterior filters are oversized as gas flow is very low -- usually a few cubic feet per hour.

While in normal operation, the retort rotates on a horizontal axis. For loading and unloading, the entire retort and insulated heater is tipped on a cross axis into an up or down position for easy loading and unloading. Figure 4 shows an unloading sequence where a sealed container can be attached to the retort so that loading and unloading can proceed in a totally gas controlled atmosphere.

Figure 4. Atmosphere controlled loading and unloading

In some tests, highly oxidized material has been reduced. At the end of the test the material has been reduced so much it is actually pyrophoric and it is necessary to unload under argon to prevent the powder from bursting into flames. In other tests, pyrophoric material has been charged into the retort under an argon atmosphere. Materials can also be injected at any time during the test and of course gases can be changed at any time and any temperature.

FEATURES

Features provided by mechanical fluidization include:

1. Perfect mixing of gas and powders. There is no possibility of unequal contact nor any variation in temperature. Mixing is perfect and temperature is exact.
2. No gas is required for fluidization. One can use exactly what is required for the process. If a cover gas only is required, the savings can be considerable because the same gas can be maintained indefinitely.
3. There is no loss of material required other than the material one is actually trying to get rid of.
4. Unlimited intimacy is possible amongst powders and between powder and gases. Gases can be kept in contact with the material as long as is desired.
5. Agglomeration is eliminated. In most powder operations, the powder sticks together and has to be mechanically separated afterwards. Mechanical fluidization keeps the material in constant motion so it doesn't agglomerate. Further, because water can be precisely removed at desired temperatures, the tendencies for agglomeration are greatly reduced.
6. Temperature control is absolute. Even under partial atmospheres or vacuum, temperature control is still precise. One cannot measure temperature differences from one end of the retort to the other with ordinary thermocouples.

The MFV system was first envisioned for treating sold parts with the powders being inert and acting only to mix and transfer heat. ERIP Grant #DE-FC01-95EE15622 was awarded to explore this facet. Subsequently, in working with the National Laboratories, the idea of treating powders was born. An Innovative Concepts (InnCon) award DE-FG51-94R020433 was awarded to explore this idea and subsequently this project, ERIP Grant #DE-FG01-96EE15666 was awarded.

- **Perfect mixing**
- **Zero fluidization gas**
- **Reduced losses**
- **Unlimited time for intimacy**
- **Eliminates agglomeration**
- **Absolute temperature control**

Figure 5. MFV Features

OBJECTIVE

The objective of this project was to build and demonstrate a machine to thermally treat up to 600 kg lots of metal and cermet powders to temperatures of 940C with low energy, cost and environmental impact.

Four tasks were undertaken

1. Quantification tests of many powders were conducted on a small machine constructed during the InnCon trials. These tests proved the basic concept and were to attract potential users to more extensive evaluations on larger machines. Results of these trials are discussed in "Quantification Tests."
2. Design machine modification was done to expand the basic machine. In actuality, this turned out to be an extensive phase which resulted in a major design improvement to the concept. "Design and Construction Work" discusses the many facets of this effort.
3. Construction and preparation of retort was expected to be the "Building Phase." This phase actually resulted in a major change to the machine which was done in conjunction with a license and building program for a major customer. Details are discussed in "Design and Construction Work."
4. Performance trials were scheduled on the final machine to prove out the concept and determine cost parameters. These trials are still ongoing at the time of this report. Some trials to date are discussed in "Performance Trials."

QUANTIFICATION TESTS

The objective of this task was to determine potential equipment deficiencies and opportunities for improvement by conducting a variety of trials under a broad range of process conditions.

The deficiencies/improvement opportunities related to:

- Inefficient loading and unloading, particularly if these operations need to be conducted under protective atmosphere
- Possible clogging of high-temperature metal filters by fine powders and/or reaction by-products
- Possible "short-circuiting" of process gas from inlet to outlet
- Inadequate cooling, as during exothermic reactions

Initial trials included:

- Iron powder and aluminum powder were reacted to make iron aluminide
- Nickel powder and aluminum powder were reacted to make nickel aluminide
- Chrome was diffused into nickel powder
- Aluminum was diffused into chrome-nickel powder
- Cobalt oxalate was reduced to pure cobalt
- Ammonium paratungstate was reduced to elemental tungsten

All trials above were sufficiently successful to warrant continued study. Many processes beyond the scope of this project have since been tried or are scheduled to be treated.

One trial of iron and aluminum was set up so samples could be extracted from a hot retort during the trial. 500 micron diameter iron particles were mixed with 2 to 20 micron aluminum particles. Samples extracted at lower temperatures showed a mixture of aluminum and iron. After some time at higher temperatures, the aluminum was found to coat the iron. Later samples showed the beginning formation of iron aluminide. Finally, the aluminum and iron were fully reacted to form iron aluminide.

Figures 6, 7 and 8 show the progressive formation of aluminum on iron and finally the reaction to form a true iron aluminide.

These first trials on 500 micron iron powder showed about 50 micron penetration of aluminum into the iron. Later trials with 50 to 100 micron iron showed uniform formation of iron aluminide throughout the particle.

Copper oxide trials were not as encouraging.

- Copper oxide reduction showed the commencement of pure copper extraction but the internal metal filters became clogged with copper before the trial was completed.
- Copper was oxidized and the same clogging of metal filters occurred.

Tests were intended to explore the limits of current filtration technology. Alternates under consideration may represent an advance in this field.

Figure 6. 500 micron iron with aluminum fines

Figure 7. 500 micron iron with aluminum coating

Figure 8. 500 micron iron with iron aluminide exterior

DESIGN AND CONSTRUCTION WORK

Design work in this project was leveraged with the design of a unit for shipment to a company who had taken an initial license for the treatment of powders. That unit was somewhat smaller but many parts were common. Later in the program a second larger machine was ordered for delivery to Pennsylvania. This larger unit has not yet been delivered.

Equipment improvements indicated by the quantification tests are as follows:

- a) Commutator life and reliability
- b) Tilt capability to facilitate loading and unloading powders
- c) Automated reversing valve for process gas flow, to periodically backflush high-temperature metal filters
- d) Increased capacity
- e) Automated back pressure valve for process gas exhaust, to assure adequate mixing of process gas and solids
- f) Retort cooling system

Design activities in each of these areas was conducted in two phases:

- Design and construction of the chassis for tilt operation, and
- Concurrent design and prototyping of the valves and cooling system, as design considerations for each component were linked by virtue of limited commutator dimensions

a) Commutator design was the subject of many trials. The seal in the commutator went through four design iterations before selecting the present design. Figure 9 shows the present commutator design.

b) A substantial reorientation of the design was required to provide the tilt-to-load and unload feature. the entire retort plus heater housings and all of the rotating parts were mounted on a frame which tilts on a cross-axis as shown in Figure 3. Tilt is controlled by a non overhauling gear motor and chain drive combination. With the addition of "soft spots" and limit switches, the unit can be precisely positioned from vertical up to vertical down.

Figure 9. MFV commutator design

c) An automated reversing valve proved very effective to back flush the internal filters. The reversing valve shown in Figure 10 allows same direction flow into and out of the valve but reverses the flow into the retort. Actuation of the valve is by an external camset controlled by an air powered cylinder. External control was chosen

because the wires passing through the commutator are not sufficiently heavy to power an actuator. The external camset is positioned by the air cylinder to cause the reversing valve to be in an up or down position. Position of the valve is normally changed every ten minutes. It is controlled from the control room by computer signal.

Figure 10. Reversing valve

d) The larger MFV unit is shown in Figure 3 (previously). The retort section which was tested under this grant will fit interchangeably between this large machine and the older linear machine shown in Figure 11.

e) A "Huff and Puff" valve was designed into the gas exhaust, controlled by pressure in the retort. Normally closed so that pressure builds slowly in the retort, the valve opens upon reaching a preset pressure of 2 to 3 psi. Because the exhaust line is unrestricted, the valve is open only a short time. The H & P valve assures that incoming gas cannot pass direct to the exhaust, assuring it mixes with powder in the retort.

f) Cooling the retort after the test occupied much design and testing. Cooling water is directed through the commutator and thence to cooling coils at the back or the retort. To avoid shocking the retort and to avoid high

Figure 11. Original MFV pre-prototype

temperature in the commutator exhaust line, a bypass line initially directs most of the water flow directly to the exhaust and bypasses the hot retort. As the retort accepts the flow of cooling water, a valve in the bypass line is slowly closed to direct more cooling water to the retort. The amount of bypass is controlled by the temperature of cooling water through the retort. As this report is written, the cooling water system is not totally satisfactory and is being replaced by an air cooling system. Testing of the air system is being supported by a licensee.

To the extent practicable, the "baseline" linear model (Figure 11) was used as a test bed for improvements before final assembly and operation of the improved system.

In general the design has proved exceedingly robust and a production version is being constructed. The cooling system was the only truly troublesome component and has not been included in the designs.

PERFORMANCE TRIALS

Final tests for this project concentrated on:

1. Testing of a large sized retort measuring 22 inches in diameter and 30 inches long. The retort can hold upwards of 4000 pounds of material such as tungsten carbide, but because of extensive customer interest and availability of material, trials were conducted with much lighter zeolite.
2. Testing of nickel and iron aluminide in larger lots than the quantification trials afforded.
3. Preliminary testing of a welded retort seal concept.
4. Expedited cooling between tests.
5. Accumulation of operating cost data -- especially maintenance concerns.

1. Large Retort Tests

A retort measuring 22 inches in diameter and 30 inches long (6842 cu. in. useful volume) with a welded connection between retort halves was constructed. This retort is interchangeable with the old and new MFV machines (with minor changes). At this time a second MFV machine is also being constructed for the Pennsylvania customer with the same 30 inch retort length but a smaller inside diameter of 18.75 inches to accommodate a bolted connection between retort halves.

Tests with zeolite showed identical fluidization, temperature uniformity and reaction as the smaller retort used in the quantification tests and an "in between" sized retort measuring 18.75" diameter and 4" length (663 cu. in. useful volume) used in other final trials.

Tests were conducted whereby 26% by weight cobalt was added to tungsten carbide. 26% weight is more than 50% volume, so the cobalt was added in two steps to assure good coverage. Samples taken after 2 hours of exposure to 13% cobalt showed insufficient bonding, but this was expected since 4 hours was the target time to assure bonding. When the second 13% cobalt was added and the final two hours of diffusion were finished, the bonding was complete and uniform. There was, however, some satellite bonding of powders which may be detrimental and needs further study.

2. Larger Lots Nickel and Iron Aluminide Tests

What was different was the control of exothermic reactions such as between nickel or iron and aluminum. In the smaller retort used for quantification trials, 15 inches diameter and 1 inch long (16 cu. in. useful volume), exothermic reactions could be easily controlled by bring heat up slowly to the exothermic reaction temperature. In the mid-sized retort the reaction proved barely tolerable with 5 % aluminum. Test beyond 5% or with the larger retort were not deemed likely of success without means to slowly insert the aluminum. Injection of aluminum was proved successful in small amounts. At this time a larger injection device is being constructed. The larger unit can inject an amount equal to 60% of the useful retort volume.

Figure 12 shows temperature versus time for 5% aluminum in nickel in the 18.75 dia. x 4" retort. Note that temperature in the retort ran ahead of heater temperature when the exothermic temperature was reached at about 480C.

3. Welded Retort Seal Concept

The welded connection proved reliable and trouble-free. It provided a 30% increase in retort volume and eliminated the need for a bypass line. However, a reliable method of making and breaking this weld is required. A design of an auto-welding machine has been developed in principle with a local company, but funds have not been available to fully develop this design.

Figure 12. Time-Temp for Iron Aluminide

4. Expedited Cooling

Expeditious cooling of the machine between runs was investigated with cooling water injected into cooling tubes at the back of the retort. The water entered and exited through the commutator. Although the design was augmented until it worked well, it was not reliable and was costly and a source of maintenance headaches. Cooling by air was simulated by opening the upper heater housing; this worked well but released heat into the area. An air cooling system using air forced from a turbine blower through the heater housing was set up and still holds promise,

though the exhaust port used in the first test was clearly too small and limited the cooling effect. This area is under active study in ongoing engineering developments.

5. Operating Cost Data

Construction Costs

Purchase cost of materials for a machine with a useful volume from 600 to 6000 cu. inches has been found to be \$50,000 to \$55,000. This cost contains no overhead or engineering charges. Probable selling price is in the range of \$100,000 to \$250,000.

Operating Cost

Operator costs for one eight-hour run of nickel aluminide:

Manpower:	1 man @ \$18 per hour for 8 hours	\$144.00
Gas		
Hydrogen	8 cu ft @ \$.25/cu ft	\$2.00
Argon	16 cu. ft @.04/cu ft -	\$.64
Electricity	80 kw/hr @ .07 kw/hr	\$5.60
		\$152.24

Maintenance (per one run)

Filters	External: 3 units (replaced once every ten runs at \$12 each (3 x 1/10 x \$12	\$3.60
	Internal: 2 unites (replaced once every five test runs at \$50 each (2 x 1/5 x \$50)	20.00
Miscellaneous		\$10.00
		\$33.60

Therefore, assuming one eight hour run of 1000 pounds of nickel aluminide, the cost is \$.19 per pound, which is very competitive. Current competitive costs run as high as \$2.00/lb.

Results of Performance Trials

The large retort design test proved:

- Temperature uniformity, mixing and general function of the MFV concept is unaffected within the size range tested and sizes much larger seem feasible.
- Means to inject exothermic-causing materials is necessary but development of a commercial system is beyond the scope of this project.
- The welded seal concept works in principle and provides significantly more useful volume because it permits a larger retort diameter. Work is still to be done on easing the welding and breaking of this joint to make it commercially acceptable.

COMMERCIALIZATION

The MFV system is the subject of several licenses by KDC to others.

A license for use of the MFV machine had been negotiated with a Canadian company before this contract was awarded and a small machine was built and delivered to that firm. Their non-exclusive license is limited to powder for thermal spray.

A Pennsylvania concern has licensed the process for surface hardening of parts not a target of this ERIP, but the test work which they are conducting in conjunction with KDC is valuable to both part and powder treatment. The Ben Franklin Technology Center of Western Pennsylvania has contributed \$100,000 of matching funds to the Pennsylvania company's efforts.

A second Pennsylvania firm is testing the treatment of ferric oxide derived from waste pickle liquor. A license program has been promised if tests are successful. This program is also different from the ERIP program intent, but the support has helped in the past few months. The Pennsylvania firm is supporting financial cost of all tests.

An option to license all the remaining technology has been signed with a new Texas corporation. During a 4 month option period commencing approximately May 1 they are supporting the program and expect to have more than \$1 million available to the program at the end of the option period. Their first efforts will be directed to the thermal spray market estimated at \$300 to \$400 million of costly powders. The second and third markets are the magnet market at \$800 million of mid-cost powders and the powder metallurgy market at \$1.2 billion of low cost powders.

FUTURE WORK

Future work is needed on:

- An economical version of the welded seal between retort halves which proved so conceptually sound in these tests. Argonne National Laboratories is considering funding this development as part of their on-going evaluation of the machine for encapsulating radioactive salts in zeolite, but no decision has been reached at this time.
- A means of injecting large quantities of solid powders in a controlled manner to match the resulting exothermic heat generation with heat losses. This work will probably be funded by the Texas licensee as part of their investigation into the production of aluminides.
- Expeditious cooling of the retort after the desired reactions have been completed. Presently the MFV unit must be opened to allow heat to dissipate. Water cooling through the commutator has not proved practicable. Tests of an air cooling scheme are presently being funded by KDC.

- Very large retorts -- a scale-up of 10 to 100 will be needed to adapt the unit to low cost treatment of ores and waste materials. A recent proposal to DOE under the NICE3 program was rejected. It will be resubmitted and other sources will also be investigated.
- The application of MFV technology to thermal storage and transfer of heat for solar energy and possibly even home heating looks promising and should be funded by another ERIP or similar grant.