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AUTOMATED THERMAL TREATMENT OF METALS WITH A MECHANICALLY FLUIDIZED VACUUM MACHINE

Final Report
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FINAL REPORT

Overview

This ERIP project "Automated Thermal Treatment of Metals with a Mechanically Fluidized Vacuum Machine" produced more benefits in unintended areas than in the original intent of the program. The first project was directed to heat treating of solid parts using a retort half filled with fine powder. The treatment of metal powders was not originally envisioned at the time of proposal preparation. This second application, where the powder itself is being treated, has turned out to be a multi-billion dollar market in which the Mechanical Fluidized Vacuum machine can create revolutionary changes. Consequently most efforts in recent years have been dedicated to further growth of the powder markets. These efforts procured a second ERIP grant titled "Thermal Heat and Diffusion Treatment of Bulk Powders."

Already three commercial licenses have resulted in the powder treatment area.

This ERIP grant, "Automated Thermal Treatment of Metals with a Mechanically Fluidized Vacuum Machine," was used to build a machine which has been extensively used for both powder and parts treatments. Considerable base data was generated. Although proposal funding has been expended, development work in solid parts treatment is actually increasing at the present time. Commercialization is expected within two years. (Powder applications have already been commercialized.)

Review of Objectives

Objective 1. Design and build a prototype machine capable of determining quantitatively the ability of the Mechanical Fluidized Vacuum retort to heat, hold and cool within a predetermined atmosphere control. The MFV machine will provide a means for performing any style heat treat to parts smaller in size than about 8 in^3, which represents the majority of parts heat treated in the U.S. It will have the ability to heat, hold and cool, allowing multi-step processes which can now only be performed through manual coordination of several devices.

A machine with a working diameter of 18.75" and a length of 4" cylindrical and an additional 4" conical section has been designed and constructed. The machine has been used for treatment of both solid parts and powders. The features of dual axle construction, full computer control and automatic control of water cooling have been included. It was originally hoped the cooling rate achieved would be suitable for quenching activities. This has not proved to be the case. However, an alternate method for achieving rapid quench, rapid media exchange, has been developed and tried in principal but not yet fully proven successful. In the media exchange method, hot media is dumped out of the retort. While the fixtured workpieces in the retort are still hot, cool media is injected into the turning retort. Full tests of this principle are expected in 1998.

The basic model as constructed is shown in Figure 1. Photographs of the actual machine are shown in Figures 2 and 3. A website accessible at http://kempdev.com describes the full capability of the machine for both powders and solid parts.

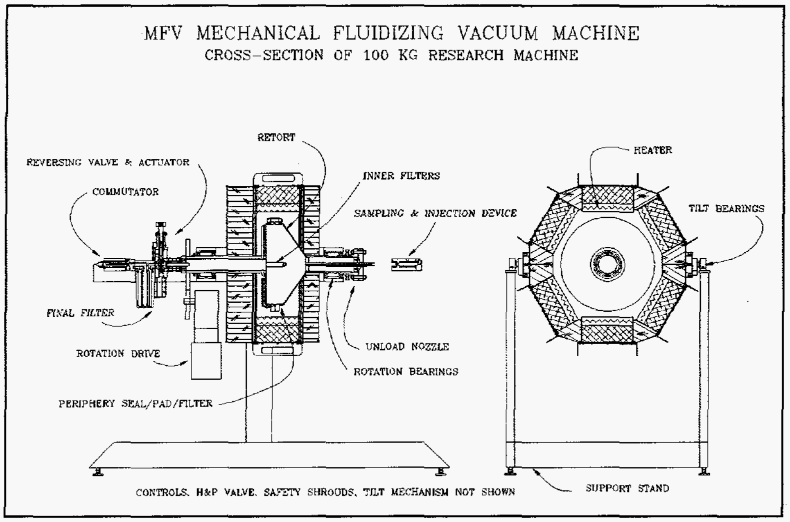

Figure 1

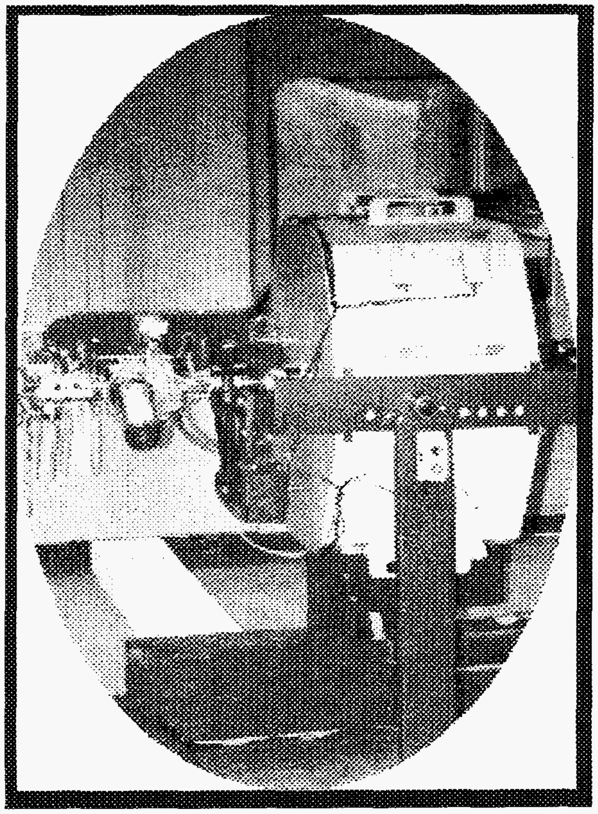

Figure 2 - Exterior View of MFV Tilt Style Machine
(Control room in background)

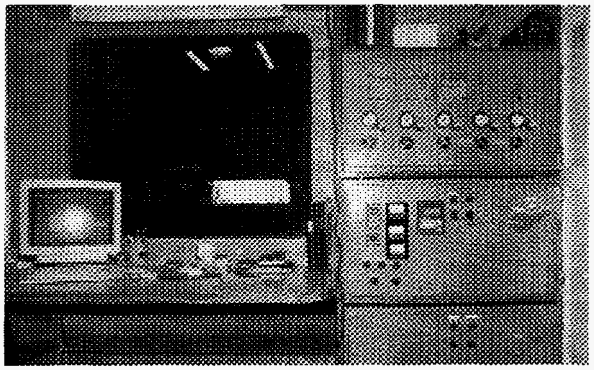

Figure 3 - Interior View of Control Room

Objective 2. Analyze the product treated in an MFV to prove the metallurgical objectives have been obtained. Photomicrographs, hardness tests and scanning electron microscope analyses are the basis upon which user engineers select and approve processes.

A number of instrumented runs have been completed with the machine using the water cooling coils on the outside back of the retort. Results of these cooling curves are shown in Figure 4. They do not compare favorably with what is achievable by air cooling of the retort exterior as also shown in Figure 4. It appears that at higher temperatures air cooling is more efficient whereas at the lower temperatures it is probable that the water cooling is more efficient. A combination method is presently being developed.

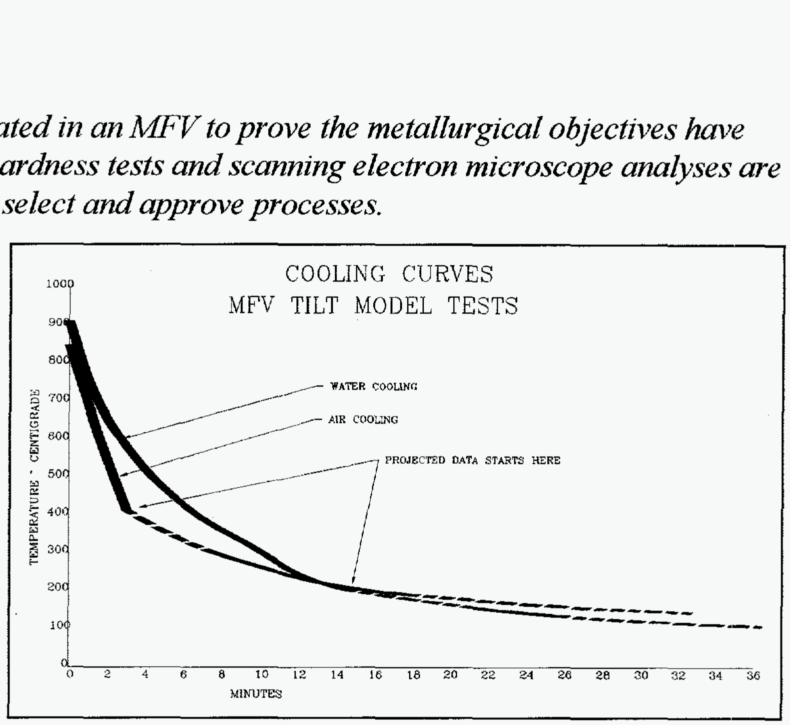

Figure 4

Internal heat treat transfer rates obtained in a mock up machine are shown in Figure 5. These were obtained by taking a calorimeter approximately 2" square. The calorimeter was first heated to about 800F in an oven then cooled in air in a vibrator bed, in an actual gas fluidized bed, and in a simulated rotary fluidizer.

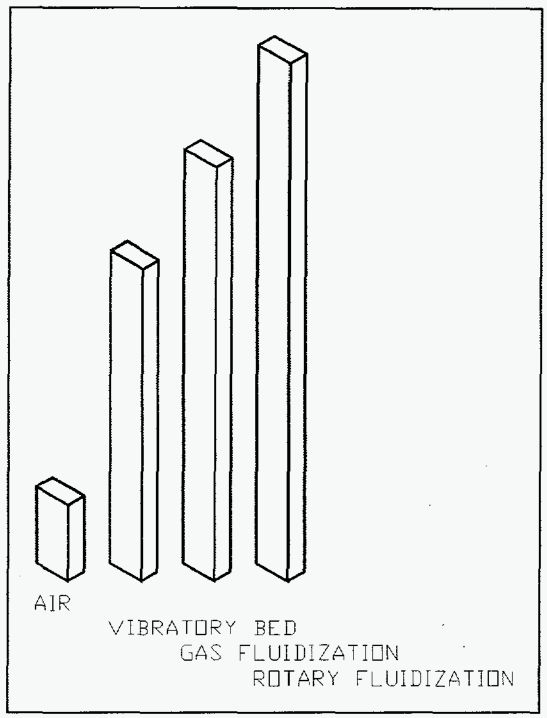

Figure 5 - Heat Transfer Rate Comparison

Instrumentation of the machine was done in preparation for extensive work to be conducted in conjunction with a CRADA at Los Alamos National Laboratories. LANL provided an engineer and equipment to KDC facilities. However the results of this test were most unfortunate. Part of the equipment did not work. Then the engineer became ill immediately after the work. As of this date, a year and a half after the test, no report has been received from LANL.

Lacking the chromatograph report from LANL, an alternate plan to achieve some of the results was invoked. A clear plastic filter bowl was used as the external filter on the tilt machine. In reduction tests where water was given off by reaction of the oxide with hydrogen, the water condensed within the clear plastic bowl. This "poor-man's chromatograph" gave some indication of when and how much water was being given off. A modicum of control was achieved and has proved quite successful in reduction of ammonia paratungstate to tungsten and cobalt oxalate to cobalt. However, both of these have been powder treatments. The technique has not yet been used for solid parts.

Sample heat treatment

The following test results have been achieved on solid parts:

1. Figure 6 is a curve of hardness versus depth achieved by carbo-nitriding of low alloy high strength bolts. This data was prepared by LANL.

2. Sample parts were blued in the machine by introducing water into the machine at temperature. A pleasing blue-black color was achieved.

3. Samples of a palladium iron compound was fabricated into electrical contractors and tested in the machine at about 800F. Purpose was to simultaneously age the material while also deburring it. The tests showed good aging but minor deburring results. In many ways this was an indication that the mechanical fluidizing action is quite gentle. It is in no way sufficient to scratch or mar the parts.

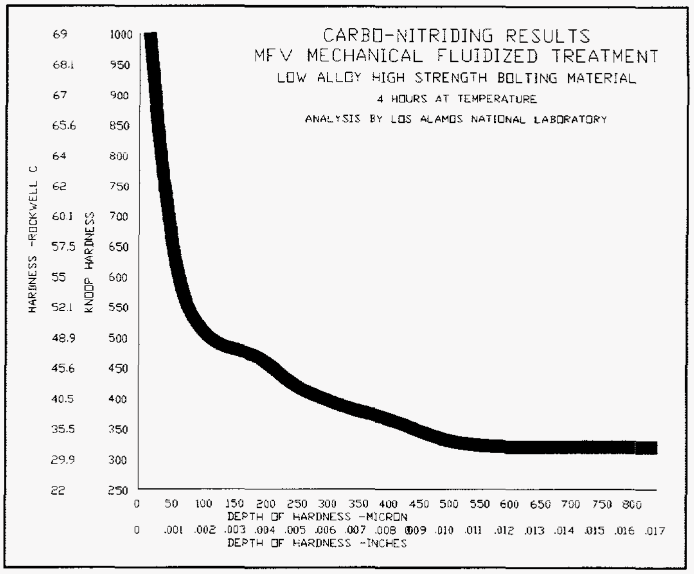

Figure 6

Samples of titanium, low strength alloy steel and stainless steel were exposed to a boronizing compound in the MFV machine at about 800F. Results analyzed by LANL indicate varying degrees of boron pickup. The results were spotty; however, the results were sufficiently attractive that the Benjamin Franklin Institute has now inaugurated an extensive program to develop the MFV for boronizing of solid parts. The work will be conducted in Pittsburgh in a machine by KDC and under joint KDC and University of Pittsburgh supervision. However, this work will be accomplished in 1998.

Objective 3. Determine the energy costs and operating costs and compare to conventional and vacuum heat treat methods. Preliminary estimates indicate significant energy savings, greater than 50% in many cases. Operating costs should be reduced through the achievement of multiple tests with a single load and unload.

Several cost calculations have been attempted based on obtained operating data. They are shown as Figures 7 and 8. The use of energy is so low as to be confused by similar costs for space and heating and air conditioning. However, the data which has been available has been sufficient to allow three separate commercial firms to proceed with the machine. Energy savings, while not accurately determined, are obviously at least 50% less than competing techniques. More accurate data will be obtained during initial commercial trials in early 1998.

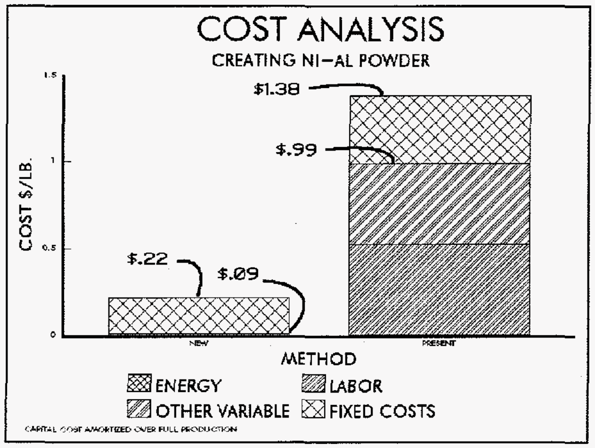

Figure 7

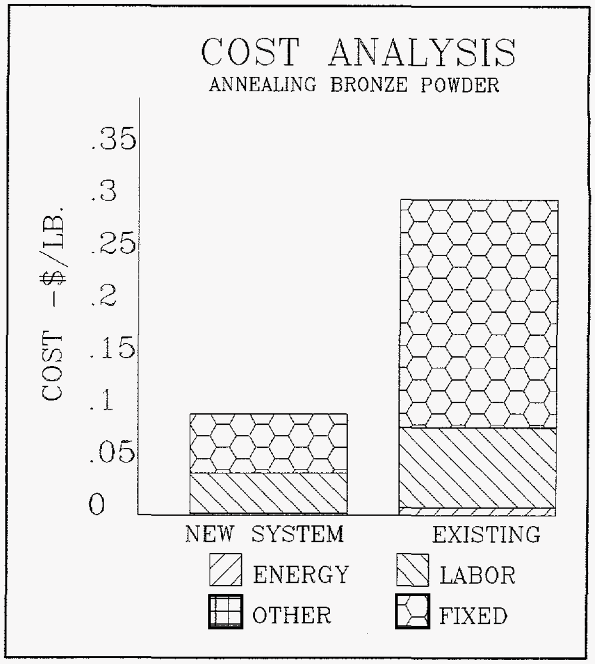

Figure 8

<u>Additional Data</u>

The following publications regarding the general aspects of the machine have been made:

Advanced Ceramics Report	November 1995
Advanced Manufacturing Technology	May 15, 1995
Advanced Materials and Processes	May 1995
Advanced Materials and Processes	July 1995
Advanced Materials and Processes	April 1996
Advanced Materials and Processes	August 1997
ASME Petroleum Division News Briefs	April 1997
Aviation Week	May 8, 1995
Bloomberg Columns, White House Watch	February 21, 1997
Ceramic Industry	April 1995
Dateline: Los Alamos	August 1995
Design News	June 26, 1995
Houston Chronicle	May 22, 1995
Industrial Heating	October 1995
Industrial Heating	February 1996
Industrial Heating	March 1996
Journal of Metals	February 1996
Journal of Metals	April 1996
Los Alamos National Lab Newsbulletin	April 28, 1995
Mechanical Engineering	January 1996
Metal Heat Treating	March/April 1995
Metal Heat Treating	May/ June 1995
Metal Heat Treating	January/February 1996

Metal Forming	February 1996
Metal Powder Report	November 1995
Networking (HTN)	Fall 1995
Networking (HTN)	Spring 1996
Networking (HTN)	Spring 1997
P/M Technology Newsletter	January 1996
Powder & Bulk Engineering	July 1995
Powder & Bulk Engineering	February 1996
Tap In To Success: Triumphant Tales of Technology Transfer	March 1996
Techbridge	June 1995
Technical Insights' High Tech Materials Alert	February 1997