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**Final Report
For
CRADA Number ORNL-531**

**Microwave Treatment as a Pesticide Alternative for Stored-Products
By**

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and

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Abstract

This CRADA was a continuation of earlier work with Micro-Grain, Inc. to develop a high power, high frequency microwave treatment process to treat insect infested grain. ORNL's role was as a subcontractor to Micro-Grain's Phase II SBIR project funded by the US Department of Agriculture. The primary objective was to develop a commercial scale prototype unit capable of treating infested grain at flow rates approaching 1 kg/sec, which is required to be viable in the grain handling industry. A flow rate of ~ 0.12 Kg/second was demonstrated at 20 kW microwave power level with 100% kill rate. The system is capable of 200 kW however waveguide arcing due to grain dust in the waveguide limited the power to 20 kW during the tests. Development tasks performed during the project included modification of an existing high-power microwave exposure facility to uniformly process large grain samples at high flow rates and improved instrumentation to detect grain flow and uniformity. Microwave processing tasks include a series of controlled exposure tests using infested grain samples provided and analyzed by the University of Oklahoma. Grain samples were infested with red flour beetles which proved the most difficult to kill in earlier tests. Most of the samples processed resulted in quite successful kill rates and a maximum grain temperature of 46°C. The facilities utilized at ORNL are located in the Fusion Energy building (9201-2 at Y-12) and include the 28 GHz 200 kW CW high power microwave facility and microwave test equipment associated with the FED Microwave Development Laboratory in 9201-2. An improved microwave exposure chamber and grain flow control and handling equipment were designed and build as a joint effort between Micro-Grain and ORNL. A number of insect infested grain tests were successfully performed although the higher power, higher flow rates were limited by arcing in the microwave waveguide and damage to the gyrotron output window. Test results and the overall performance of the applicator system are very favorable for continued development of the concept. Further tests were performed in a large high power 2.45 GHz microwave applicator in batches. These samples were also quite effectively treated which supports the concept that a lower cost, lower frequency microwave system might be more successful due to the improved economics and simpler operation and maintenance of the low frequency system. Follow-on work is still possible however the untimely death of Steve Halverson, founder of Micro-Grain, has essentially brought the development work to a close for now. Micro-Grain is being run by relatives at a low level who are not actively pursuing further funding.

Introduction

The purpose of this Cooperative Research and Development Agreement (CRADA) between ORNL/UT-Battelle and Micro-Grain, Inc. was to investigate high-power microwave treatment of infested grain products as an alternative to current treatment methods involving pesticides. It was expected that this CRADA would demonstrate, on a commercial scale, practical and economical alternatives to the use of insecticides. The continued use of pesticides is likely to be restricted or banned by the Environmental Protection Agency. For example, the chemical methyl bromide, which is routinely used to fumigate grain storage facilities is proposed to be banned in the near future.

Previous scoping studies performed by the Micro-Grain & ORNL and others have successfully demonstrated the potential effectiveness of high-power microwave treatment of infested grain samples. These studies indicate that a high insect mortality rate is possible while maintaining the vitality of the wheat itself for replanting. Microwave fields, which can penetrate some distance into the bulk wheat, are directly absorbed, primarily by the unbound water component of the insects, through the dielectric absorption process. A careful balance between underheating of the insects and overheating the wheat must be maintained. One approach is to operate at high microwave frequencies such as 28 GHz or higher where the power is preferentially absorbed in the moist insects and not the relatively dry grain.

To date, high-power microwave exposure tests have been performed on infested grain samples at a variety of frequencies including 2.45 and 10.6 GHz and 28 GHz, and medium power tests at 12, 15, 18 and 55 GHz. Most of these tests were preliminary in nature; however, statistically significant numbers of samples were treated. The highest power 28 GHz tests performed during the previous ORNL/Micro-Grain CRADA (96-0439) were quite successful in demonstrating the process in medium sized batches. Additionally, low power-swept frequency data on the dielectric loss of wheat and insects has been obtained using a network analyzer. A trend indicated by analysis of the data and modeling indicates preferential heating of the insects occurs as the microwave frequency is raised. The increase in loss of the bulk wheat with increased frequency levels makes high-frequency applicator design more difficult so one of the challenges faced during this CRADA was to develop an applicator capable of handling 5 Kg/sec or higher grain flow rates while maintaining a >99% insect kill rate. The grain must be dispersed adequately for microwave field penetration throughout the treatment volume.

Concerns of the U.S. government over the impact of pesticides on human health and the environment have prompted a search for alternatives for the control of insects in stored-products, specifically for methyl bromide, in accordance with the Clean Air Act (Section 602). Evidence that insects are becoming increasingly resistant to chemical pesticides, requiring increased dosages to be effective, and that pesticide residues in foodstuffs are potential pathogens in humans are also compelling reasons for developing alternative control methods. The proposed project is significant to the resolution of these problems and will continue evaluate the use of microwave and millimeter wave energy as an effective, economical and practicable alternative means of controlling insects in stored products at much higher flow rates than previously achieved. A successful system could eliminate the use of methyl bromide in the quarantine/ commodity category (~1.2 tons in 1990 in North America alone, Taylor 1994). It can also offer alternatives to the general use of pesticides in the high-value commodity sector of the stored product industry.

Project Objectives

Specific CRADA tasks for ORNL were:

- (0) Plan new experiments in conjunction with Micro-Grain and Univ. Of Oklahoma and prepare required safety documentation.
- (1) Develop an exposure tank and grain handling apparatus with adjustable flow rate and a grain diffuser to achieve highly uniform microwave exposure level. Prepare 28 GHz gyrotron and waveguide system for full-power operation and perform an efficiency calibration with a dummy load located inside the exposure tank.
- (2) Reinstall & checkout the grain flow-through system and associated instrumentation. Add improvements to reduce interference with flow instrumentation caused by grain dust.
- (3) Establish gyrotron operating parameters for tests including settings for various power levels and pulse lengths.
- (4) Perform tests on an agreed upon number of samples provided by Micro-Grain
- (5) Perform 4 weeks of actual testing
- (6) Cleanup the facility after tests are complete.

Prototype Applicator Tests

These tests are intended to provide a proof of principle test of infested grain treatment using high frequency microwave energy on an industrial scale. The existing 80-cm ID microwave exposure tank was modified to accommodate flow in 20-cm diameter inlet and exit feed pipe. A microwave compatible grain diffuser and collector was developed to permit maximum spread of the grain inside the tank to allow for uniform exposure to the microwave energy as the grain falls through the tank. ORNL assisted Micro-Grain in design the needed components to modify the tank. Specific ORNL tasks performed for this phase included:

- (1) Install new components on 28 GHz tank including top & bottom transition, valve, LED electronics
- (2) Re-route waveguide to side launch on the tank
- (3) Setup grain diffuser, pressurized air system, and other controls as needed. Study the grain flow pattern to check for uniformity of dispersal.
- (4) Checkout new system, establish gyrotron parameters and test system efficiency with water load placed inside tank

Success in meeting the objectives of the CRADA

Most of the objectives of the CRADA were met as outlined in the technical discussion. ORNL and MicroGrain developed a prototype applicator for high grain flow rates and performed high power 28 GHz flowing grain treatment tests on infested samples. The flowing grain tests were quite successful in demonstrating the technique on a nearly full scale based on the power capability of the system. A flow rate of ~ 0.12 Kg/second was demonstrated at 20 kW microwave power level with 100% kill rate. The system is capable of 200 kW however waveguide arcing due to grain dust in the waveguide limited the power to 20 kW during the tests. The power limit difficulty encountered was primarily due to grain dust entering the waveguide, which likely would be alleviated by use of a self-cleaning quartz bell jar window inside the applicator chamber. Batch processing of samples at a 30 kW at 2.45 GHz was also successful. This system is capable of 500 kW

Demo Unit Design and follow-on tasks

ORNL provided high power microwave expertise for a paper design for Phase III industrial-scale demonstration unit. ORNL was to provide publication input as needed for a write-up of the project results and also participate in a Phase III SBIR proposal as requested by Micro-Grain. Unfortunately, due to the untimely death of the Steve Halverson, Principal Investigator and founder of Micro-Grain, the effort for the Phase III demonstration has not yet been undertaken.

DOE benefit from the CRADA

This CRADA was not directly funded by DOE. This was a funds-in CRADA fully funded by Micro-Grain and USDA. However, the DOE Office of Science- Office of Fusion Energy program benefited through the continued development of high power high frequency microwave technology relevant to fusion energy research as well as possible future benefit to the Office of Industrial Technology if this technology is applied to other applications.

Technical discussion of the work accomplished

See the attached appendix for additional technical details and results of the research

Inventions made or reported

Micro-Grain (the originator of the concept) submitted an invention disclosure on the process and received a patent before this particular CRADA began. ORNL was cited as co-inventor of portions of the technology based on earlier contributions. An ORNL invention disclosure form was completed for the record.

Assessment for Commercialization

The concept appears to be commercially viable based on the flowing grain treatment results and also the lower frequency batch tests. A pilot plant study will likely lead to commercial success if suitable financial support can be obtained. The death of Steve Halverson, founder of Micro-Grain has made continuing the project as originally managed quite difficult.

Plans for future collaboration

Micro-Grain's new staff continue to work on furthering the project at a low level and would like ORNL to assist in further efforts.

Conclusions

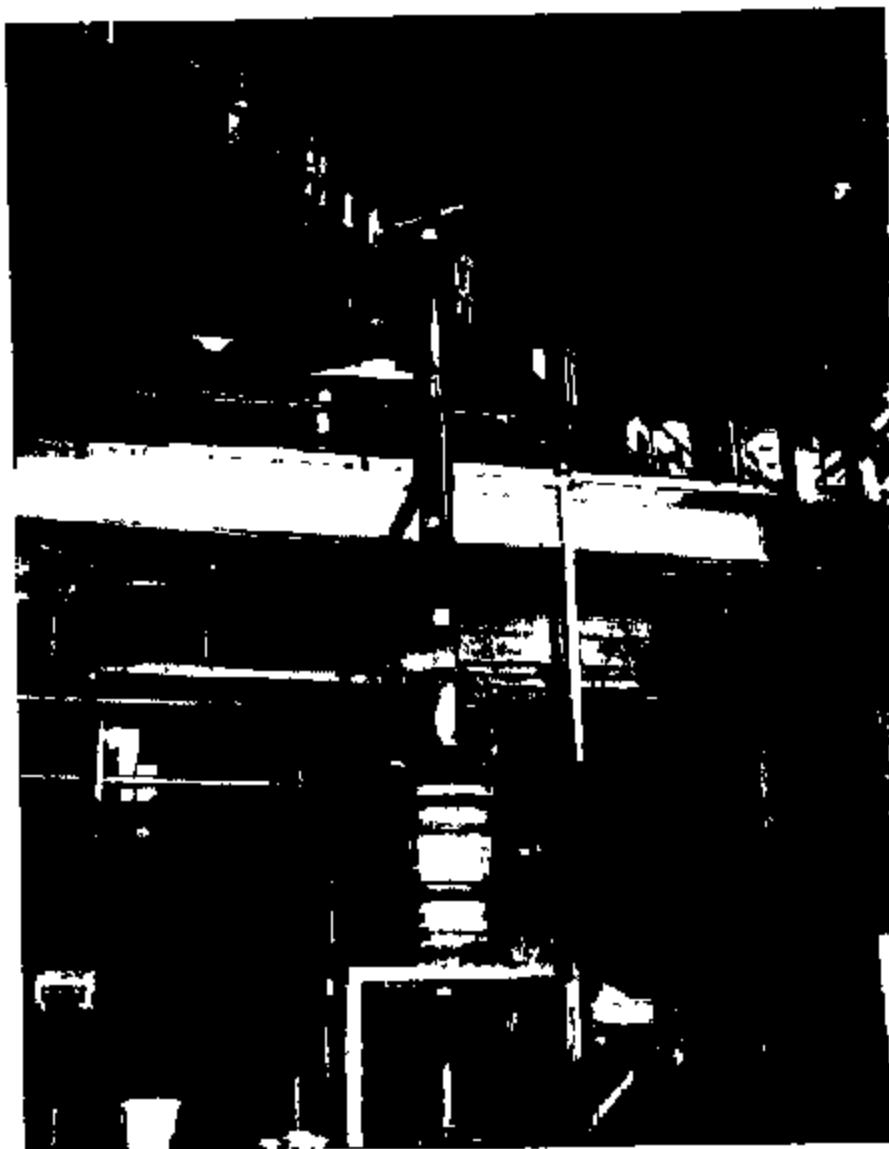
The CRADA has proven quite successful to date and very beneficial to all parties involved. ORNL needs to keep its partially used facilities available as a basis for future work. Large grain flow rates of several Kg/sec can be processed and deinfested by the grain applicator developed. A technical detail of microwave arcing in the waveguide due to dust accumulation can be solved by the addition of a self cleaning quartz window protruding into the grain flow path. Funding appears justified for a further pilot plant study which could be performed at ORNL FED facilities with grain supplied by a grain storage company. Venture capital and funding grain storage industrial partner would be the most likely supporters.

Details on Work Accomplished

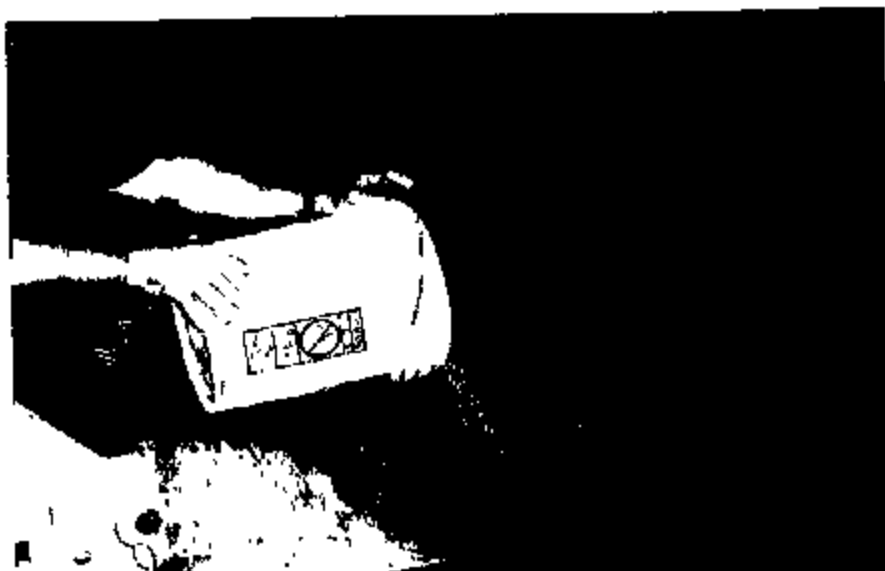
Some photos of hardware developed and tests in progress are shown below.



Looking up at grain diffuser inside microwave tank from below



View of grain hopper, waveguide from gyrotron and microwave exposure tank



Bill Halverson adding a grain sample in preparation for a run.



Stan Forrester (FED) inserting a batch sample in the 2.45 GHz batch applicator

Summary of infested grain exposure tests

Table 1 below summarizes the results of the insect mortality trials with the treated wheat from 7/2001 tests at ORNL. Despite the many problems encountered, the insect numbers look quite good when waveguide arcing did not occur. Due to the problems with the 28 GHz system, batch tests at high power 2.45 GHz were also performed with the remaining samples. These tests were also surprisingly good.

The most successful 28 GHz treatments were buckets 1, 2, and 6 treated at 20 kW, which was actually the dose at the low end of the proposed effective power range. There were no surviving insects from these three treatments. For the controls it is interesting to see how much simply being in the grain (buckets 18 and 19) affected survival, and how the additional dropping through the applicator with no microwaves affected survival even more. The jar-1 and jar-2 data given show (embarrassingly) that we at OSU were not very accurate at counting 150 larvae into the jars, but they also show pretty high survival if not put into wheat buckets.

The data from the 2.45 GHz static trials in the kiln chamber also look quite promising as indicated in table 2 below. Application of the 2.45 GHz to the flow through applicator to deliver that power to a 1-sec. dwell time of one bushel would be an interesting test however uniformity might not be optimum at such a low frequency. This could be solved using a larger chamber.

Table 1. The effects of microwave radiation at 28GHz on 150 larvae of the red flour beetle in 5-gallon lots of wheat treated in a flow-through system at Oak Ridge National Lab., July 20 and 21, 2001.

| Bucket Number | Treatment Description | No. of Survivors |
|---------------|---|------------------|
| Jar 1 | Control, larvae in small jar of flour remaining at OSU | 157 |
| Jar 2 | Control, larvae in small jar of flour, in luggage on trip | 147 |
| 18 | Control, in bucket, remain in lab. at OSU (no flow) | 116 |
| 19 | Control, in bucket, remain in lab. at OSU (no flow) | 103 |
| 3 | Control, dropped through applicator with no microwaves | 68 |
| 5 | Control, dropped through applicator with no microwaves | 71 |
| 10 | Control, dropped through applicator with no microwaves | 63 |
| 1 | 20 kW, T1=26, T2=45°C | 0 |
| 2 | 20 kW, T1=26, T2=45 | 0 |

| | | |
|---|--|----|
| 6 | 20 kW, T1=26, T2=45 | 0 |
| 7 | 26 kW, T1=25, T2=55; brief arc at start of treatment | 3 |
| 4 | 20 kW attempt; major arcing, T1=26, T2=48 | 46 |
| 8 | 26 kW attempt; major arcing, T1=26, T2=48 | 18 |
| 9 | 26 kW attempt; major arcing, T1=26, T2=48 | 45 |

Table 2. The effects of microwave exposure from a klystron at 2.45 GHz on 150 larvae of the rod flour beetle in 5-gallon buckets of wheat treated in a rectangular chamber (for wood drying) at Oak Ridge National Lab., July 21, 2001.

| Bucket Number | Treatment Description | No. of Survivors |
|---------------|--------------------------------------|------------------|
| 14 | Control, stay in bucket at ORNL | 121 |
| 15 | Control, stay in bucket at ORNL | 121 |
| 11 | 30 kW, 4 min 15 sec, T1=25, T2=60-65 | 0 |
| 12 | 30 kW, 1 min 28 sec, T1=25, T2=50-72 | 2 |
| 13 | 30 kW, 1 min 28 sec, T1=25, T2=50-72 | 0 |

Some Relevant Publications

Halverson, S.L., Bigelow, T.S. US Patent 6,192,598 issued on 2/27/01

Halverson, Steven L., Rudy Pierre, Wendell E. Burkholder, Timothy S. Bigelow, Mark E. Misenheimer and Erik V. Nordheim 1996a. Effects of SHF and EHF energy on the mortality of *Sitophilus zeamais* in soft white wheat. Paper No. 963013, ASAE Annual International Meeting, Phoenix AZ, July 14-18, 1996

Halverson, Steven L., Rudy Pierre, Wendell E. Burkholder, Timothy S. Bigelow, and Mark E. Misenheimer 1996b. SHF and EHF microwave radiation as a pesticide alternative for stored products. Paper no. 55, 1996 Annual International Research Conference on Methyl Bromide Alternatives and Emissions Reductions, Nov. 4-6, 1996, Clarion Plaza Hotel, Orlando, FL

Halverson, Steven L., Wendell E. Burkholder, Timothy S. Bigelow, Erik V. Nordheim and Mark E. Misenheimer 1996c. High-Power microwave radiation as an alternative insect control method for stored products. *Journal of Economic Entomology, Stored-Product Entomology*, 89: 1633-1648

Halverson, Steven L., Rudy Pierre, Wendell E. Burkholder, Timothy S. Bigelow, Mark E. Misenheimer and John H. Boeske 1997a. Recent Advances in the control of insects in stored products with microwaves. Paper No. 976098, ASAE Annual International Meeting, Minneapolis, MN, August 10-14, 1997a

Halverson, Steven L., Rudy Pierre, Timothy S. Bigelow, and Kate Lieber 1997b. Advances in the use of EHF energy as a fumigant for stored products. Paper no. 111, 1997 Annual International Research Conference on Methyl Bromide Alternatives and Emissions Reductions, Nov. 3-5, 1997, Double Tree Hotel, San Diego, CA

Pierre, R., S. L. Halverson, W. E. Burkholder, T. S. Bigelow, M. E. Misenheimer, J. H. Boeske and E.V. Nordheim 1997. Effects of High-Power Microwave Radiation on *Sitophilus zeamais* Motsch. (Coleoptera: Curculionidae) at Different Frequencies. ANPP-Fourth International Conference on Pests in Agriculture, Montpellier, France, 6-8 January 1997.