

**Proceedings of the
22nd International Microwave Power
Symposium Summaries**

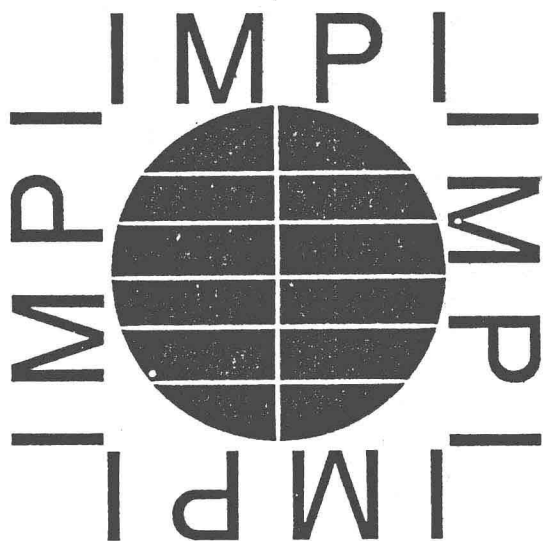
Proceedings of the
22nd Microwave Power Symposium

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PACKAGES INCORPORATING MICROWAVE INTERACTIVE HEATERS

Timothy H. Bohrer

Packaging for microwavable food products, like all packaging, must fulfill the functions of protection, instruction and utility. This paper describes the unique utility that can be provided by packages incorporating interactive heater elements. Application theory and examples of successful use are described.

Packages provide a variety of functions; primary ones include protection utility and motivation. Packages protect the product through storage, shipping and distribution, first to the point of sale and then to the point and time of use. Graphic and structural design and execution provide a vehicle to position the image of the product and attract the purchaser. For the consumer, utility can include opening and reclosure features, special use instructions and the like. For the packer, ease of filling, closing, etc. are utility aspects that are critical specifications elements.

It has become a growing desire for the package to provide additional functional utility to the ultimate user, the consumer. In particular, the ability of the

package to act as a disposable cooking container has grown in importance. Earliest examples of this functionality were the pressed aluminum foil trays used for frozen entrees, dinners and desserts. These products were developed for cooking in hot air ovens and foil containers met the needs of the markets very well indeed.

Proliferation of home microwave ovens, and the concurrent development of high-quality frozen foods with premium price tags, led to a need for more versatile packages. Attributes like dual ovenability, non-arcing, cooking speed, upscale appearance and others began to be described and satisfied by a surprising variety of materials, starting in the mid to late 70's. These approaches satisfied the needs of packagers to supply packages the consumer felt confident in placing in their new microwaves. But their utility was limited to products that had high sauce content, or for which homogeneous texture was acceptable. Products that required browning or crisping were not among offerings that provided microwave cooking instructions.

The fundamental differences between conventional and microwave cooking lay at the root of this roadblock to product development and food product sales. A comparison of the cooking techniques points up the difficulty encountered.

CONVENTIONAL OVEN

- Hot air convection
- Air hotter than food
- Heat conducted from surface
- Conduction differences relatively small
- Surface dehydration before interior
- Surface browning before bulk dehydration

MICROWAVE OVEN

- Polar molecule excitation
- Air cooler than food
- M/W's penetrate food
- Absorption differences relatively large
- Surface and interior dehydrate
- Surface browning after bulk dehydration

Early approaches to deal with the problem of browning/crisping deficiencies included browning dishes, browning elements and combination reci-

pes. These approaches have the following benefits and limits.

Browning Dish

Benefits

- M/W compatibilizes foods
- High thermal mass products possible

Limits

- Preheat time
- Hot to touch
- One side at a time
- Clean up (oven and dish)

Browning Elements

Benefits

- Browns top of food
- Concurrent with M/W cooking

Limits

- One side at a time
- Fire hazard
- Cost

Combination Recipes

Benefits

- Combine both techniques

Limits

- Extra time required
 - Complexity
 - Inconvenient
-

An analysis of these limits led to the following technical needs, and attendant opportunities:

- Size/shape flexibility
- Rapid thermal response
- Low thermal mass
- Ease of cleanup
- Disposability

In response to these opportunities, Qwik Crisp® interactive heating elements were developed. These elements consist of thin layers of controlled thickness metal, vacuum deposited on a heat set plastic carrier film. This film is then laminated to a fiber base to provide rigidity as well as dimensional stability at use temperatures. Laminations to heat resistant polymer sheets for rigidity and stability are also possible.

The elements have a number of interesting and useful attributes. Through resistive heating in the thin metal film, microwave energy is converted into sensible heat. These elements have a rapid heat rise yet level off and self-limit in the highly useful temperature range of 350–400°F. The plastic carrier film acts as a safe food contact surface at cooking temperatures. Due to the typical package fabrication processes employed, these elements are handled most easily in rectangular shapes.

A variety of food package applications have been developed and commercialized using this technology. Examples of pizza, popcorn, potatoes, french bread pizza, garlic bread, waffles, sandwiches and others exist and are shown. Success in utilizing this technology for a specific application requires simultaneous manipulation of food and package properties. The food and package are highly interdependent and close cooperation between the food technologist and the package expert greatly enhances successful system developments.

Examples of food factors that can affect the cooking process are: mass, density, moisture content and distribution, temperature, geometry, thermal conductivity and specific heat. These factors are variables in the optimization equation, along with

package design. By working both parts of the equation, more degrees of freedom are available, and the chances of success are greater. In addition to the examples of success in the market place, other products are under development today.

As we look to the future, the question becomes one of how do we continue to coax higher performance out of the existing package/product combination and how do we leverage the technology to gain even broader application? Three areas of opportunity suggest themselves initially, others exist and will receive development efforts.

The first is more sophistication in food formulations for microwave and interactive package cooking. In particular, better control of moisture distribution in the product through shipping and storage and up to the time of cooking would open a number of product lines to more use with browning and crisping packages.

The ability to economically provide packages with heaters that are temperature tuneable is a continuing challenge for the package developer. Of special interest would be a package in which different areas could be controlled to different temperatures. Finally, the ability to configure current technology into complex shapes and patterns offers the opportunity to broaden the utility of heater packages. Packages of this type are in the early stages of evaluation and commercialization.

In summary, interactive heating elements incorporated into packages for microwaveable foods have catalyzed the development and introduction of new food products previously not considered appropriate for microwave cooking. Additional technology advances in both food and package technology can be expected to further expand the use and success of this packaging advances.

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Heater elements

NEOPENTANE PYROLYSIS ON MICROWAVE IRRADIATED ZEOLITES

G. Roussy, J-M. Thiebaut, M. Anzarmou, C. Richard* and R. Martin

Pyrolysis of neopentane on microwave irradiated 13X-Na-zeolites gives more methane and less isobutene than when the reaction is performed on zeolites heated in a furnace.

When a sample of 13X-Na-zeolites is irradiated by an intense microwave field, the adsorbed molecules and the Na ions which are present inside the solid receive rotational or translational kinetic energy. Because of collisions, energy is then exchanged between the constituents. The system relaxes, but before it reaches its thermodynamic equilibrium, it is possible to carry out gas-solid reactions, under special conditions.

In the homogeneous gas phase the pyrolysis of neopentane gives some methane and isobutene. The stoichiometric equation is



In our experimental conditions, the granular zeolites are placed in a WR 340 rectangular waveguide. From the dc signals given by four detectors placed in front of the sample, the intensity of the electric field inside the sample is known and the position of the short circuit can be adjusted so that the sample is permanently located at a maximum of the electric field of the standing wave distribution. The computer also adjusts the level of the incident power so that the imaginary part of the permittivity of the sample remains constant, during the chemical reaction. ϵ'' being a function of the temperature inside the solid, this procedure is in fact a temperature regulation. The study of the dynamics of the servo control has been discussed and published elsewhere.

Chemical results. More methane is produced when zeolites are irradiated by microwaves than when the zeolites are heated in a classical furnace; consequently less isobutene is obtained.

Typical results, at 500°C, will be presented. No complete description of the reaction mechanisms can presently be given. We can nevertheless suppose that cracking occurs for isobutene on the zeolite surface more easily when the microwaves irradiate the solid than when it is heated classically in a furnace. Such results open up the possibility of modifying many catalytic reactions by irradiating the catalyst with an intense electromagnetic field.

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S
Pyrolysis

USE OF MICROWAVE IRRADIATION COMPARED WITH THERMAL HEATING IN A CHEMICAL COAL CLEANING PROCESS

G.E. Fanslow, C. Hou, C.K. Richardson, D.D. Bluhm and R. Markuszewski

When a chemical cleaning of coal using microwaves for heating is compared with a similar process that uses thermal heating, it is found that microwave processing takes approximately 6 minutes and produces temperatures below 200°C, while thermal heating requires an hour at temperatures above 350°C.

It is known that heating can initiate and/or enhance a chemical reaction. Similarly it is known that electrical excitations, such as ionizations, can initiate and/or enhance chemical reactions. Thus, it is reasonable to assume that the times and temperatures required to produce a given chemical reaction will be different if the material is subjected to a combined thermal and electrical treatment rather than if the material is treated only by heating. The purpose of this paper is to present experimental evidence supporting this assumption. The study compares the reaction times and temperatures required for coal demineralization and desulfurization using coal/caustic mixtures and dielectric heating, *i.e.*, a thermal-electrical treatment, with similar coal cleaning methods using coal/caustic mixtures and only thermal treatments. It shows that thermal-electrical processing takes less time, and the chemical reactions occur at lower temperatures than those required for thermal processing.

The chemical coal cleaning methods that are compared are microwave heating of coal/caustic mixtures [1, 2], and a procedure similar to the TRW Gravimelt process that heats coal/caustic mixtures thermally [3]. The latter process has been the most effective method for removing mineral matter, including pyritic sulfur, and some of the organic sulfur from coal [4].

The coal used for the microwave heating of coal/caustic mixtures was pre-cleaned by a float/sink process, mixed with caustic, moisture was added, and the mixture was heated by microwaves. The thermally heated coal was not pre-cleaned nor was moisture added before heating was begun. After heating both products are successively put through a water wash, an acid wash and another water wash. Optimized results for the microwave heated method were less than 2% ash and less than 0.7% sulfur. The product from the thermally heated coal had 0.56% ash and 0.57% sulfur. Processing parameters are given in the following Table.

| Processing Parameter | Microwave [1, 2] | Thermal [3] |
|------------------------|-----------------------------|-----------------------------|
| Coal | Illinois No. 6 ^a | Illinois No. 6 ^b |
| Sample Size | 50 g | 50 g |
| Processing Time | ~6 min. ^c | ~60 min. ^c |
| Processing Temperature | 150°–200°C | 370°C |
| Input Power | ~1.5 kW | — |
| Caustic/Coal Ratio | 1:1 | 10:1 |
| Particle Size | 28 × 0 mesh | 12 × 0 mesh |

^aRiver King Mine #1 (Peabody Coal Co., St. Clair County Freeburg, Illinois).

^bCaptain Mine (Percy, Illinois).

^c3-Processing cycles of ~2 minutes each.

Discussion. Experimental results show that the combined thermal and electrical excitations of coal/caustic mixtures by microwave (dielectric) heating have a synergistic effect in producing the chemical reactions required for cleaning the coal. It is assumed that, in addition to the chemical reactions associated with material temperatures, there will be additional charge carriers produced in the presence of the electric fields. These charge carriers, principally electrons, can be accelerated and they, in turn, can produce additional ionizations through impact. Because it is possible for these ionizations to be produced with little or no increase in material temperature, increased chemical activity occurs and reactions can take place more rapidly at lower temperatures than would be required if the only form of excitation were temperature alone.

Additional information will be presented on the possible correlation of the increased chemical ac-

tivity and the increase in the conductivity of the coal/caustic mixture during heating.

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Coal

A VERSATILE MICROWAVE PLASMA APPLICATOR

Ji Tian Ren and J.E. Gerling*

A review of the design and performance of a microwave applicator designed to introduce high levels of microwave power into a plasma chamber over a large area with good electric field uniformity.

In the last ten years, research scientists have been attracted to microwave excited plasmas because of certain advantages over dc and low frequency plasmas. Some of the advantages of microwave excited plasma can be summarized as follows:

1. It produces a much higher degree of ionization and disassociation that gives commonly 10 times higher yield of active species than other types of electrical excited plasma.
2. It is possible to sustain a microwave plasma over a very wide pressure range, from 10^{-5} torr to several atmospheres, giving more possibilities to plasma application than most other forms of electrical stimulated plasma.
3. The electron-to-gas temperature ratio T_e/T_g is very high so the carrier gas and undertreated substrate remains moderately cool but with the very high electron energy. Many reactions can

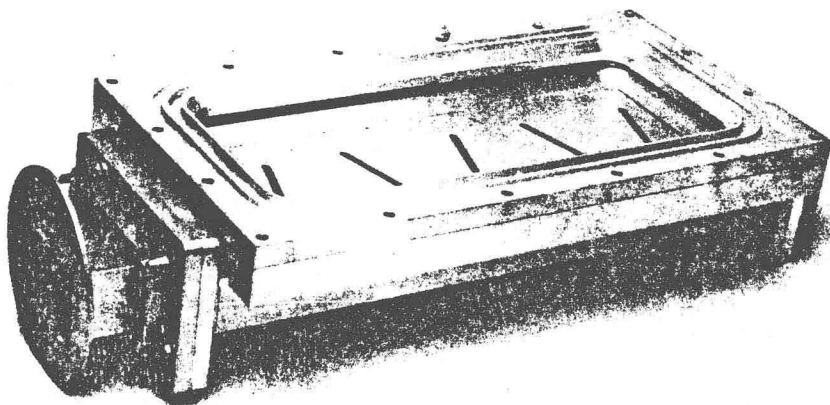


Figure 1. Applicator design.

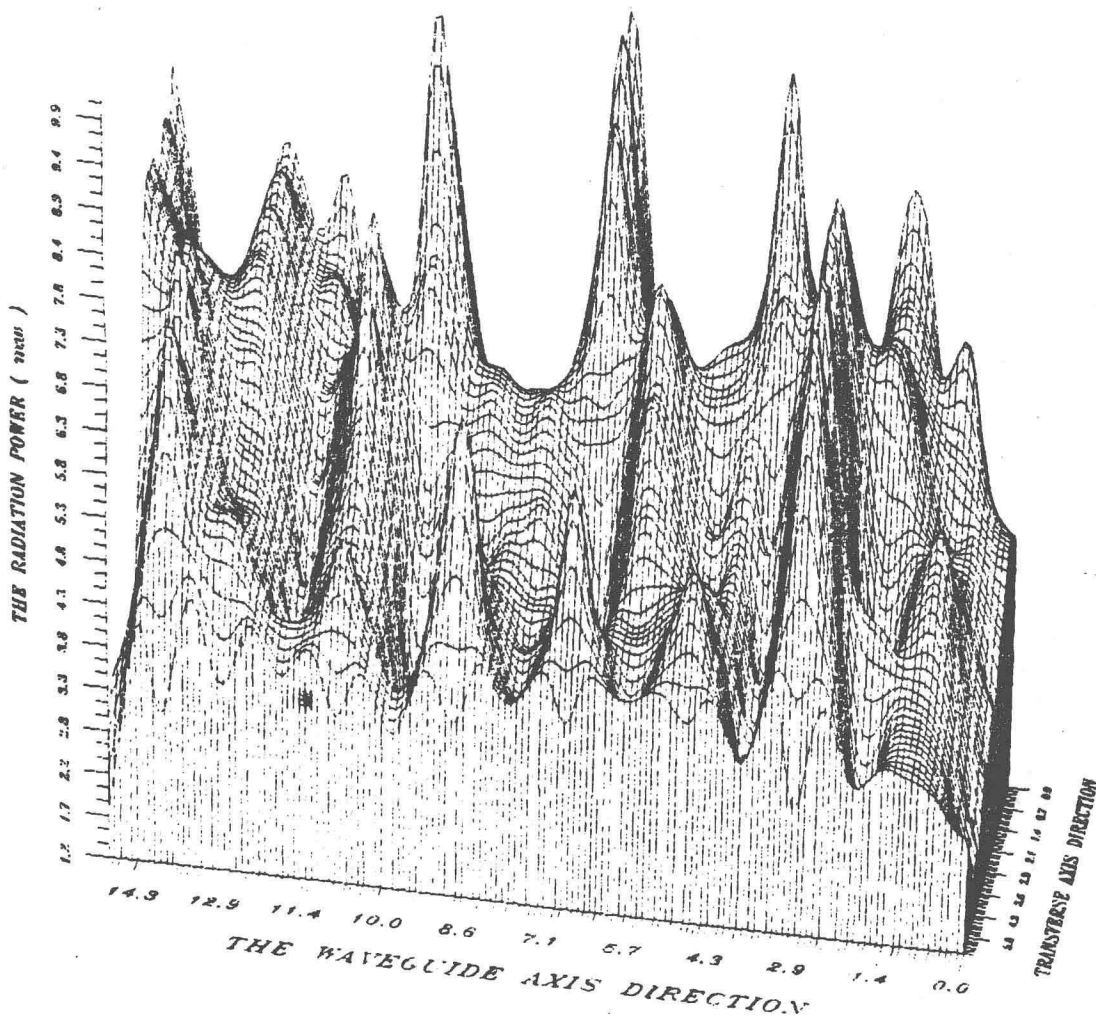


Figure 2. Three dimensional representation of the free space energy distribution.

take place in the process with a relatively low ion energy. The effects of sputtering or decomposing by the incident ions on the treated surface can be decreased.

4. The absence of internal electrodes removes a source of contamination and makes reaction vessels simpler.
5. The state of the art of microwave engineering at high power levels is relatively far advanced.

Promising applications of the microwave plasma have been appearing in the fields of chemical processes and semiconduction manufacturing. Applications include processing the surface of an object with nitriding, carbonization etching or other surface-processing; to promote organic reactions, to improve synthetic polymers; to use the microwave plasma as new light sources, for pumping lasers, generating plasma torches *etc.*

The paper describes a general purpose microwave plasma device which we have developed to introduce microwave energy into a plasma chamber over a wide area with high transfer efficiency and good energy uniformity to permit researchers to study processes on a larger scale than heretofore possible.

A slotted rectangular waveguide combined with a quartz window was chosen as the basis of the applicator design. Based on the theory given by Oliner [1] and in [2] we calculated the impedance properties of several different kinds of slots in the broad face of rectangular waveguide WR430. In order to evaluate the impact of the quartz window on the design, a quantity of cold tests and low power simulating tests were conducted to heuristically determine the optimum dimensions and geometry. We designed a simple low power testing system to measure the three dimensional spatial energy distribution of the applicator in free space. With the aid of a computer program three dimensional graphics and pattern contours were plotted. Figure 1 is a photograph of the final design; Figure 2 gives a dimensional representation of the free space energy distribution.

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A COMPREHENSIVE RESEARCH PROGRAM FOR DIELECTRICALLY-ENHANCED DRYING

P.S. Schmidt and A. Karp*

This paper describes the approach, objectives, and long-range research plans of a program at the University of Texas at Austin for the study of dielectrically-enhanced drying. The program includes flexible test facilities for drying experiments in both microwave and rf regimes, dielectric and thermal property measurements, and mathematical modeling of material behavior and dryer design.

Drying is one of the most common and energy-intensive unit operations in industry, and frequently is the rate-limiting step in the overall production cycle. Dielectric heating has been shown by many investigators to significantly enhance drying rates, as well as providing other potential benefits such as reduced thermal degradation of the material.

A program has been established at the University of Texas at Austin, under the sponsorship of the Electric Power Research Institute, to investigate drying phenomena under combined dielectric heating and convection. The objective of the program is to contribute to a better understanding of dielectrically-enhanced drying as part of EPRI's overall effort to promote development of high-productivity industrial electrotechnologies.

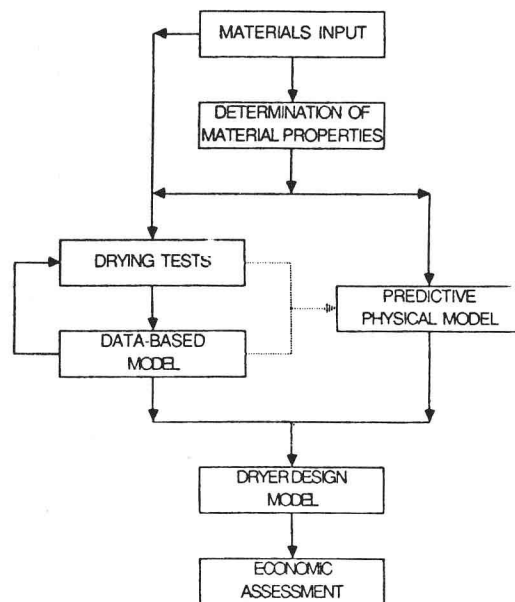


Figure 1. Evaluation process of dielectrically enhanced drying of industrial materials.

Analytical modeling efforts include statistical methods for reducing test data to the form of characteristic drying curves, finite-element modeling of basic heat and mass transfer in porous media, and drying equipment design/economic assessment

Major elements of the program are illustrated in Figure 1. Experimental facilities built or under construction include a 6 kW microwave test system and a 10 kW rf system. Both facilities incorporate air handlers with the capability of closely controlling air velocity and temperature over the sample. Instrumentation is provided for continuous measurement of forward and reflected power, air temperature and velocity, ambient humidity, and sample mass. Sample internal temperatures are determined at up to eight points using fluoroptic probes, and internal pressures can be measured at up to four points. A computer data acquisition and

control system logs all data and software is being developed for real-time display of drying data and system control. In addition to these facilities, two bench-top rigs have been constructed for small-sample tests. Dielectric property measurements in the microwave regime are being made using the open-ended coaxial line technique; other apparatus is planned for measurements at lower frequencies. models. The overall strategy for coordinating the modeling and experimental aspects of the program are discussed, as well as efforts to coordinate these activities with other investigations.

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| *Electric Power Research Institute | Enhanced drying |

Medical Applications in Microwave Energy

Session Chairman:
Dr. Carl Sutton
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USE OF NON PERTURBING THERMOCOUPLES (NPT's) FOR HYPERTHERMIA CLINICAL THERMOMETRY

H.I. Bicher, J. Shani and K. Reesman

Metallic Thermocouples readings are easily disturbed in strong microwave fields. Probes are described that use the thermocouple principle but are constructed of non metallic leads made of 2 different carbon carrying plastic materials pressured together to produce effective and reproducible junctions. These sensors can be easily adapted to available computer interfaces, have a linear output between 20°C to 50°C, are easy to calibrate with a reproducible output of 20-40 microvolts per degree centigrade. The probes have been miniaturized and used in phantoms as well as animals and human hyperthermia treatments.*

| | |
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| Valley Cancer Institute, Panorama City, CA | 1240 M |
| *Equipment supplied by the HBCI Medical Group, Inc. Patent pending. | TM Thermocouples |

A THREE APPLICATOR MICROWAVE PHASED ARRAY SYSTEM (TRIPAS) FEATURING MATHEMATICAL PREDICTION AND RELOCATION OF THE HYPERTHERMIA CLINICAL TARGET "HOT SPOT"

H.I. Bicher and S. Afuwape

The problem of heat focusing at body depth in the clinical application of hyperthermia requires a mathematical predictive solution that affords standardization and includes in its parameters the actual permittivities and conductivities of the treated tissues as well as dictating the actual positioning of the treating applicators to allow reproducible optimization of treatment planning. We are presenting a tentative configuration of three applicators encircled and subtended by an equilateral triangle in order to target and relocate a "hot spot" for achieving significant tumoricidal heat level at depth. A prototype clinically applicable "TRIPAS" system has been built. Results of phantom experiments correlate well with computer model predictions.

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| Valley Cancer Institute, Panorama City, CA | 1241 M |
| Equipment: HBCI Medical Group, Inc. - Patent pending | DE Hyperthermia |

MICROWAVE VACUUM DRYING IN PRETREATMENT STEP OF ACID HYDROLYSIS

G. Lightsey and G. Bharat

Microwave vacuum drying of lignocellulosic residues was attempted in order to 1) Increase drying rates, 2) Reduce drying temperatures, 3) Reduce acid consumption in pre-treatment step and 4) Improve conversion of crystalline cellulose to its amorphous state, theoretically, giving quantitative yields of fermentable glucose upon hydrolysis.

Cornstover is one of the most abundant agricultural residues available in the United States. Total cellulose and hemicellulose content of cornstover is 50–60% providing a relatively good feed stock for acid hydrolysis of these celluloses to fermentable sugars (Figure 1). Hemicellulose is easily hydrolyzed but Cellulose must be converted to the amorphous state before it can be hydrolyzed. The crystallinity of cellulose is broken down in a pre-treatment step by impregnating the solids with acid and then drying the solids to concentrate the sulphuric acid from 15–75% (Wt%). Since cellulose is degraded in acid at high temperatures, drying of

the stover has to be done at temperatures below 80°C (175°F) and should be accomplished as rapidly as possible. If these conditions are not maintained then cellulose tends to rapidly degrade at higher acid concentrations.

The drying rates of acid impregnated cornstover were compared using microwave heating at one atmosphere, microwave heating in 20 in. Hg vacuum and conventional vacuum oven at 80°C and 20 in. Hg vacuum. Comparison of results obtained showed that microwave vacuum drying was the most efficient method of drying in terms of length of time required to reach high acid concentrations and the amount consumed.

The effectiveness of microwave-assisted vacuum drying is a result of the low heat transfer coefficient of cornstover. In a conventional vacuum dryer, heat must be transferred primarily by conduction from the dryer walls to the fibrous cornstover solids. In contrast, microwave energy is absorbed directly by water molecules in the cornstover.

Microwave heating equipment is more expensive than conventional heating equipment. However, microwave assisted-vacuum drying of cornstover appears to be more economical than the conven-

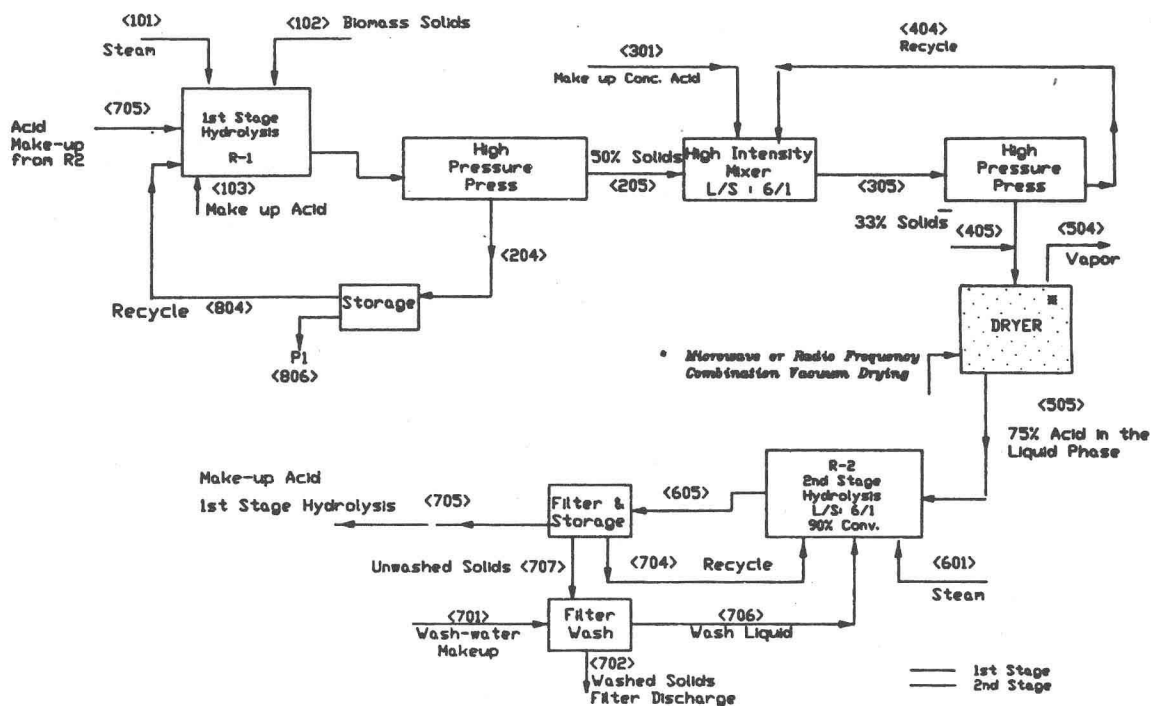


Figure 1. First stage acid recycle and second stage direct acid addition with conc. acid recycle.

tional vacuum drying. The use of microwave energy for vacuum drying of biomass material deserves further study.

- 1 Smith, F.J. (1979). Microwave hot air drying. The Microwave Newsletter, XII(6).
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- 3 Smith, R.D. (1984). *Microwave Power in Industry*, Thermo Energy Corporation, California.

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APPLICATIONS OF MICROWAVE ENERGY IN EXTRACTIVE METALLURGY

S.L. McGill and J.W. Walkiewicz

Minerals and reagent-grade compounds were tested to determine their receptivity to microwave heating. Descriptions of equipment and methods, and temperature data are included. Extractive applications were investigated and stress fracturing along mineral grain boundaries in a gangue matrix was observed.

Research done by the Bureau of Mines has shown that valuable minerals, such as galena, ilmenite, magnetite, and chalcopyrite, are heated by microwaves, whereas common host rock minerals, such as quartz, calcite, and feldspar, are not heated. This selective heating by microwaves has potential extractive metallurgy applications.

Experiments were conducted to collect heating data, investigate stress fracturing, and determine possible applications utilizing microwave heating. Except for the fracturing studies which were run at 3 kW, microwave heating data were obtained with a 1-kW, 2,450 MHz commercial oven with a vented 1,000 mL water load. Twenty-five-gram samples or a constant volume of 18 mL for low-density materials were irradiated in a closed system under an inert atmosphere. Each sample was placed in a 20-mL alumina cylindrical crucible and enclosed in a 250-mL Pyrex beaker. The beaker was corked with a microwave-transparent gum rubber stopper which had been fitted for thermocouple insertion. The thermocouple was type K with an ungrounded tip sheathed in Inconel 702 (for sulfide samples the sheath was Stainless 440).

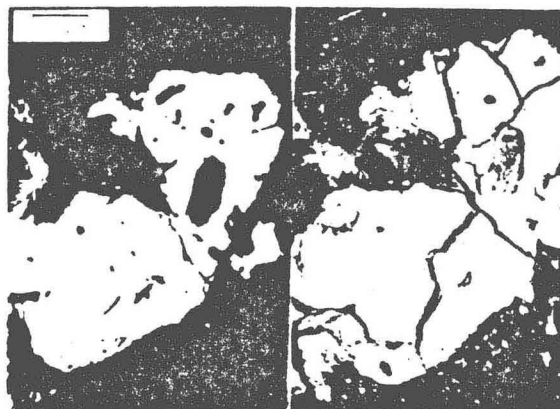


Figure 1(a). Non-microwaved pyrite ore. Light phase is pyrite, dark phase is quartz matrix, $\times 100$. (b) Microwave-heated pyrite ore showing stress cracking along and across grain boundaries, $\times 100$.

The microwave heating characteristics of a variety of reagent-grade elements, compounds, and high-purity natural minerals are categorized in Tables 1 and 2. All solid samples were powders; some had particles as large as 60 mesh and some were minus-325 mesh. Three liquids, Hg, SnCl_4 , and TiCl_4 , were tested. The indicated temperature was the maximum obtained and the time was the interval to attain maximum temperature from ambient temperature ($20\text{--}28^\circ\text{C}$). For high heating samples, the test was terminated when the temperature approached $1,100^\circ\text{C}$ to prevent melting of the thermocouple. The time value is included so that a comparison of heating rates can be made. The highest temperatures were obtained with the metal oxides NiO , MnO_2 , Fe_3O_4 , Co_2O_3 , CuO , and WO_3 . Most metal sulfides heated well, but no consistent pattern was observed. A number of other samples exhibited an extremely rapid rate of heating. A carbon sample heated from an ambient temperature of 29°C to $1,170^\circ\text{C}$ during the first 30 s or 38.0°C/s and to $1,283^\circ\text{C}$ after 1 min. Although metals reflect microwaves, this was not the case with metal powders, which heated well. Metal powder heating is ascribed to inductive effects. Except for CuCl , SnCl_2 , and ZnCl_2 , the anhydrous chlorides tested did not heat above 112°C . Common gangue minerals, quartz, calcite (marble), and feldspars (albite and orthoclase), did not heat.

Since microwaves selectively heated some minerals but not common gangue minerals, selective heating of ore minerals within the host rock was

Table 1. Effect of microwave heating on the temperature of reagent grade elements and compounds^a.

| Chemical | Temp, °C | Time, min | Chemical | Temp, °C | Time, min |
|--|----------|-----------|--------------------|----------|-----------|
| Al | 577 | 6 | Mo | 660 | 4 |
| AlCl ₃ | 41 | 4 | MoS ₃ | 1,106 | 7 |
| C | 1,283 | 1 | NaCl | 83 | 7 |
| CaCl ₂ | 32 | 1.75 | Nb | 358 | 6 |
| Co | 697 | 3 | NH ₄ Cl | 31 | 3.5 |
| Co ₂ O ₃ | 1,290 | 3 | Ni | 384 | 1 |
| CoS | 158 | 7 | NiCl ₂ | 51 | 2.75 |
| Cu | 228 | 7 | NiO | 1,305 | 6.25 |
| CuCl | 619 | 13 | NiS | 251 | 7 |
| CuCl ₂ ·2H ₂ O | 171 | 2.75 | Pb | 277 | 7 |
| CuO | 1,012 | 6.25 | PbCl ₂ | 51 | 2 |
| CuS | 440 | 4.75 | S | 163 | 6 |
| Fe | 768 | 7 | Sb | 390 | 1 |
| FeCl ₂ | 33 | 1.5 | SbCl ₃ | 224 | 1.75 |
| FeCl ₃ | 41 | 4 | Sn | 297 | 6 |
| FeCl ₃ ·6H ₂ O | 220 | 4.5 | SnCl ₂ | 476 | 2 |
| Fe ₂ O ₃ | 134 | 7 | SnCl ₄ | 49 | 8 |
| Fe ₂ (SO ₄) ₃ ·9H ₂ O | 154 | 6 | Ta | 177 | 7 |
| Hg | 40 | 6 | TiCl ₄ | 31 | 4 |
| HgCl ₂ | 112 | 7 | V | 557 | 1 |
| HgS | 105 | 7 | YCl ₃ | 40 | 1.75 |
| KCl | 31 | 1 | W | 690 | 6.25 |
| Mg | 120 | 7 | WO ₃ | 1,270 | 6 |
| MgCl ₂ ·6H ₂ O | 254 | 4 | Zn | 581 | 3 |
| MnCl ₂ | 53 | 1.75 | ZnCl ₂ | 609 | 7 |
| MnO ₂ | 1,287 | 6 | Zr | 462 | 6 |
| MnSO ₄ ·H ₂ O | 47 | 5 | | | |

^aMaximum temperature obtained in the indicated time interval.

Table 2. Effect of microwave heating on the temperature of natural minerals^{a,b}.

| Mineral | Chemical composition | Temp, °C | Time, min |
|--------------|---|----------|-----------|
| Albite | NaAlSi ₃ O ₈ | 82 | 7. |
| Arizonite | Fe ₂ O ₃ ·3TiO ₂ | 290 | 10. |
| Chalcocite | Cu ₂ S | 746 | 7. |
| Chalcopyrite | CuFeS ₂ | 920 | 1. |
| Chromite | FeCr ₂ O ₄ | 155 | 7. |
| Cinnabar | HgS | 144 | 8. |
| Galena | PbS | 956 | 7. |
| Hematite | Fe ₂ O ₃ | 182 | 7. |
| Magnetite | Fe ₃ O ₄ | 1,258 | 2.75 |
| Marble | CaCO ₃ | 74 | 4.25 |
| Molybdenite | MoS ₂ | 192 | 7. |
| Orpiment | As ₂ S ₃ | 92 | 4.5 |
| Orthoclase | KAlSi ₃ O ₈ | 67 | 7. |
| Pyrite | FeS ₂ | 1,019 | 6.75 |
| Pyrrhotite | Fe _{1-x} S | 886 | 1.75 |
| Quartz | SiO ₂ | 79 | 7. |
| Sphalerite | ZnS | 87 | 7. |
| Tetrahedrite | Cu ₁₂ Sb ₄ S ₁₃ | 151 | 7. |
| Zircon | ZrSiO ₄ | 52 | 7. |

^aMaximum temperature obtained in the indicated time interval.

^bHigh purity as identified by X-ray diffraction.

possible. Rapid heating of ore minerals in a microwave-transparent matrix generates thermal stresses of sufficient magnitude to create microcracks at mineral grain boundaries. Inducement of microcracking before beneficiation or extraction will improve and simplify crushing, grinding, and concentrating operations for ores. Stress-fracturing studies were conducted on different types of metal oxide and sulfide ores. Scanning electron microscope examination before and after microwave heating showed that thermal stress fracturing was induced in the samples. The specimen shown in Figure 1A is chalcopyrite-pyrite ore from Bingham Canyon, UT. The light phase is pyrite, and the dark phase is a fine-grained quartz gangue. Stress-induced cracks at the grain boundaries and across the grains are shown in Figure 1B. These effects should decrease grinding energy requirements, improve the liberation of valuable minerals, and lead to better metal recoveries.

The addition of a good microwave absorber, such as magnetite, to a feed which is transparent to microwaves was attempted to broaden the range of potential applications for dielectric heating of ores

and concentrates. This technique was feasible for extracting mercury from a cinnabar concentrate and converting pyrite to magnetic pyrrhotite.

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A CIRCULAR CYLINDRICAL APPLICATOR FOR ENHANCED HIGH TEMPERATURE PROCESSING

M.E. Brodwin, D.L. Johnson*, Y-L. Tian* and K-Y. Tiu**

A circular cylindrical cavity is described with mirror smooth silver coated walls for high temperature sintering of ceramics. The walls reflect thermal energy back to the sample to achieve 2000°C with 650 W.

Microwave thermal processing has recently been applied to the sintering of ceramics and it has been found that not only is the process extremely energy conservative, but also produces improved properties arising from finer grain size. Prior applicators, based upon rectangular waveguide designs, achieved temperatures of 1500°C with less than 500 W of available power. For higher temperatures, it was found that the principal heat loss was due to radiation and that significant increases in power were required to produce relatively small increases in temperature. This result led to the investigation of structures that would return radiated power back to the sample and thereby improve thermal efficiency.

In this talk, we describe a circular cylindrical applicator with mirror smooth silver coated reflecting walls operating in the TE_{11n} mode. The sample is an axially located ceramic rod translated and rotated through the applicator. The applicator is waveguide coupled with a replaceable iris and with adjustable high power end walls for tuning and coupling variation. In addition, it is pressurized to raise the breakdown electric field to increase absorbed power.

The choice of the TE modes rather than the more common TM modes was dictated by the lack of knowledge of the high temperature electrical characteristics of the sample. Thus it was necessary to have an easily tunable cavity in contrast to the lowest order TM mode. Furthermore, to prevent possible mode confusion, it was desirable to choose a mode developed from the dominant propagating mode. The disadvantage of the TE

modes is that the field, being transverse, was more likely to produce non-uniform heating in addition to requiring the solution of a more complex boundary value problem for determining the complex permittivity.

Our prior work also showed that it was essential to critically couple the applicator to ensure heating of room temperature high resistivity materials. Coupling adjustment in this design is provided by moving the endwalls symmetrically with respect to the iris. Asymmetric adjustment controls the resonant length. For the $n = 1$ mode, the coupling can be varied from overcoupled through critical to undercoupled as the iris is displaced from the central position.

A major problem is the development of adjustable non-contacting end walls capable of operating with large available power under critical coupling conditions without arcing. The successful design is based upon the familiar dumbbell device with a unique stepped feature to prevent possible arcing.

Measurements were made to evaluate the design. These consisted of Q and coupling measurements at room temperature with and without a SiC sample for the as-machined cavity. The same measurements were repeated for a mirror smooth silver coated cavity at room temperature and with a warmed sample. The Q measurements showed that the endwall design did not appreciably add to the losses and that the expectations of adjustable coupling and tuning were verified.

Optical pyrometer measurements of surface temperature were performed as a function of absorbed power and the results showed the increase in temperature of the sample with silvered walls in contrast to the sample temperature with the as-machined walls. The pressurized applicator enabled us to obtain surface temperatures up to 2000°C with an absorbed power of 650 W.

Other advantages of the thermally reflecting wall design are improved temperature uniformity, faster heating rates, and higher temperatures at the onset of plasma formation.

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ELECTROMAGNETIC ENERGY FOR DRYING RUBBER FOAM

W. Van Look

High frequencies and microwaves can be applied in the production of rubber foam for foaming, curing and drying.

An investigation has revealed that high frequency drying of the final 20% moisture content is feasible on an industrial scale.

In industry various materials are foamed to obtain a porous or sponge-like product. These processes have several distinct phases: foaming, curing, drying and washing for removing chemicals and for cleaning which requires subsequent drying. Large scale hot air or steam heaters are common for the fabrication processes.

Electromagnetic energy is capable of performing all phases for production of rubber foam. However microwave experiments indicate that precise control of the applied energy is required for foaming and curing. The processes are very sensitive to hot spots degrading the quality of the product.

Hot-air drying curves and calculations indicate negligible capillary forces and the presence of only free water in the material. The energy requirement is 3 to 4 kWh/kg evaporated water for the final conventional drying to about zero moisture con-

tent. Measurements on existing dryers reveal up to 10 kWh/kg water.

Experimental procedures were used to investigate the application of microwaves and high frequencies for the removal of water and comparing drying times, product quality and energy consumption to the conventional final drying. In the analyses of the process requirements and the economics, mechanical water extraction has been considered. The overall energy needed for this type of drying can be as high as 1 kWh/kg water at only a small percentage of the investment cost for a high frequency dryer. The compromise has been mechanical water removal down to 20% and final high frequency drying. The measured overall electricity use is 2.3 kWh/kg removed water.

Several 100 kW high frequency units combined with hydro extraction are now being used in these industrial processes [1].

- 1 Van Look, V.M. (1987). Les recherches en Belgique sur les applications industrielles des hautes fréquences et des micro-ondes. Maîtrise de l'Energie dans l'Industrie, 2ième Salon Professionnel, Paris, France, (3 avril).

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Foam rubber

Radio Frequency Heating Applications

Session Chairman:

John Zimmerley
PSC Inc.

PLASMA PROCESSING FOR MICROELECTRONIC APPLICATIONS USING MICROWAVE EXCITATION

J. Paraszezak, J. Heidenreich, M. Hatzakis and M. Moisan*

Considerable interest has recently been aroused in the use of microwave excited plasmas for microelectronic processing. This paper will discuss the advantages of this technique and will report on recent results obtained in our laboratory.

For the last two decades, rf (13.56 MHz) plasmas have been successfully used to etch, roughen and deposit a wide variety of materials in the semiconductor industry. These materials include silicon, its oxides, metals and polymers. In the last 5 years, several advantages of exciting these plasmas using higher (microwave) frequencies have been discovered. These include (1) A reduction of damage due to ion bombardment, (2) In-

creased throughput, due to higher available rates of etching and deposition and (3) The ability to change the cross-sectional profiles of etched materials by varying electrical parameters in these plasmas. Although this is an emerging technology, several commercially available tools for etching have recently become available. This paper will discuss the characteristics of these plasmas in comparison to the lower frequency (rf) plasmas, methods employed to generate these plasmas, and recent results in using these plasmas, in particular with reference to the etching of polymers and silicon.

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RADIO FREQUENCY REDRYING OF VENEER— A CASE STUDY

T.W. Gault and J.B. Wilson*

Radio frequency redrying of veneer offers an alternative to present practices of redrying veneer in a conventional primary dryer. In order to establish its commercial viability, PP&L and OSU Forest Research Laboratory co-sponsored a demonstration study of a commercial rf redryer.

Radio frequency (rf) redrying of veneer offers an attractive alternative to present practices of redrying veneer in conventional primary dryers. To assess the commercial viability of the technology, a comprehensive study was conducted of a rf oven that was being installed at a Boise Cascade plywood mill in Yakima, Washington. The study encompassed the operation and economics of the rf oven as well as how to integrate its operation to other work stations in the mill. The study was co-sponsored by the Forest Research Laboratory of Oregon State University and Pacific Power & Light Company.

When considering the installation of a rf oven for redrying veneer, there are several significant features that make it attractive. These features include: a small floor space requirement of 18 by 40 feet (which include the inlet and outlet conveyors), no labor is required, and there is no down grading loss of veneer that typically occurs in conventional redrying practices.

The operation of the rf oven basically consists of loading a bundle of veneer (4 × 8 ft. sheets) stacked 25 to 29 in. high onto the inlet conveyor. The bundle is conveyed into the oven, the doors close and a metal platen that acts as a capacitor plate lowers onto the bundle. The oven then provides a quickly changing electric charge on the plates at a frequency of 14 MHz which causes the water molecules in the veneer to vibrate until sufficient heat is generated to evaporate the moisture in the veneer. The bundle continues to dry until the oven senses that a sufficient amount of water has been removed. At that point the doors open and the dried bundle is conveyed out of the oven.

Late in 1985, Boise Cascade installed a new 300 kW oven manufactured by SPECO of Beverly, Massachusetts to redry 135 MSF (3/8 basis) of veneer per day. The veneer consisted of 75% douglas fir and 25% white fir. The plant was short of drying capacity at the time of installation. As a result of installing the rf oven and other associated changes, Boise Cascade was able to increase the plywood production by 24% of 525 to 650 MSF per day.

The investment by Boise Cascade in the rf oven was \$900,000 for equipment and \$100,000 for the building to house the equipment and the additional warehouse floor space needed to store the veneer for cool down after drying. The simple payback on this investment was approximately one and a half years. The savings of using an rf oven are due primarily to the elimination of veneer losses during conventional redrying and to reduced drying costs in the primary dryers. At a 15% redry rate, veneer losses amount to 1.5 to 3.75% of the total plant production. When redrying with a rf oven there was no loss or downgrading, which results in a significant savings in raw materials. In addition, the primary dryer cost was reduced from the industry average of \$29/MSF to \$24.32/MSF with the use of rf redrying.

In addition to the investment costs of \$1 M for the equipment and building, costs for redrying the veneer in the rf oven included \$2.55 per MSF (3/8-inch bases) for electrical power, \$0.58 per MSF for parts replacement, and \$0.18 per MSF for maintenance expenses.

The rf oven was able to redry three to four bundles of veneer per hour. The average moisture content of the veneer entering the oven was approximately 7 to 10% with a targeted exit moisture content at about 4.5%. The rf oven not only removed moisture from the bundle but it also did an excellent job of redistributing moisture from the wet to the dry portion of the veneer, resulting in a very uniform moisture distribution. During the drying process the bundles rose to temperatures of 250 to 300°F, which meant they had to cool before being used in layup. It took approximately four days for a bundle to cool to the desired maximum temperature of 100 to 110°F for lay up.

To obtain the optimum use of the rf oven it was recommended that the redry rate of the primary dryers at the mill be increased to 35% for douglas fir sap, 20% for douglas fir heart and 50% for white fir. By increasing the redry rate for the primary dryers and by redrying in the rf oven the total dried veneer production was increased by 24%. Overall, the rf redrying of veneer in a plywood operation appears to be a very attractive proposition resulting in significant savings from reduced operating costs. With a simple payback period of approximately one and a half years for the installation of a rf oven, this method deserves serious consideration when additional drying capacity is needed in a plywood plant. With the increased application of technology for recovery on the green end of the plywood mill operation, most plants are dryer-limited and could benefit significantly from the installation of a rf oven.

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RADIO FREQUENCY DRYING OF TEXTILE YARN IN THE SIZING PROCESS

Morton W. Reed and Warren S. Perkins

Use of radio frequency energy for drying of textile yarns in sizing, a continuous process wherein a sheet of yarns is coated with a film-forming chemical, will be discussed. The research is focused on quality of yarn produced by radio frequency drying as compared to yarn dried on steam-heated cylinders, the conventional method for drying yarn in the sizing process.

An investigation of the use of radio frequency (rf) energy for drying of textile yarns in sizing, a continuous process wherein a sheet of parallel yarns is coated with a film-forming chemical substance, has been underway at Auburn University for about one year. The objectives of the work are to determine the optimum conditions for drying yarn using rf energy either alone or in combination with conventional drying techniques, to determine the effect of rf drying on quality of yarn produced, and to evaluate costs of using rf energy for yarn drying. Results of initial experiments using rf energy on a laboratory scale yarn sizing machine have been positive and will be discussed in the presentation.

The purpose of sizing is to enhance physical properties of the yarn by coating the yarn with a protective material which will improve the performance of yarn in the weaving process. Since high efficiency in the weaving process is vital to the economics of textile fabric manufacturing, the research is focused on quality of yarn produced by radio frequency drying as compared to yarn dried on steam-heated cylinders, the conventional method for drying yarn in the sizing process.

Many possible benefits can be visualized with the use of radio frequency drying in the sizing process. Improved control of moisture content of sized yarn should result because of the absence of thermal lag associated with cylinder drying. Radio frequency should also be somewhat self-limiting so as to not overdry or overheat especially with mate-

rials of low loss factor such as polyester. The ability to instantly turn the power off and on should be a boon to processes such as sizing which inherently require frequent machine stops for various lengths of time. Drying rate might also be better using rf because it should eliminate the "skinning" effect which inhibits moisture removal during conventional drying.

A radio frequency dryer has been added to a laboratory sizing machine. The rf drying is of the strayfield type and operates at frequency of about 22 MHz. The rf power supply has a rating of 12.5 kW. The sizing machine is capable of speeds up to 42 yards per minute and has radiant and conduction drying capabilities in addition to rf. Models which allow determination of drying rates have been developed. Power consumption by the rf dryer is being measured for comparison to power requirements using conventional cylinder drying techniques. A dynamic model for operational control has been developed and used in current control studies.

Another useful feature of the dryer for research purposes is the use of screen wire and plastic covers on two sides of the dryer to allow visual observation of the yarn in the dryer. The moisture removed from the yarn is vented from the dryer cavity by fans to prevent condensation of moisture on the electrode system.

Yarn properties that are important in order for sized yarn to perform well in weaving include abrasion resistance, hairiness (fuzziness), strength and elasticity. The relationship of these yarn properties to the drying method employed is being studied and will be reported in the paper.

While the project concentrates specifically on the sizing process, the results produced will have implications regarding the use of radio frequency energy for heating and drying in other textile processes such as dyeing and application of chemical finishes.

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The work is joint venture with the
Tennessee Valley Authority (TVA)
and **West Point Foundry and Machine
Company, Inc.** as part of the TVA's
Electrotechnology Development Program.
Significant contributions were made by
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Textiles

**THE IEC PERFORMANCE MEASUREMENT STANDARD
FOR MICROWAVE OVENS—BACKGROUND AND
RELEVANCE**

Per O. Risman* and Thomas Ohlsson**

The International Electrotechnical Commission (IEC) will soon publish a new performance measurement standard. The work is described and analyzed, as is the current continuing

work. The microwave power output measurement as a problem of method and relevance is also dealt with.

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**A FIBER OPTIC REFRACTOMETER FOR SPECIES
CONCENTRATION MEASUREMENT IN A
MICROWAVE ENVIRONMENT**

T.L. Bergman

A miniature fiber optic refractometer has been developed and used to measure the species concentration of a fluid which is heated in a microwave oven. The probe, which measures the local fluid temperature and refractive index, is easily constructed using a commercially available fiber optic thermometer and standard laboratory equipment.

Recently, interest in microwave processing of various industrial materials and foodstuffs has developed, for example [1]. It is well known that if the process involves drying, moisture removal will subsequently affect the rate of microwave absorption and, hence, the thermal response of the material.

If the drying process involves a product containing a binary fluid (for example, brine) or if a chemical reaction occurs, the situation is further complicated by temporal and/or spatial variations in the species concentration of the sample, which can subsequently affect microwave absorption rates [2]. It has also been suggested that spatially selective microwave or rf heating can be achieved by controlling the spatial distribution of a species in the sample [3].

In order to understand the interaction between the samples' thermal, species, and electric fields, it is necessary to develop instrumentation capable of measuring local temperatures and species concentrations. To this end, it is useful to be cognizant of the considerable progress which has recently been made in utilizing fiber optic refractometry to monitor species distributions in binary fluids [4, 5].

With this method, light losses from the optical fiber to the surrounding fluid may be related to the refractive index of the fluid. Since the fluid's refractive index will generally vary with its temperature and species concentration, an independent temperature measurement must be made [4]. This companion measurement can be obtained in a microwave environment using commercially available fiber optic thermometry probes [6].

The fiber optic refractometer concept, coupled with fiber optic thermometry, was used in this study. A 0.4 mm-diam fiber, consisting of a 0.372 mm-diam polystyrene core with 0.014 mm thick methyl-methacrylate cladding and rubber coating, was inserted into a 3 mm inside diam laboratory glass support tube as shown in Figure 1a. The refractometer probe materials were chosen to minimize microwave absorption. The cladding and coating were selectively removed from the bent fiber, allowing intimate contact between the optical fiber and the binary fluid. A Luxtron MIA thermal probe was also inserted into the glass tube and was used to measure the fluid temperature. A nearly constant intensity light source (Spectra-Physics 105P 5 mW He-Ne laser) illuminated the fiber, and the light loss at the sensing tip was determined by measuring the intensity of laser light transmitted through the optical fiber with a photodiode (Newport 815, 816).

Prior to its use in a microwave (2450 MHz) environment, the probe was calibrated by placing it in samples of NaCl/water solutions of known refractive index. The result is shown in Figure 1b. Note that, since light loss (and hence the output voltage of the photodiode I/V transformer) depends on both the refractive index of the polystyrene core and the salt water, and since the fiber refractive index, n_f , varies with temperature, the