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Upcoming Conferences

The 33rd IEEE International Conference on Plasma Science (ICOPS) Traverse City, MI June 4-8, 2006

For more information:
www.icops2006.org
info@icops2006.org

The 40th Annual IMPI Microwave Symposium will be held at the Hotel Sonesta, Boston, MA August 9-11, 2006
For more information contact:
www.impi.org

Highlights from Mumbai - Microwaves - A Promising Option for Tomorrow

THE INDO-US WORKSHOP ON MICROWAVE TECHNOLOGY FOR MATERIALS PROCESSING

By: Bernie Krieger
Dinesh Agrawal

We had a great meeting in India and I was particularly proud that many of the presenters were from the Microwave Working Group. David Clark asked me to briefly write up the meeting for the newsletter. Below are my thoughts together with the specifics prepared by Dr. Dinesh Agrawal of Penn State University who was one of the organizers and whom I am very happy to say is also a member of the Microwave Working Group.

The workshop took place from February 1-3, 2006 in Mumbai, India. It was organized by the Indian Chapter of the Materials Information Society (ASM International), and it was part of the Indo-US Science and Technology Forum established under an agreement between the governments of India and the United States. Being an autonomous, not for profit society this chapter ASM International promotes and catalyzes the Indo-US bilateral collaborations in science, technology, engineering and biomedical research through substantive interaction among government, academia and industry.

The conveners were Mr. Pradeep Goyal of Pradeep Medals LTD and Dr. Dinesh Agrawal, Director of the Microwave Processing Engineering Center at Pennsylvania State University and a member of the Microwave Working Group. There were nine technical sessions, each covering a particular industry or area of interest including chemistry, ceramics, pharmaceuticals, metallurgy, high temperature microwave technology, commercialization, rubber, and emerging areas of opportunity.

I was delighted that a number of the presenters are members of or colleagues involved in the Microwave Working group, including Dinesh Agrawal, Robert Schiffmann, Bernard Krieger, Jon Binner, Motoyasu Sato, David Clark, and Ed Ripley.

There were a total of 85 delegates and I was particularly impressed by the intense interest for developing more relations and contacts between India, the United States, Japan and Europe. The economy in India is growing extremely rapidly and they are eager to implement the latest technologies. They want to build relationships, buy technology, and at the same time sell services, systems and equipment to companies in the west who want to get a competitive advantage from their lower labor costs.

I hope you can recognize the presenters in the attached photograph. There are also several photographs of the President and the Vice

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If you are interested,
please let the editor
know!

President of the Microwave Working Group (Bernard Krieger and Bob Schiffmann) getting acquainted with some local inhabitants of the area. Our technical discussion during the photo opportunity with the elephant centered on my question to Bob whether my laptop has more memory than an elephant. When it comes to scientific questions of this type, I always ask Bob. He explained that the elephant wins because they are able to process information in parallel. Further, while it is said that an elephant never forgets old guys like us seem never to remember. Perhaps it's a non-thermal effect!

For more information on the conference and proceedings please contact Dr. S. A. Borkar: saborkar@rediffmail.com



Some of the invited speakers and attendees at the Microwave Technology for Materials Processing Workshop.



Bob Schiffman and Bernie Krieger with some new friends they met in Mumbai!

SCIENCE

MICROWAVE SINTERING OF LUNAR SOIL

By: L.A. Taylor and R.L. Schulz

Since man first stepped on the moon in 1969 until the final mission in December of 1972, many people from all walks of life have wondered: "When will we go again?" In the early days of the Apollo missions the

answers were clear. But, since Cernan, Schmitt and Evans of Apollo 17 left lunar orbit, more than three decades have passed and no one has returned to the moon's surface.

However, there is some hope on the horizon. The current administration vowed American astronauts would return to the moon by 2020 as the launching point for missions further into space. Europe, China, and India are all pursuing space programs, and the private sector has also thrown its hat into the ring. At this point, you may be wondering what this has to do with microwave energy. While the missions have ceased, scientific study has not stopped into the moon's make-up and how native materials could help sustain human life on the lunar surface.

Research scientist Prof. Larry Taylor and his team, at the Planetary Geosciences Institute of the University of Tennessee; have been investigating the use of microwave energy to sinter lunar soil. In a recent article published in the *Journal of Aerospace Engineering* (July 2005, Vol. 18, No. 3, pp. 188-196) the author's demonstrate the unique properties of the lunar soil and why is it particularly susceptible to processing with microwave energy.

The lunar soil is composed of disintegrated rock with a large component of impact-produced glass and an abundance of nanophased Fe^0 , the material that provides the lunar soil with its unique susceptibility to microwave processing. Once identified, speculation began into the origins of the nanophased Fe^0 (np- Fe^0). It is believed that np- Fe^0 was created thru a series of events beginning with micro-meteorite impacts creating a melting of small portions of lunar soil. Some of this melt was at such high temperatures ($>2000^\circ\text{C}$) that effective vaporization of the melt took place, releasing silica, iron, and other elements into the vapor state. Solar winds donated hydrogen protons to reduce FeO to elemental Fe^0 . The vapor subsequently condensed upon the outer coatings of most of the soil particles, as a thin (200nm) rim of silica-rich glass in which are suspended millions of nano-sized metallic Fe grains (3-30 nm). The major portions of the un-vaporized glass was also reduced by the solar-wind hydrogen, but this glass effectively cemented aggregates of soil particles together into agglutinates. This abundant glass further underwent shattering into fine particles, such that the fine particles of glass give the lunar soil a strong magnetic susceptibility. The $<20\ \mu\text{m}$ fraction, amounting to $\sim 20\ \text{wt}\%$ of the lunar soil, can be picked up almost entirely by a simple hand-held magnet.

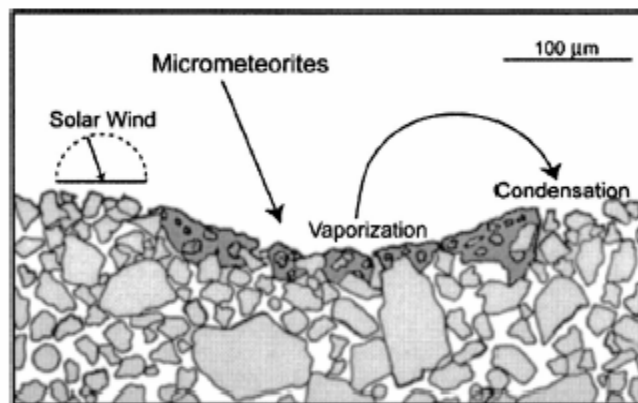


Figure 1. Schematic illustrating the formation processes of lunar soil, including comminution (breaking/crushing), agglutination (aggregation with glass binder), and vaporization (with condensation). (Taylor in: *Engineering, Construction and Operations in Space I*, ASCE, New York, 67-70, 1988).

Early microwave processing results, presented by Tom Meek and colleagues (Los Alamos Nat. Lab), were challenging to decipher because the effect of nanophase Fe^0 was not well understood. Furthermore, the “simulated samples” used did not really represent the make-up of actual lunar soil. Recent experiments with actual lunar soil have shed light on the unique reactions of the lunar nanophase Fe with microwave radiation. Taylor and Meek explain, “...the minute sizes of the nanophase metallic Fe are small enough such as to be less than the skin depth of the microwave energy, This makes each of the metallic Fe grains into a conductor versus a typical reflector, separated from the other metallic Fe particles by the dielectric glass (see Figures 2,3 below). The conductor abilities of the metallic Fe act as an absorber of the microwave energy, thereby creating “energy sinks” with the effective generation of large quantities of heat.” (*J. Aerospace Eng.* Vol. 18, No. 3, pp. 188-196, July 2005).

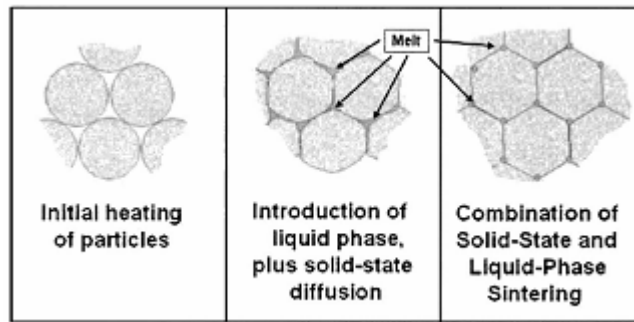


Figure 2. Schematic representation of sintering of lunar soil (L to R). The nanophase Fe^0 promotes the development of a transient-liquid phase, and it appears that temperatures along the grain boundaries are far in excess of the centers of the individual grains (*J. Aerospace Eng.* Vol. 18, No. 3, pp. 188-196, July 2005).

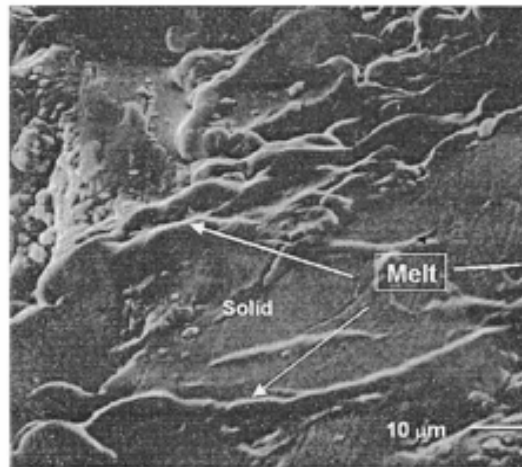


Figure 3. Scanning electron microscope photo of the surface of lunar soil grains after sintering with 2.45 GHz microwave radiation. Note the presence of a glassy region along the grain boundaries. The “measured temperature” of the sample was 900°C , however, the temperature necessary to produce the melt along the grain boundaries was 1200°C (*J. Aerospace Eng.* Vol. 18, No. 3, pp. 188-196, July 2005).

Because of the $np\text{-Fe}^0$, the lunar soil is particularly well suited to microwave sintering. Benefits such as rapid-heating rates ($>1000^\circ\text{C}/\text{min}$), enhanced reaction and diffusion rates, faster-sintering kinetics, shorter-processing time and lower-processing temperatures have been demonstrated. In addition, considerable energy savings are realized. Another advantage would be lighter-weight equipment that would need to be transported to carry out the processing. The authors have proposed a machine that could be easily adapted to a number of processing scenarios. One such adaptation is illustrated in Figure 4. For more information on this fascinating work, please contact Prof. Larry A. Taylor at the University of Tennessee (lataylor@utk.edu).

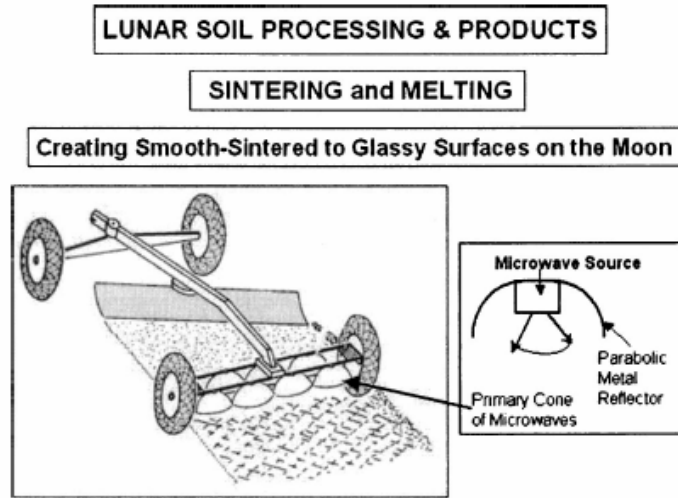


Figure 4. Artist rendition of possible microwave processing equipment for paving the lunar surface to form a road, thereby militating against the deleterious effects of the abundant lunar dust (*J. Aerospace Eng.* Vol. 18, No. 3, pp. 188-196, July 2005).

TECHNOLOGY

MICROWAVE INDUCED PLASMAS AT ATMOSPHERIC PRESSURE

By: John Gerling

Applications of microwave induced plasmas (MIP) at atmospheric pressure have been around for decades. Indeed, methods and apparatus for atmospheric MIP were established as early as the 1960's for applications mostly in atomic emission spectroscopy. By the 1980's and 1990's a variety of microwave plasma torches operating at atmospheric pressure had been developed for applications in thin film deposition, heat treating, waste treatment and other processes. More recently, large volume atmospheric MIP have been applied to bulk materials processes such as carburizing, joining, coating and sintering.

With the exception of the large volume atmospheric MIP processes, most applications mentioned above require a substantially high electric field strength in order to ignite the plasma. An e-field of approximately 1.5 kV/cm is required to breakdown air at atmospheric pressure, whereas

the e-field developed in WR284 waveguide with 1kW at 2450 MHz propagating in the TE₁₀ mode is less than 250 V/cm. Thus, much of the application development was focused on practical resonant cavity geometries that achieved the necessary high e-fields. Starting with a simple waveguide applicator, an intuitive solution is to reduce the waveguide height by tapering down from full height (proportionally increasing the e-field) and locating the plasma discharge tube ¼-guide wavelength from the shorted waveguide termination (doubling the e-field). However, a waveguide height reduction also effectively increases cavity Q, an advantage by further increasing the e-field but consequently resulting in increased frequency sensitivity and tuning difficulty.

Much of the early work in developing practical atmospheric MIP resonant cavities for atomic emission spectroscopy was to overcome the inherent frequency sensitivity by implementing adjustments for frequency tuning. The ubiquitous "Evenson" cavity evolved from a fixed geometry half-height ¼-wave short waveguide to a compact adjustable ¼-wave coaxial resonator. The adjustability provides the means for frequency tuning and impedance matching, and its efficient high-Q design enables reliable atmospheric plasma discharges at microwave power levels of only a few hundred Watts.

Other industrial processes, however, require much higher microwave power levels and gas flow rates than can be handled using an Evenson type cavity design. The microwave plasma torch also began as a reduced height ¼-wave short waveguide cavity resonator from which several designs emerged having very different geometries. The "Okamoto" cavity is an example of one that produces an large diameter annular shaped (toroidal) plasma. An important design constraint for many applications is that operation is continuous for long periods. Thus, the ability to ignite the plasma is of less concern than to sustain it. This observation allows designers to use alternate means for breakdown (e.g. high voltage spark or high intensity UV lamp) that do not require an e-field any higher than necessary to sustain the plasma.

Large volume atmospheric MIP processes are conducted in multimode cavities having relatively low Q and where the e-field is much lower than required for normal breakdown. Rather than utilizing a resonator geometry to boost the e-field for breakdown, the process gas chemistry is altered such that breakdown can occur at a much lower e-field. Once breakdown is achieved the plasma is easily sustained at normal microwave power levels. Mode stirring provides relatively uniform average distribution of plasma intensity within the reaction volume. These methods enable large scale processes for a wide variety of existing and new applications.

APPLICATIONS

DO YOU HAVE A CASE OF MAXWELLITIS?

By: Bernard Krieger

I am frequently asked to estimate the size of the industrial microwave market. While I have been at it for forty years, it is still a perplexing question. Sure, Bob Schiffmann can tell you how many millions of home microwave ovens are made worldwide and distributed by country but that is a different question. "Home microwave ovens" are a specific product, the term "industrial microwave" describes a technology that is implemented in many different forms, and is not a specific product. Ok, then you can ask: "What are the yearly sales of the various microwave

competitors?" Unfortunately, that number will be disappointingly low because it is difficult to define a "microwave competitor."

The problem I believe is that we are fixated on the word "microwave" a word which describes a technology and not an application or a product. If we add the word "oven" to the description we are closer to something more meaningful. We should then also add the application of the oven to get an even better definition and thereby measure the markets for industrial food, rubber, ceramics, etc. But here is where we run into an even bigger problem in defining competition. Let's take an example: If the application can be fulfilled by microwaves or by hot air, do we include "hot air" in the definition of competition to measure the market size? I certainly do and I also include infrared, steam, and perhaps even the sun if they are applicable. If you don't consider these, you could be making a big mistake and may be suffering from "Maxwellitis."

You can counter that we are only trying to define "microwave" markets and we don't care about "conventional" ovens. If we think that way we are myopic because we are thinking in terms of what we do and not in terms of what the customer needs. This is a definite symptom of Maxwellitis and a sure way not to grow markets.

This of course does not mean that microwave suppliers should make every kind of oven. What it does mean is that we should be focused on solving the customer's need in the best way possible and often it may not be with microwave energy.

If you believe you are establishing new beachheads by developing techniques to heat materials which are not very happy in a microwave oven, you should first ask yourself "is there a beach to that beachhead?" We have to think in terms of the customer's bottom line. I suggest thinking beyond Maxwell to include Fourier and Reynolds because what we do fundamentally involves heat transfer, mass transfer and diffusion. There are numerous processing alternatives to accomplish those criteria.

The real competition for microwave energy is very big and the markets are tremendous. The only thing that may be small is the way we think about it. What then are the pros and cons when deciding to use microwaves either alone or in a hybrid application? I plan in future issues of this publication to cover this subject and in particular, trends and new applications for our technology.

ASK THE EXPERTS

If you have a question about microwave or RF processing, equipment, or research, and would like it answered by qualified experts in the field, please send your inquiry to the editor. Selected questions will be answered in the next newsletter. All questions received will be answered on the MWG website. In this issue research activities carried out at Virginia Tech are highlighted.

MICROWAVE RESEARCH AT VIRGINIA TECH

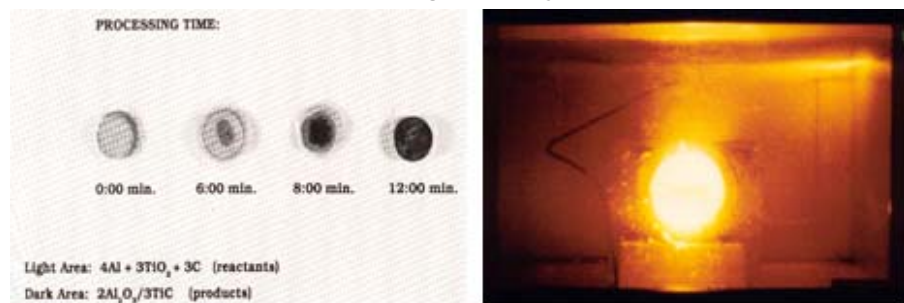
By: D.C. Folz

Since moving from the University of Florida in 2001, David Clark and research faculty member Diane Folz have built the Microwave Processing Research Facility at Virginia Tech's Department of Materials Science and Engineering. After 20 years of research in microwave energy, Clark has seen this processing technology grow from a few researchers scattered across the country to a viable manufacturing operation and global funding

opportunities for academic as well as industrial researchers. This unique laboratory is equipped to perform research on materials systems using microwave energy ranging from 2.0 to 18.0 GHz with both fixed and variable frequency capabilities and power levels from 200W to 6.4 kW. Current microwave research in the lab supports three faculty, three graduate students and five undergraduates and focuses on waste remediation and recycling, nanomaterials synthesis and processing, rapid formation of glass-ceramics, sterilization, and medical treatment technologies. The group holds seven patents and has over 200 publications and presentations in the area of microwave processing.

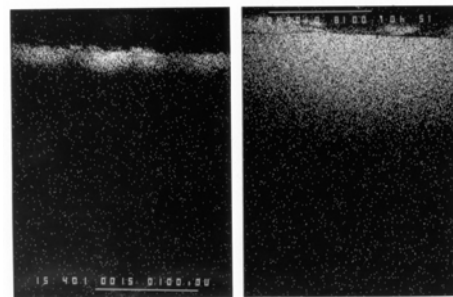
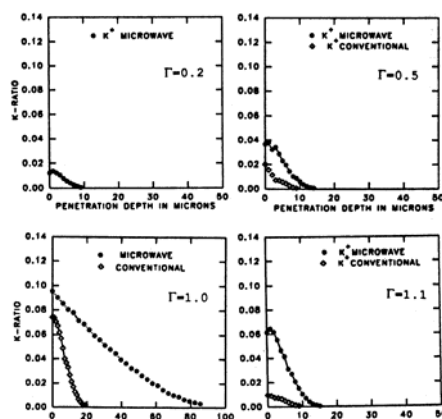
The early work conducted by graduate student, Arindam De, showed the real promise of microwave energy as a processing technique for ceramic materials. It was seen that alumina densified using microwave energy yielded a significant increase in microstructural uniformity throughout the cross-section of the samples than that which could be achieved using conventional processing. It also showed that uniformity could be increased still more with increased sample size. This research also confirmed what had been predicted by Janney et al at Oak Ridge National Labs and Sutton et al at United Technologies Research Center; that microwave energy penetrated the samples to heat "volumetrically" and to produced temperatures that were higher on the inside of refractory materials than those measured on the surfaces of the samples.

Self-propagating high-temperature synthesis (SHS) was proposed as a rapid method for producing composite powders and monoliths of composite materials for a wide variety of industrial and military components. However, control of conventionally initiated SHS reactions in monolithic pieces proved to be a technical barrier to implementing the technology in manufacturing of these materials. Conventionally, reactions were initiated by igniting one surface of the sample via radiant heat or laser techniques. The reaction propagated, uncontrolled, throughout the sample until most of the materials had reacted. Later investigations focused on the use of microwave energy to ignite the SHS reaction in composite materials of aluminum-titanium-carbon. It was found that the microwave energy penetrated the material to ignite the reaction in the center of the material and that the reaction wavefront propagated radially outward to the surface. By adjusting the applied power, the rate and degree of reaction of the materials could be controlled. This process showed enormous potential for efficient, controllable manufacture of ceramic and ceramic-metal composite components. It also may lead to a method for rapid production of high-quality complex shapes.



One of the advantages of microwave interactions with materials is that diffusion appears to be enhanced. Starting with the well-known sodium aluminosilicate glass system, Fathi and Clark conducted experiments to observe the substitution of potassium for sodium during surface modification processes. The Figure 2 below shows the penetration depth of sodium into the glass for conventional and microwave processing. The

SEM images further highlight the improved surface modification.



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COMPOUND	SR-8 (ppb)		SR-9 (ppb)	
	A	B	A	B
Benzene*	5838.9	22.2	1415.6	139.5
Toluene*	8146.6	15.7	4215.9	158.7
Ethylbenzene*	1147.4	nd**	4557.0	5.2
Styrene*	1666.9	6.2	20012.0	38.4
Napthalene*	355.5	nd	2403.6	27.9
m/p Xylenes*	2259.0	nd	510.6	nd
1,3,5 Trimethylbenzene	1564.0	nd	378.7	64.3
1,2,4 Trimethylbenzene	904.7	nd	171.8	nd


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A = Before microwave off-gas treatment; B= After microwave off-gas treatment

*Listed as hazardous air pollutants in the Clean Air Act, as amended, 1990

** nd= not detected (< 1ppb)

Over the past decade, Clark's group continues to be active in organizing major U.S. meetings in microwave processing of materials. Clark and Folz have taken leadership roles in the organizing committees of the four World Congresses on Microwave and Radio Frequency Processing and Applications, the last of which was held in November 2004 in Austin, Texas. They also have been lead or co-editors for all of the Congress proceedings published to date. They have collaborators in industry as well as government agencies and are willing to work with groups interested in advancing the basic science knowledge and developing manufacturing processes based on microwave science and technologies. Microwave technology will not be the answer for every process, but it does offer significant advantages for many process applications and can provide a method to produce materials not possible



with current conventional techniques. For more information please contact Diane Folz: dfolz@mse.vt.edu (540)-231-3897.