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Microwave Processing and Engineering Center (MPEC) at Penn State: The Latest Developments

by Dr Dinesh Agrawal

Professor of Materials and Director of MPEC

Dinesh Agrawal

Since 1984 the Materials Research Institute (formerly known as Materials Research Laboratory) at The Pennsylvania State University has been a pioneer institution in the microwave processing of a whole range of ceramics, composites, and metallic materials. In the 1980s under the guidance of Prof. Rustum Roy, we had remarkable successes in sintering and synthesizing many traditional ceramics such as alumina, zirconia, ZnO, [NZP], hydroxyapatite, zeolites, mullite, silica, etc. Then in the 1990s the focus of our research moved to new materials and in new directions. Many electroceramics such as PZT, BT, BMT and transparent ceramics were successfully synthesized, fabricated and sintered in microwave fields. Remarkable advantages were noticed using microwave technology for the sintering and synthesis in terms of substantial enhancement in the kinetics and dramatically reduced cycle times. Then for the first time we launched programs to sinter non-oxides, especially WC/Co based products. The success made in this area led to the innovative approach of continuous microwave sintering which made possible the commercialization of the technology for WC/Co based products applied in the cutting and drilling industry. Then in 1996 we made another big advancement in the microwave field by successfully sintering powder metal parts very effectively and efficiently with improved

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Editor's Comment

I am delighted that Professor Dinesh Agrawal of Pennsylvania State University has accepted my invitation to report on the activities of the Materials Research Institute. The origins of this unit can be traced back to the early 1980's when under the guidance of Professor Rustrum Roy much was done in the area of sintering and synthesizing of many traditional ceramic materials.

The 3rd World Congress has taken place in Sydney Australia and B Vaidhyanathan from the University of Loughborough reports on its proceedings on page 3.

At the last AMPERE Committee meeting in Loughborough University, John Bows very kindly offered to start a CASE STUDY column where we can report on interesting industrial developments in our field of activity. Their first report appears on page 5. John would appreciate any material from any member for inclusion in future issues. I have also had one interesting comment regarding the move to AMPERE Europe Ltd and this will be incorporated in the articles before completing the matter with the solicitors.

Do note that I have moved office and I am now based at St John's College, where my full particulars are St John's College, Cambridge, CB2 1TP, UK tel: +1223 338646 otherwise my e-mail for the present remains the same. My collaboration with the Engineering Dept continues as there are a couple of students completing their PhD's. Further the AMPERE accounts and subscriptions are from now on handled by Jon Binner and his staff at the University of Loughborough. Ann Martin has retired as AMPERE administrator and I am sure you would like to join me thanking Ann for her stirling efforts to AMPERE throughout the last eight years and wishing her all the best in the future.

Ricky Metaxas

St John's College, University of Cambridge performance and better mechanical properties. This had opened up completely new avenues of research and commercial exploitation of microwave technology in new applications. Most of our innovations here have been dominated to date by serendipitous discovery, because the interaction between microwaves and the materials of widely varying properties and sizes and shapes is so complex that it has defied quantitative analysis to fathom the real mechanisms behind the "microwave-effect".

With the discovery of sintering of metallic materials in microwaves we launched new programs systematically with a generous financial support from EPRI (Energy and Power Research Institute) and established **"Microwave Processing and Engineering Center" (MPEC)** in 1997. The establishment of this Center aimed to exploit new advances made in the field of microwave processing, and apply them to fabricate a variety of materials of various shapes and sizes with improved properties leading up to their commercialization. The Center's goals include the following:

- Conduct process and development studies on candidate materials for producing better performed product.
- Conduct research in the microwave processing and sintering of ceramic powders.
- Conduct research in new areas where microwaves can be effective.
- Embark on a systematic investigation of the microwave effect in various traditional and advanced materials systems.
- Establish co-operative and collaborative research programs with industries on specific materials development using microwaves.
- Provide a state-of-the-art equipment facility and technology in microwaves processing to the relevant industry.
- Render assistance in technology transfer and commercialization of microwave processing and products development to the sponsoring members.

Since 1997 our microwave center has made tremendous success in conducting research in new areas and also transferring developed technologies for speciality products to several relevant industries leading to the commercialization of many products. Our efforts have been always to take the successes achieved in the laboratory to the market place. Though in the past much research had been done in the microwave processing of ceramics, not much was translated into commercial success, mainly due to the limitations and difficulties faced in the scaling-up operations. At our microwave center we have designed and developed several microwave systems, which can circumvent these problems and can be used for commercialization of the developed technology.

Our Center is equipped with a variety of microwave systems capable of conducting research on oxides, non-oxides and metals, demonstrating feasibility studies for large scale production, and design and building of prototype microwave systems for scale-up operations. At present the Center consists of the following microwave facility:

- Regular microwave systems (1 kW, 2 kW, 2.45 GHz, multimode)
- Single mode MW systems (1.5 kW, 2.45 GHz)
- Continuous microwave systems (3kW, 6 kw, 2.45 GHz, multimode)
- Batch process microwave systems (3 kW, 6 kw, 2.45 GHz, multimode)
- High Power 915 MHz Microwave systems (30 kW, single- and multi-mode)
- Dual Frequency microwave system (2.45 and 5.8 GHz, multimode)
- High vacuum multimode microwave system (6 kW, 2.45 GHz)

The most recent developments at our microwave center involve the study of heating, reaction kinetics and sintering of various materials in separate electric (E) and magnetic (H) fields at microwave frequencies. The experiments performed so far in this area show very interesting results which would help in identifying the answers for yet unexplained mechanism(s) of microwave-matter interaction.

At present MPEC is involved in the following main projects. These projects are sponsored by industry and government agencies:

- Development of microwave technology for WC/Co based products including diamond composites for gas and oil industry
- Microwave processing of metallic materials including refractory metals
- Microwave brazing of super alloys
- Microwave sintering of ZnO varistors
- Microwave sintering of alumina based products including transparent aluminas
- Fabrication of transparent ceramics of AlON, AlN, alumina, MgO etc.
- Fabrication of MLCs for electroceramic devices

- Synthesis of phosphors including fluoro-apatites
- Synthesis and sintering of ferrites
- Investigation of the effect of E and H fields (2.45 GHz) on heating behaviour of materials
- Fabrication of light weight ceramic armor
- Eradication of long horn Asian beetles using microwave technology

Some of the highlights of our key achievements are listed below:

- Processing of hard metal tools based on tungsten carbide composites. These products are now manufactured at lower cost than the standard tools, and also with substantially improved performance.

- Synthesis of ceramic phases such as $BaTiO_3$, $Ba_3MgTa_2O_9$, and $Pb(Zr,Ti)O_3$ in a few minutes at the astonishing temperatures of 300° - 700°C by using reduced TiO_2 or Ta_2O_5 or partially stabilized ZrO_2.

- Fabrication of transparent and translucent ceramics of Al_2O_3, AlN, AlON, Spinels, Mullite, in a few minutes without using any pressure: Transparent ceramics are highly advanced materials for special applications and always are difficult to achieve by traditional techniques. It is possible to fabricate them now in a microwave field with little innovation in a single step process and ambient pressure.

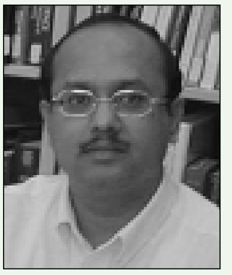
- Sintering of typical steels, powder metals, alloys, and refractory metals to high density and fine microstructure. In almost all microwave sintered powder metal samples, the mechanical properties were found to have substantially improved.

- Design and setting-up of second-generation microwave sintering apparatus, and development of a new technique in which green ceramics of typical commercial applications can be processed *continuously* with high energy efficiency to yield perfectly formed products. This system is a key for any commercialization of microwave processed material.

For more information, please visit our website: http://www2.mri.psu.edu/centers/mpec/ or contact Prof. Dinesh Agrawal dxa4@psu.edu 814-863-8034, 814-863-3617 (Fax)

3rd World Congress on Microwave and Radio Frequency Applications

by Dr B. Vaidhyanathan, University of Loughborough


B. Vaidhyanathan

The 3rd World Congress was held between 22-26th of September 2002 in Sydney, Australia. Breathtakingly beautiful Darling Harbour housed the venue for the conference, the Sydney Convention Center. Darling Harbour, with fantastic day and night views, a wonderful skyline, lush green parks, buzzing shopping arcades, numerous water fountains and restaurants serving a range of international delicacies, certainly lived up to its name. There was plenty of hotel accommodation around the convention center to suit moderate budgets and for those delegates who preferred to stay further away, the venue was extremely well connected by every type of transport you can think of: monorail, city rail, bus, tram, ferry, land and water taxis. Though a couple of delegates would have chosen a cheaper venue elsewhere in Sydney, there was a definite consensus that the slightly higher cost of hosting the conference in Darling Harbour (ameliorated by the relatively weak Aussie dollar) was worth it for the view alone. The conference centre itself consists of three self-contained floors with 3WC being split across the top two - probably to indicate its superior status! Or else the organizers wanted to provide a bit of exercise to the delegates (though very few used the staircase)! The theme of the congress was to bridge Science, Technology and Applications and the schematic of the magnificent Sydney Bridge on the cover of the abstract book thoughtfully represented this.

Around 180 delegates attended the conference, representing twenty countries across the globe and more than 90 oral and ~30 poster presentations were presented, covering a wide spectrum of microwave and RF applications. Whilst this number was smaller than the attendance in previous world congresses, the reasons probably include the longer travel distances and associated expenses involved, and the changed funding scene in the US and other countries after 11th September. However the smaller number provided more one to one contact and better interaction among the participants.

There were three parallel sessions on each day and these technical sessions covered a range of topics including inorganic materials, industrial applications, systems & diagnostics, semiconductors & electronics, waste remediation, chemistry and organic materials, biomedical applications, modelling etc. It was comforting to note that on three out of four days there was a full session dedicated to applications, which is very important if the gap is to be bridged and laboratory science transferred to industry and on to consumers. There was also a full day of sessions dedicated to bio-medical aspects and health effects of RF and microwave radiation and it was heartening to find a whole new field of research coming to prominence, though a few participants involved in other research disciplines conveniently used this to explore Sydney (and one can't blame them considering the shear beauty of the place and the fact that many of them have to leave Australia immediately after the conference). In her plenary speech, Maria Stuchly gave a wonderful account of microwave applications in medicine. With overwhelming evidence she demonstrated the use of microwaves in both diagnostic and therapeutic applications and the significant progresses made in breast cancer detection – a very heartwarming development. After the 2nd World Congress Jon Binner (July 2000 issue) indicated his desire to have a large number of papers in disciplines other than ceramic processing and it has definitely been fulfilled at this year's conference.

There was an extremely interesting paper presented by Bob Schiffmann, where he demonstrated a new type of microwave oven with a very high level of field homogeneity achieved using a "Match Plate" resonator (which provides constant impedance irrespective of the load and its changes). It was astounding to note that any number of metallic shelves can be included and materials can be processed uniformly on all of them without any diminution of field homogeneity and with absolutely no arcing. When asked about the capabilities of retrofitting the existing cavities with his new technology, beaming Bob gave an affirmative 'yes'. Though the basic science aspects of such novel microwave equipment have not been clearly understood yet, this is worth investigating from the applications point of view.

The open forum organized by David Clark was outstanding and mention should also be made of the excellent turnout for this event. An overwhelming number of the participants indicated that the World Congresses are worth continuing and there were some positive suggestions made for improvement viz., bringing in more consumers on site, having parallel meetings with other focused technology organizations, increasing student participation, increasing the involvement from the RF community, concentrated efforts to bring down the cost, etc.

Whilst it would be wonderful to discuss the stimulating presentations in each of the different areas of research, unfortunately space does not permit this. The student participants, though their number was found to be less than in previous World Congresses, indicated that the conference provided an authentic overview of various applications of high frequency heating in different areas and was very useful.

The trade exhibition was very well attended. Holding the exhibits, poster sessions and the refreshment breaks in the same hall was found to be a tremendous success and this view was shared by all the exhibitors and presenters I spoke to. The social program of the congress was extremely good and the people who attended the various social tours and technical tour gave a clear thumbs-up to the organizers. The delegates thoroughly enjoyed each others company and were found going out together for dinner in the evenings, to places of interest such as Sydney aquarium, Chinese garden, IMAX theatre complex, etc (indeed they were helped by the discount vouchers thoughtfully provided by the conference organizers). The conference dinner cruise was a major success. It not only provided the wonderful opportunity to enjoy the stunning beauty of the Sydney Harbour, Bridge and the Opera House covered in vibrant lights, but also helped in contact building, technology transfer and in establishing personal relationships. Three awards each were presented for professional and student posters and I am personally delighted that the microwave processing group at Loughborough won a prize in each category. Another important social event was the tradition of World Congresses in giving a free-swimming lesson to the Technical Program chair (John Booske) for all his hard work, this time in the cold waters of a fountain.

Last but not the least, Tour Hosts Pty Limited, the conference managers (the Ladies in Pink as Luke Nadj called them) did an outstanding job in helping all the delegates and in bringing it all together. The superior technical program made the 3rd World Congress highly valuable and the cuddly koalas, kangaroos, and the beautiful city of Sydney made it even better. The 4th World Congress in 2004 will be back in the US and I hope the saga continues.

Case Studies No 1: Cosmetic producers get ROI after 6 months with microwave installation

Cosmetic producers Johnson & Johnson (Penaten, Germany) and Beiersdorf (Bode, Germany) installed microwave production lines during 1997 and 1998 (respectively) for the processing of Panthenol, an active ingredient in many cosmetic products. Figure 1 shows an installed process for up to 200kg batches of Panthenol.

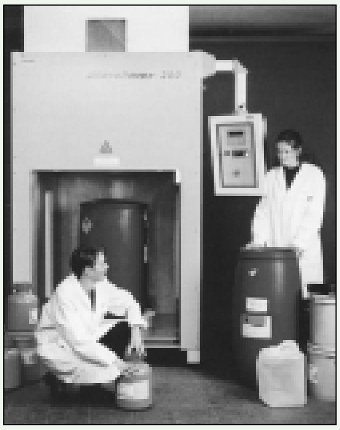


Figure 1

Panthenol exists either in the state of dexpanthenol or in the form of its L-isomer. Dexpanthenol acts as a vitamin as it is oxidised in the body to yield Vitamin B5 (pantothenic acid), but the L-isomer does not provide any vitamin activity. Consequently, Dexpanthenol is one of the most frequently used vitamins in cosmetics.

Dexpanthenol is an ideal ingredient for use in cosmetics for two reasons. Firstly, because Panthenol increases the water binding capacity of skin, nails and hair. On hair, it forms a protective coating, increasing thickness and strength. It also causes a pleasant feeling on skin and hair. Secondly, dexpanthenol penetrates into the skin cells where it is metabolised into Vitamin B5. Epithelial growth is stimulated, which, along with the anti-inflammatory properties of dexpanthenol, explains its successful application as the major component in wound healing preparations. In cosmetics, applications include after-shave lotions, suncare and after sun preparations, as well as baby care formulations.

The addition of 0.5 - 2% dexpanthenol is able to help small wounds, abrasions, and blisters heal more quickly. However, Panthenol comes in two forms: the more expensive form as a 75% concentration, which is already liquefied, and whose formulation is protected by patents, and a 100% concentration form. The much cheaper 100% concentration is a highly viscous material (105,000 mPas at 30°C) and needs to be liquefied prior to processing into the various cosmetic formulations.

Typically, in conventional production, the 100% concentration form was heated up to 70°C over several hours in a water bath or in an incubator. Overheating needs to be avoided to assure a stable active ingredient. This time consuming process brings high costs and impairs the flexibility of production. A method of liquefying Panthenol with microwave heating was developed by the material producer (Daiichi Pharmaceutical Europe GmbH) in co-operation with a producer of industrial microwave equipment (Puschner Microwave Energytechnique). The challenge was to heat material in plastic packaging (typically barrels) ranging from 10 to 200kg under controlled conditions.

Panthenol dielectric properties were characterised, showing penetration depth increasing with temperature (at 20 and 50°C, 10 and 20 cm penetration depth respectively at 2.45 GHz). Pilot work was performed on 10kg bottles of dexpanthenol using a 5 kW applicator. Maximum temperature differences of 20°C C from periphery to core were achieved, with the highest temperatures in the upper section in the bottle (Figure 2). Heating 10kg dexpanthenol from 18°C up to the core temperature of 70°C required 7 or 15 mins at 1.5 or 5 kW microwave power respectively. The liquefying process was approximately 50 times faster than by using conventional techniques.

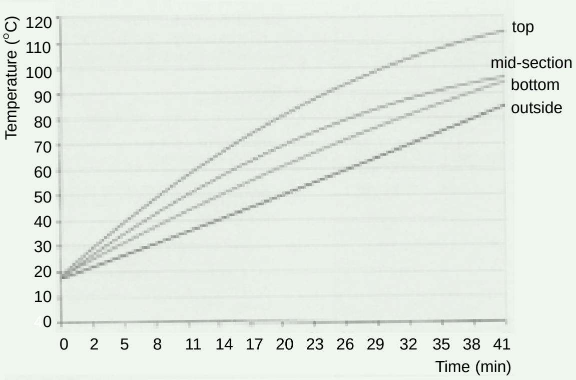


Figure 2

To achieve an even heat distribution in the production unit, the applicator consisted of a multimode cavity with 6 coupling port applying microwave energy. Constant energy input zones were designed to avoid over-exposure (the 6 coupling ports can be switched in way that a 10 kg or 200 kg barrel can be heated rapidly and uniformly). Product-specific temperatures programmes were developed. On-line infrared cameras were installed to monitor the pack wall temperature as a process control measure.

Degradation products from microwave heating dexpanthenol were thoroughly evaluated and the process control that could be achieved generated product that was within specification. For example, racemization of dexpanthenol into L-panthenol

NEWS & EVENTS

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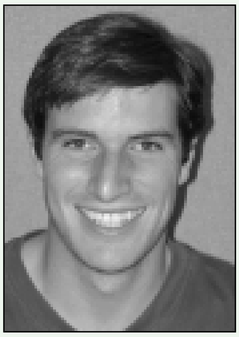
RICHARD EHLERS JOINS AIRBUS



Dr Ehlers has left the EUG at Cambridge and joined AIRBUS Industries presently based in Hamburg where he will be involved in new control systems for the aircraft industry. He will also continue his research with the numerical work on microwave heating usine finite elements in collaboration with Ricky Metaxas at St John's College, Cambridge. He can be contacted at rae@cantab.net. We wish Richard every success in his new environment.

Case Studies No 1

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from prolonged heating at 85°C for 5 hours, overheating to 125°C, off-odours from production of amino-propanol and pH variations were all within specification.

Batch processing plants have been constructed for heating dexpanthenol in plastic packings up to 200 kg for exposure to 4/8/12 kW microwave energy, such as the mWavePower200 for 200 kg plastic packs (Figure 1 previous page). A 200 kg batch takes 40 minutes to heat using 12kW.

A critical success factor was the return on investment (ROI) of 6 months for Johnson & Johnson, calculated from the processing of 60 tonnes Panthenol per year and the price difference between the 75 and 100% forms. A further benefit was that the plant could be used for a larger range other raw materials.

Further information can be found in the article "Microwaves and Dexpanthenol: A problem solution marks a novel standard in the production of cosmetics" by S.Bodenbach & P.A.Puschner in SOFW-Journal-Sonderdruck, volume 123, May 1997, pp 310-316. Thanks are due to Peter Puschner for supplying additional details.

John Bows, Unilever Research Colworth, UK

CPI Autowave (TM)

CPI (Los Angeles, US) have recently launched *Autowave* (TM), a microwave furnace for laboratory use.

Working areas within the cylindrical chamber are stated as 30 x 30 x 30 cm. A second model is available at 50 cm length. Operating powers and frequencies are 2.45 GHz at 2.5 or 5.0kW powers, or 18 GHz at 2.5 kW, with continuously variable power from 10 to 100%. Each system is computer controlled based on *Labview* (TM) software.

Highest working temperatures up to 2000°C are possible, dependent on the chamber material. Systems come with a stub tuner for matching the cavity, vacuum and gas handling capability, and two view ports. Optional extras include temperature sensors (infrared pyrometers, fibre optic probes) and high vacuum capability. Uniform heating in the work area is claimed through use of a mode stirrer, a cavity design much bigger than the work area and computer designed systems. CPI also offer scale-up services, and prices start from around $100,000.

For more details, visit
http://www.cpii.com/bmd/cpinew/Products/autowave/Autowave.html