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INDUSTRIAL SYMPOSIUM II

MICROWAVE PROCESSING OF MATERIALS

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INDUSTRIAL SYMPOSIUM II MICROWAVE PROCESSING OF MATERIALS

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Scope

Microwave processing of materials is emerging as an innovative technology for future applicable to many diverse fields and variety of materials. Recent developments in this field worldwide are attracting attention of various industries. This full-day industrial symposium aims to provide to the relevant and interested industries to such recent developments, commercialization of the microwave technology in the area of ceramics, composites, metals, etc. Symposium topics will also cover other microwave applications especially related to chemistry, waste processing, and alternative and unconventional energy sources.

Detailed Technical Program

Time	Programme	Presenter
08:30 - 08:50	Registration	
08:50 - 09:00	Welcome and Opening Remarks by Prof. Dr. Dinesh K. Agrawal	
	Session I Chairperson: Assoc. Prof. Dr. Manoj Gupta	
09:00 - 09:30	Keynote: The Growth of High Temperature Microwave Processing in China	Hu Peng
09:30 - 10:00	Application of Microwave Energy for Materials Processing: A Global Perspective	Dinesh K. Agrawal
10:00 - 10:30	Tea Break	
	Session II Chairperson: Assoc. Prof. Ir. Dr. Ramesh Singh	
10:30 - 11:00	Hybrid Microwave Heating and Metal Based Materials	Manoj Gupta
11:00 - 11:20	Effect of Atmosphere on Microwave Sintering of Stainless Steel	Shivanand Borkar
11:20 - 11:40	Stabilization and Demulsification of Crude Oil Emulsions via Microwave Heating Technology	Abdurahman Nour
11:40 - 12:00	Hard Magnet $Nd_2Fe_{14}B$ Nano Powders by Microwave Assisted Hydrothermal Synthesis	V. Swaminathan
12:00 – 14:00	Lunch & Poster Session	
	Session III Chairperson: Mr. Garimella Balaji Rao	
14:00 - 14:30	Microwave Materials Processing Research in Japan: Recent Activities	Noboru Yoshikawa
14:30 - 15:00	Microwave Processing of Tetrahedrally Coordinated- and Compound- Semiconductors	Subhash Kashyap
15:00 - 15:20	Effects of Ceramic Crucibles on Properties of Microwave Sintering of Tin Based Alloy	Azrina Arshad
15:20 - 15:40	Enhance Degradation of Chlorobenzene in Aqueous Solution Using Microwave-Induced Zerovalent Iron and Copper Particles	Chien-Li Lee
15:40 - 16:10	Tea Break	
	Session IV Chairperson: Dr. Qingfa Li	
16:10 - 16:40	Microwave Research Activities in Europe	Cristina Leonelli
16:40 - 17:00	Microwave Sintering of Refractory Metals/Alloys: W, Mo, Re, W-Cu, W-Ni-Cu and W-Ni-Fe Alloys	Avijit Mondal
17:00 - 17:20	Study of Mechanical Properties of Microwave Processed YSZ	Anirudh Singh
17:20 - 17:30	Closing Remarks by Dr. Hu Peng	

ORAL SESSION

The Growth of High Temperature Microwave Processing in China

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The growth of high temperature microwave processing in China has accelerated over the past decade. After the buildup of academic knowledge in the late 1980's through the 1990's companies sought to apply this knowledge to engineer systems to produce products using microwave energy to take advantage of the inherent economic and ecological benefits that are pervasive in the literature. To date, the production of countless classes of ceramics and both common use and specialty metals has been achieved using high temperature microwave technology. Microwave systems manufactured to process industrial products are very diverse and range from 1kW systems for R&D to megawatt systems for large volume production. Next generation systems have been proposed for a number of industries and ongoing projects show promise.

Keywords: Microwave; Ceramic; Education

Application of Microwave Energy for Materials Processing: A Global Perspective

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Microwave energy has been in use for over five decades in diverse fields such as communication, medical applications, food processing, rubber vulcanization, chemical reactions, drying of various products, and materials processing. This shows the versatility of the application of the microwave energy. It has been widely recognized that microwave processing provides distinct and substantial advantages over the conventional processes due to primarily the manner in which microwave field interacts with the matter and transfers the energy to the work-piece. Some of the main advantages of microwaves in materials processing are: energy efficiency, properties improvements, rapidness, and eco-friendliness.

For the last 20 years many research groups all over the world have been conducting extensive research mainly in the ceramics, composites and recently metallic materials processing. Now, due to specific new developments, especially at the Penn State, in Japan, and China that the successes achieved in microwave processing of variety of materials at the laboratory scale are slowly getting translated into viable and successful commercial operations in many fields. This presentation will discuss these developments and provide an overview of the Penn State's two decades' work in microwave materials processing.

Keywords: Ceramic; Microwave; Energy

Hybrid Microwave Heating and Metal Based Materials

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The use of microwaves as an energy efficient solution to process a wide range of materials is strongly emerging from last two decades. The rapid heating capability of microwaves is often utilized to minimize microstructural coarsening and to realize enhanced properties. This presentation will highlight the concept and use of hybrid microwave heating to synthesize a wide range of metal based materials including lead free solders, aluminum and magnesium based materials including nanocomposites. The observations include energy saving in excess of 80% and up to 96% with no compromise on the end properties of the metal based materials.

Keywords: Microwave; Energy; Light Materials

Effect of Atmosphere on Microwave Sintering of Stainless Steel

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The most recent applications of microwave technology have been reported in processing of metallic materials specially in sintering, brazing/joining and melting. Many commercial powder-metal components of various alloys and different shapes prepared by using different sintering techniques have been reported by researchers. It was reported that for obtaining better properties, control of atmosphere plays important role as it decides the total processing time and end properties. The paper presents and discusses effect of atmosphere on final properties of compacted stainless steel powders sintered by microwave technique.

Keywords: Microwave; Energy

Stabilization and Demulsification of Crude Oil Emulsions via Microwave Heating Technology

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Traditional ways of breaking emulsions using heat and chemicals are disadvantageous from both economic and environmental perspectives. In this research, the potentials of microwave technology in demulsification of water-in-crude oil emulsions are investigated. The study began with some characterization studies to provide understandings of fundamental issues such as formation, formulation, and breaking of emulsions by both chemical and microwave approaches. The aim was to obtain optimized operating conditions as well as fundamental understanding of water-in-oil emulsion stability upon which further developments on demulsification processes could be developed. It was found that emulsion stability was related to some parameters such as, the surfactant concentration, water content, temperature, and agitation speed. Experimental results found that microwave radiation method can enhance the demulsification of water-in-oil emulsions in a very short time compared to the conventional heating methods.

The results obtained in this study have exposed the capability of microwave technology in demulsification of water-in-oil emulsion. Further works are nevertheless required to provide deeper understanding of the mechanisms involved to facilitate the development of an optimum system applicable to the industry.

Keywords: Energy; Microwave; Surface Characterization

Hard Magnet $Nd_2Fe_{14}B$ Nano Powders by Microwave Assisted Hydrothermal Synthesis

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Nd-Fe-B based magnets have excellent hard magnetic properties, especially high-coercivity; hence such magnets are widely used in numerous industrial and commercial products such as high magnetic anisotropy Nd-Fe-B based nanocomposite exchange spring magnets consist of an intimate mixture of the magnetically hard phase $Nd_2Fe_{14}B$ and a high saturation magnetization soft phase such as α-Fe. Due to non uniform grains that affect the exchange coupling the maximum value of energy product achieved experimentally is well below the theoretically predicted maximum energy product. Hence, novel synthesis resulting in homogeneous nanoscale grains can enhance the exchange coupling, improve the properties and extend the range of applications. Rapid synthesis of $Nd_2Fe_{14}B$ nano powders was achieved by microwave irradiation using glycine as the fuel source with chlorine or nitride as an oxidizer. A homogeneous mixture of the precursor was prepared with glycine through sonication. The precursor was evaporated, a low viscous gel was obtained and subjected to microwave irradiation in a modified domestic microwave oven. The resulting homogeneous and ultra fine multi component amorphous oxide powders were then further reacted to produce nanocomposite exchange spring magnets. $Nd_2Fe_{14}B$ based powders were produced through a reduction and diffusion reaction, in argon atmosphere, of CaH_2 and the amorphous oxide powders. The structural, morphological and magnetic properties were analyzed, XRD confirms the formation of $Nd_2Fe_{14}B$ and α-Fe phases. Better magnetic properties were obtained for samples prepared using the nitride oxidizer. The results show that homogeneous $Nd_2Fe_{14}B$ nanoparticles have been successfully produced by microwave hydrothermal route. The synthesis, characterization and hard magnetic properties have been studied and the results of $Nd_2Fe_{14}B$ production by microwave assisted hydrothermal process will be presented.

Keywords: Microwave; Magnetic Materials; Nanodevices

Microwave Materials Processing Research in Japan: Recent Activities

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Microwave heating was discovered sixty years ago, and has been applied to various fields. In materials processing, many studies were performed around 1990, as known from the fact that there were five MRS symposium meetings held between ’88 and ’96. It was at end of 1990’s, techniques of microwave processing have been extended to heating of metals for the full sintering. Further, the researchers gradually became aware of the importance of microwave magnetic field, which contributes not only to generating the induction heating but also to the heating of magnetic materials. This aspect opened a new direction in the field of microwave heating research. In Japan, many researchers in this field also became further active in last decade. One major project sponsored by Japanese government was awarded to a consortium of universities and research institutes, involving mainly the high temperature reaction fields excited by microwave energy. Along with this, many microwave focused symposium and research workshops were organized by Japan Institute of Metals (JIM) and Iron and Steel Institute of Japan (ISIJ). In this talk, selected topics of microwave heating of metals, such as microwave heating of metal glasses, metal thin films and metal/glass composite materials will be presented. Other trend of microwave research, recently becoming popular in Japan is its application to metal production. The microwave application is taken into consideration as an alternative method to meet the environmental problems, such as reducing CO_2 emission, and energy savings. Microwave iron making has been proposed. Because microwave is effective for heating of materials directly, internally and rapidly, its application to metal smelting, which is mostly a reduction (endo-thermic) reaction can be accomplished without supplying hot air (mostly by burning fuels: CO_2 producing process). Use of renewable energy for electric power generation for microwave heating is expected to be a key technology in this century. Handling of metal production wastes, such as slag, dust and sludge has been attempted in order to recycle metals from them. This talk will also introduce such activities of microwave researchers in ISIJ.

Keywords: Microwave; Ceramic; Energy

Microwave Processing of Tetrahedrally Coordinated- and Compound- Semiconductors

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The microwave processing of materials in multimode and single mode resonator cavities has opened a new vista of industrial applications and materials research due to its unique advantages. It is not only a rapid and energy efficient technique of sintering/calcination of different materials (including ceramics, composites, semiconductors and even metals) through precise and controlled volumetric heating, it can even result in phase transformation, new phase- and alloy- formation from elemental constituents. In this paper, a summary of our work on the microwave (MW) processing of tetrahedrally coordinated- and compound- semiconductors is presented. The starting materials are processed either in a multimode or in a single mode (H_{011}) and cylindrical MW resonant cavity at 2.45GHz at a suitable power and for different duration to obtain desired phases and dimensions.

Extremely long (several microns) and randomly oriented nanowires having nearly uniform diameter (less than 100 nm) have been grown by single mode MW processing of the pallets of Si powder for 5 minutes. Strong visible photoluminescence was observed in the synthesised nanowires. The carriers generated due to absorption of visible radiation undergo ballistic transport and radiatively recombine at interface thereby giving rise to photoluminescence. In another set of experiments, MW processing of Si and Ge mixture has resulted in the growth of a large quantity of $Si_{1-x}Ge_x$ alloy nanowires in a short duration of 5 minute. It is also found that processing time has an important role on the dimensions of nanowires. The XRD has confirmed the alloy formation, and XPS has revealed the presence of ultra thin oxide layer enveloping the nanowires. It has also been possible to prepare a polycrystalline $Zn_{0.95}Co_{0.05}O$ semiconductor devoid of Co clusters and exhibiting room temperature ferromagnetism as evidenced by XPS studies and magnetic measurements. The saturation magnetization and coercivity of these samples are found to be 0.6 emu/g and 4 mT, respectively.

Polycrystalline samples of $Si_{1-x}Ge_x$ alloys have also been synthesised in a single mode and multimode microwave chambers at ~ 1000°C in either argon or ambient atmosphere in 5 minutes. The carrier transport studies revealed that the increase of germanium concentration (x = 0.70) increases in the hole mobility in the resulting alloys. Such synthesis of Si-Ge alloys was not achieved by conventional method.

It is thus demonstrated that a simple, clean and fast technique of MW processing can be successfully employed to prepare polycrystalline and nano-dimensional samples of tetrahedrally coordinated- and compound-semiconductors.

Detailed results and their discussion will be presented in the talk.

Keywords: Microwave; Magnetic Materials; Semiconductor

Effects of Ceramic Crucibles on Properties of Microwave Sintering of Tin Based Alloy

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The effects of ceramic crucibles on the properties of microwave sintered tin alloys have been investigated in this study. Tin based alloy samples were subjected to microwave heating with the assistance of crucible. Sintering of green metal powder compact by conventional radiant heating technique is quite common and widely applied on the powder metal industry. The most recent development in sintering industry is the application of microwave energy to sinter metal products. In the microwave heating the energy directly couples with the material enabling rapid sintering often producing superior properties to the conventional product. A modified 2.45 GHz, 1100 W commercial microwave was used to sinter metal based materials. This technique has managed to obtain higher temperature after optimization of processing parameters that affect the material in absorbing microwave energy.

Principally, certain crucible materials can be classified as having the same function as a susceptor. It is used as an additional heating element to the sample and aids in microwave energy penetration into the body. The penetration depth of microwaves varies greatly varies from material to material. It is also dependent on many factors such as the dielectric and magnetic properties, microwave frequency and powder, temperature, conductivity, size and densification of the materials. Performance of tin based alloy was evaluated by using different types of crucibles inside the microwave sintering cavity and heated at 220°C temperature. Zirconia (ZrO_2), Alumina (Al_2O_3) and Quartz (SiO_2) crucibles were used in this study. There are a few reasons for choosing these materials such as their dielectric constants, thermal and mechanical properties. These materials have the capacity to withstand high temperatures without melting or decomposing and inert when exposed to severe environments. In addition, the ability to provide thermal insulation and resistance to thermal shock are often important considerations.

The tin based alloy powder was compacted at room temperature into a pallet of 20 mm diameter, followed by microwave sintering for 10 minutes reaching a temperature of 220°C. Sintered samples were characterized for their microstructure, hardness and densification properties. Mechanical behavior of tin based alloy was found to be superior when using ZrO_2 crucible. Finer grain size, 10% higher density and hardness were obtained by using ZrO_2 compared to other crucibles. Zirconia is an extremely refractory material and has a relatively higher thermal shock resistance, up to 2100°C with dielectric constant of 12.4. When comparing the heating time, SiO_2 crucible shows a significant lower heating time as compared to other crucibles. Microwave sintering assisted ZrO_2 crucible is able to sinter tin based alloy in 10 minutes at the temperature of 220°C. In SiO_2 crucible, total sintering time was increased by 33% to reach almost the same temperature as in ZrO_2 crucible. Moreover, the experimental

results signify that the crucible plays an important role in the sintering process by enabling the entrapment of heat.

Keywords: Microwave; Ceramic; Magnetic Materials

Enhance Degradation of Chlorobenzene in Aqueous Solution Using Microwave-Induced Zerovalent Iron and Copper Particles

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Microwave is applied to reduce the activation energy of chlorobenzene (CB) in aqueous solution, and enhance its removal using nano-scale zero-valent iron (Fe^0) or copper (Cu^0) particles as the dielectric media. Laboratory data demonstrate that the zero-valent iron or copper particles in CB solution will achieve better CB removal if exposed to 250 W MW for 300 sec than without MW irradiation. When Fe^0 and Cu^0 particles absorb MW energy, the electrical potential difference generated causes the metal electrons to rotate faster thus producing more heat. The MW-induced iron and copper particles reduce the CB activation energy by 6.1 kJ/mol (13.3 kJ/mol vs. 19.4 kJ/mol for Fe^0) and 5.4 kJ/mol (15.8 kJ/mol vs. 21.4 kJ/mol for Cu^0), and enhance the CB removal 4.1 time (82.8% vs. 20.4%) for Fe^0 and 3.7 time (72.1% vs. 19.5%) for Cu^0. Fe^0 has a higher standard reduction potential than Cu^0 (E^0 = +0.447 V vs. E^0 = -0.34 V). Hence, it is capable of removing more CB than Cu^0 (82.8% vs. 72.1%). Using the microwave induced nano-scale iron or copper particle is effective in treating toxic organic substances as demonstrated in this laboratory study.

Keywords: Microwave; Zero valent Iron; Zero valent Copper; Chlorobenzene (CB); Activation energy (Ea)

Microwave Research Activities in Europe

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Microwave, electromagnetic waves with wavelengths ranging from 1 mm to 1 m, or frequencies between 0.3 GHz and 300 GHz, and radiofrequencies, 3 Hz to 300 GHz, have been widely used to process materials, produce remote sensoring devices and activate plasma generation since many years.

The presence of many active research groups in Europe is very wide and encompasses academia, commerce, domestic as well as industrial applications. Historically the countries with dated activities are France, Germany, Sweden and UK, recently followed by Spain, Italy, Belgium and Switzerland. There is also a well established cooperation between industry and academic institutions as many joint patent and products developments may testify.

The European AMPERE- Association for Microwave Power in Europe for Research and Education is active in organizing biennial conferences, workshops, technical committees, periodical publications, specialist conferences and meetings and educational programmes.

The most recent developments are in the area of process intensification, chemistry, plasma treatments, cultural heritage preservation, nanotechnologies and pharmaceutics.

In particular a new method was optimized to obtain a basic molecule for pharmaceutical products without solvent and catalyst, within times reduced between 5 and 10 times. The extremely fast kinetics matches the requirement of modern industrial chemical processes which look for efficiency and green chemistry as well. Efficient essential oil extraction or bio-diesel production has been optimized under microwave irradiation, the latter with appropriate catalyst design. Continuous flow large size reactors have also been designed and developed for recycling exhausted catalysts- in this case column filling was extremely important.

Concerning chemistry, microwaves have been proved to be particularly active either in solution chemistry and in gas phase reaction as novel routes to fabricate advanced materials (nanoparticles, films, composites, nanostructures). In this field also research lab equipments have been optimized for continuous flow reaction in inorganic as well as organic chemistry.

New plasma sources or complex submicronic to nanometric structures preparation are possible thanks to newly patented devices active at MW frequency.

Very recently some Italian applicators industries and research laboratories have been active in designing and manufacturing of systems for pest control treatments in reverberation chambers. Not only cereals and legumes, but also many wooden, paper and textiles objects with historical or artistic interest are subject to the deterioration due to the infestation by various biological forms. The treatments, non-invasive, maintain the pieces integrity as proved already on different masterpieces of Italian art.

Keywords: Microwave; Energy; Ceramic

Microwave Sintering of Refractory Metals/Alloys: W, Mo, Re, W-Cu, W-Ni-Cu and W-Ni-Fe Alloys

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Refractory metals and alloys are well known for their high mechanical properties which make them useful for wide range of high temperature applications. However, owing to the refractoriness of these metals and alloys, it is very difficult to

consolidate these alloys and consequently P/M processing is the only viable manufacturing technique. One of the constraints in conventional sintering is long residence time which results in undesirable microstructural coarsening. This problem gets further aggravated when using smaller (submicron and nano) powder sizes material. Furthermore, conventional heating is mostly radiative, which leads to non-uniform heating in large components. This review article describes recent research findings about how these refractory metals and alloys (W, Mo, Re, W-Cu, W-Ni-Cu and W-Ni-Fe) have been successfully consolidated using microwave sintering and a comparative study with conventional data has been made. In most cases, microwave sintering resulted in an overall reduction of sintering time of up to 80%. This sintering time reduction prevents grain growth providing finer microstructure and as a result better mechanical properties have been observed.

Keyword: Microwave

Study of Mechanical Properties of Microwave Processed YSZ

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The microwave technique has been employed for the fabrication of partially yttria stabilized zirconia (YSZ) a solid electrolyte. The precursors of YSZ, $Zr_{1-x}Y_xO_{2-x/2}$, of composition x = 0.02, 0.04, 0.06, 0.08 and 0.1 have been prepared by mixed oxide method by mixing monoclinic zirconia and yttria in stoichiometric ratio. The consolidated cylindrical pellets have been sintered in microwave energy of frequency 2.45 GHz. This precursor behaves as reactive powder in the presence of microwave energy and is able to produce high density sintered YSZ. The effect of dopant concentration on stabilization and sintering of zirconia with variation in sintering time and temperature has been studied. It has been found that the highest densification occurs in the $Zr_{1-x}Y_xO_{2-x/2}$ of composition x = 0.08 and 0.1. The YSZ of same compositions have been prepared by conventional processing also. The variations in Vickers hardness of microwave processed YSZ with composition have been also studied. The results of microwave processed YSZ have been compared with those of conventionally processed products of identical composition and have been found that Vickers hardness of microwave processed products are quite higher than those of conventionally processed products.

Keywords: Ceramic; Microwave

POSTER SESSION

Microwave Processing of Calcium Doped Ceria (CCO) an Electrolyte for Intermediate Temperature Solid Oxide Fuel Cells

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In the present work chemically homogenous calcium doped ceria (CCO) electrolyte of composition $Ce_{0.8}Ca_{.02}O_{1.8}$ has been prepared at 1300°C in soaking time period of within 15 minutes using microwave energy of 2.45 GHz. The precursor of the CCO has been prepared by mixing cerium oxide and calcium carbonate in stoichiometric ratio corresponding to the composition $Ce_{0.8}Ca_{.02}O_{1.8}$. It is reactive sintering where formation of solid solution of CCO and sintering occurs simultaneously. In order to minimize the minimum temperature of formation of CCO, the reaction kinetics have been studied at different temperatures (1100-1400°C) for various time periods(5-20 minutes). The CCO of the same composition has been prepared by the conventional processing at 1400°C in soaking time period of 4 hours. The formation of CCO at lower temperature, during microwave processing, has been explained by the “anisothermal” heating. Study of variation in density with temperature and time shows that the maximum density of CCO, 98% of its theoretical density, is obtained at the sintering temperature of 1300°C and soaking time period of 15 minutes. The electrical and mechanical properties of the sintered products have been measured and correlated with their microstructures. The properties of microwave processed products have been compared with those of the products prepared by conventional processing.

Keywords: Ceramic; Microwave; Fuel Cells

Improvement in Electrical Properties of Microwave Processed YSZ

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The potentiality of microwave energy has been evaluated in fabrication of $Zr_{1-x}Y_xO_{2-x/2}$ of composition x = 0.06, 0.08, 0.10, 0.12, 0.14 and has been compared with the conventional processing. In this study, precursors of $Zr_{1-x}Y_xO_{2-x/2}$ have been prepared by mixed oxide method. Though the distribution of constituents in the samples obtained by the mixed oxide method is at particulate level, the microwave energy has been able to fabricate almost single phase YSZ of composition $Zr_{0.96}Y_{0.14}O_{1.93}$ within 20 minutes of soaking time period. The single phase YSZ of the same composition could not be prepared by conventional

processing even in four hours of soaking time period. $Zr_{1-x}Y_xO_{2-x/2}$ for composition x = 0.06, 0.08, 0.10, 0.12 obtained by either microwave processing or conventional processing have mixture of phases (monoclinic, tetragonal and cubic). The density of the microwave processed YSZ of each composition is higher than that of conventionally processed YSZ. Due to the higher monoclinic phase and low dissolution of $YO_{1.5}$ in conventionally sintered YSZ for the compositions $Zr_{1-x}Y_xO_{2-x/2}$ (x = 0.06, 0.08, 0.10) the grain growth does not occur in these samples and their microstructures have crack like pores which is reflected in their poor densities also. The microwave processed products of the corresponding compositions have inhomogeneous microstructure, but no crack like pores are observed. The bulk electrical conductivity of the microwave processed $Zr_{1-x}Y_xO_{2-x/2}$ of each composition is higher (~10^{-3} $\Omega^{-1}cm^{-1}$) than that of the conventionally processed products (~ of 10^{-4} $\Omega^{-1}cm^{-1}$) of the corresponding composition.

Keywords: Ceramic; Fuel Cells; Microwave

Microwave-assisted Production of Nanoporous Solid Acid Catalysts Derived from Waste for Biodiesel Production

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Microwave-assisted sol-gel synthesis of metal (Al and Cu) incorporated mesoporous silica (MCM-41) as solid acid catalysts for the production of biodiesel where the Si and Al sources were derived from municipal waste, namely coal fly ash (CFA) were successfully attempted. The Si and Al sources were derived directly from the supernatant of the waste while copper source was from copper salt. The X-ray diffraction patterns of solid acid catalysts microwave-irradiated at 40 min exhibited ordered 2-dimensional hexagonal structure even after calcinations as compared to those irradiated at 30 min. The incorporation of active sites (metal) into the nanoporous silica was confirmed by the appearance of resonance signal attributed to AlO_4 bonding. In addition, energy dispersive X-ray spectra of calcined solid acid catalyst also indicated the existence of metal elements.

In order to study the effect of metal incorporation concentration on structural properties and catalytic activities of calcined solid acid catalysts, the Al/Si ratio was varied at 0.04 to 0.07 using aluminum salt in addition to the Al source derived from CFA supernatant. In addition, to study the effect of dual metal incorporation onto the silica framework of solid acid catalyst, Cu was incorporated at Cu/Si ratio = 0.01, 0.02, 0.03, 0.04 and 0.05. Both type of calcined solid acid catalysts exhibited high surface area at ca. 690 m^2/g to 750 m^2/g. The median pore diameter estimated by the Dollimore-Heal (D-H) method were at ca. 2.2 nm to 2.5 nm with the pore wall thickness increased as the metal/silica ratio incorporated into the nanoporous silica framework increased.

Keywords: Microwave; Nanofabrication; Energy

Microwave Synthesis of Vanadium Nitride for Industrial Applications

Jia-Wu Huang

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Microwave carbothermal nitridation was used to prepare vanadium nitride from vanadium pentoxide and carbon black, under flowing nitrogen gas at atmospheric pressure. The effect of synthesis temperature, soaking time and the initial concentration of carbon on the final nitrogen content was studied. The nitrogen content in the products reached 12 to 16 weight percent, well within the current requirements of the steelmaking industry.

Keywords: Microwave; Ceramic

Synthesis of High Performance Magnesium Nanocomposites Using Powder Metallurgy Technique Incorporating Hybrid Microwave Sintering

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The current study addresses the development and synthesis of high performance magnesium nanocomposites using blend-press-sinter powder metallurgy route followed by hot extrusion. For sintering of materials, innovative hybrid microwave sintering method was used instead of conventional sintering to realize time and energy savings. Magnesium nanocomposites were synthesized using ceramic (yttria) nanoparticulate reinforcement. Magnesium hybrid nanocomposites were prepared with simultaneous addition of ceramic (yttria) and metal (copper and nickel) nanoparticulates into pure magnesium. Mg/Yttria nanocomposites showed an increase in both strength and ductility of magnesium. Mg/(Yttria+Copper) and Mg/(Yttria+Nickel) hybrid nanocomposite systems realized a significant improvement in 0.2% YS and UTS as compared to pure magnesium and Mg/Yttria system. All nanocomposite systems achieved better deformability than that of pure magnesium. Characterization studies indicated the influence of microstructural features such as grain refinement and good distribution of reinforcements and intermetallic phases on improved tensile properties of synthesized nanocomposites.

Keywords: Light Materials; Microwave

Microwave-assisted Carbon-carbon Bond Formation Reaction Catalyzed by Palladiummodified Mesoporous Materials under Solvent-free Condition

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The development of synthetic methods for carbon-carbon bond formation has been aided by the advancement of transition metal catalysis. The palladium-catalyzed cross-coupling reaction has been received increasing attention to the carbon-carbon bond in a single operational step. In this presentation our recent results of the Suzuki reaction, Heck Reaction and Sonogashira reaction catalyzed by palladium-modified mesoporous materials under solvent-free condition are described. It has been found that the microwave-assisted cross-coupling reactions catalyzed by palladium-modified mesoporous materials, such as Pd-SBA-15, Pd-MCM-41, or Pd-VSB-5, under solvent-free condition are performed in good to excellent yield and the reaction time is reduced exceedingly. The results of thermal palladium-catalyzed cross-coupling reactions under solvent-free condition will be also presented. The microwave-assisted organic reaction reduces reaction time and increases the yield compared with the thermal reactions. Application of the microwave-assisted reactions catalyzed by palladium-modified mesoporous materials to natural product synthesis is under investigation in our research group.

Keywords: Microwave; Heterostructures; Carbon

A Benchmark Study of Modified Microwave Novel System for Engineered Polymer Dissolution Process

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The aim of this research to investigate the benchmark study of novel modified microwave system for engineered polymer dissolution process. A modified microwave technique (MW) was applied to prepare the polyethersulfone and dimethylformamide (PES/DMF) casting solution for membrane fabrication. Fortunately, most of the synthetic polymeric membrane materials are polar and dipolar which is a very important factor in microwave processing. This fact provides a prospective approach for an efficient interaction between the electromagnetic field and dipolar materials for membrane process. Thus, the system performance was evaluated which included calibration of the maximum microwave output power and determination of the microwave distribution in the modified cavity. The performance of modified microwave was assessed on PES/DMF dope solution in terms of power absorbance, volume rate of heat generation and dielectric loss. It was observed that modified microwave could be successfully used for PES/DMF dissolution with the maximization of power efficiency. It was observed that the maximum output power of modified microwave to be 870 W, which is 93.4% with water and 510.27 W, which is 56.64% of nominal value of 900 W with PES/DMF dissolution.

Keywords: Energy; Microwave; Surface Characterization

Optimisation of Microwave Processing of Materials by Design of Experiments (DoE) Techniques Coupled to Numerical Simulation

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DoE techniques can be successfully applied to numerical simulation, in order to reduce the number of simulations runs and to investigate non trivial interactions among the variables. In this case, a series of "virtual experiments" is performed, and the simulation results are used as a measure of the expected results. This approach is particularly useful when a large number of physical experiments are not feasible, due to lack of time or high costs, and when optimisation procedures involve many interacting variables and multiple conflicting objectives.

In this paper, DoE techniques and numerical simulation using a commercial FDTD software have been used to optimize the binder removal from metal injection molded (MIM) and ceramic injection molded (CIM) parts, as well as to design an innovative plasma source for materials processing.

The software Design Expert v.6 was used to reduce the number of virtual experiments needed to gather information regarding the optimisation, in terms of speed, heating homogeneity and energy efficiency of the early stages of the microwave assisted debinding of MIM/CIM parts in an existing 2-feeds multimode applicator operating at 2.45 GHz. The software Concerto 4.0 (Vector Fields, U.K.) was used to perform the virtual experiments. The obtained model was used to determine the optimum debinding conditions, which were experimentally tested in the multimode applicator, operating at 2.45 GHz. Sample characterisation, before and after sintering, was used to confirm the validity of the obtained results.

A novel plasma source was designed optimizing the waveguide-to-coaxial transition and the nozzle geometry, considering a variable load in terms of electron density number of the microwave-generated plasma. Coupling DoE techniques and numerical simulation allowed to identify non trivial correlations between the microwave applicator geometry and the energy efficiency of the whole system. A prototype of the plasma torch is currently under testing and development.

Keywords: Microwave; Ceramic; Heterostructures

Enhance Degradation of Chlorobenzene by Microwave-Induced Through Zerovalent Iron: Particles Effect and Activation Energy

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Microwave (MW) is applied to enhance chlorobenzene (CB) removal using micron-scale iron particles (Micro-Fe), nano-scale zero-valent iron particles freshly prepared in our lab (Pre-Fe^0), and commercial nano-scale zero-valent iron particles (Comm-Fe^0) suspended in the CB solution as the dielectric media. The results show that for all types of zero-valent iron particles, better CB removal can be achieved when the CB solution is irradiated with 250 W MW for 150 sec than without MW irradiation. The MW radiation penetrates the CB solution to reach the Fe^0 surface for increasing iron oxidization rate and surface activity, thus enhancing the CB removal. The MW-induced iron particles cause the CB activation energy to drop 34.0% (32.7 kJ/mol vs. 49.5 kJ/mol for Micro-Fe), 15.1% (18.6 vs. 21.9 kJ/mol for Pre-Fe^0) and 16.1% (18.3 vs. 21.8 kJ/mol for Comm-Fe^0), and are capable of removing 13.6 times (Micro-Fe), 2.8 times (Pre-Fe^0) and 3.6 times (Comm-Fe^0) more CB. Using the microwave induced nano-scale iron particles is effective in treating toxic organic substances as demonstrated in this laboratory study.

Keyword: Microwave

Fast Crystallization of Microwave Annealing with Carbon on Amorphous Si Film

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Crystalline silicon (c-Si) thin film is extremely important for low-cost, high performance Si-based devices, such as thin-film transistors and solar cells. We demonstrate that microwave irradiation with carbon is able to fast crystallize amorphous silicon (a-Si) film on glass substrate at a short annealing time, less than 300 s, without the help of a metal capping. This is extendable to produce c-Si on various substrates such as glasses, on a large area base. The reason of fast crystallization is attributed to the extra thermal energy supplied by the microwave irradiation due to the dielectric properties of the heated a-Si, and facilitated nucleation of Si-crystallite due to enhanced atomic vibration by microwave irradiation.

Keywords: Microwave; Thin Films

Effects of Microwave Sintering on the Sinterability of Hydroxyapatite for Clinical Applications

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The sinterability of synthesized hydroxyapatite (HA) samples by microwave sintering was investigated over a temperature range 1000°C to 1300°C and compared with that prepared by conventional sintering. The nanocrystalline HA powder used in the present work was synthesized using a novel wet chemical precipitation technique. The studies have shown that microwave heating was beneficial and sintered bodies can be produced in very short sintering cycle when compared to conventional sintering. Decomposition of HA phase was not observed in the sintered samples regardless of sintering conditions. Although the relative density of microwave-sintered HA was slightly lower than the conventional-sintered HA throughout the sintering regime employed, taking into account the heating and soaking periods, the time taken by microwave sintering to achieve a relative density of 96.5% was about 3% of the time consumed for samples consolidated by the conventional method. In addition, as the temperature increases, considerable grain growth was observed in the conventional-sintered HA. The maximum hardness of 7.21 GPa and 6.38 GPa was obtained for HA sintered at 1050°C by the conventional method and 1150°C by microwave sintering, respectively. The maximum fracture toughness measured for the microwave-sintered and conventional-sintered HA were 1.45 $MPam^{½}$ at 1050°C and 1.22 $MPam^{½}$ at 1000°C, respectively. The present work revealed that microwave sintering is more economical, as it requires about 1.8 kJ of energy as compared to 190.9 kJ for conventional sintering to consolidate dense HA body at 1300°C.

Keywords: Hydroxyapatite; Bioceramic; Microwave sintering; Conventional sintering; Mechanical properties

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Microwave Dental Sintering Furnace

SYNOTHERM's HAMiLab system that specially designed for dental laboratories for **90 minute sintering** of zirconia and other dental ceramics with high level of accuracy, control, and speed. Microwave energy coupling only on the dental ceramics without heating the entire chamber, typically **cutting energy costs by more than 90%** compared to conventional furnaces and offers the **lowest possible carbon footprint** for cleaner environment. More importantly, the result is a **denser, up to 20% stronger and white** finished products. Now, you will have less stress without having to continuously run your furnace during periods of inactivity.

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Maximum temperature	1550°C	
Microwave power	0.2 ~ 1.4kW variable	0.4 ~ 2.8kW variable
Cooling system	Air-cooled	
Overall dimensions L×W×H (mm)	600 × 490 × 520	700 × 595 × 620
Effective heating zone (mm)	95 × 95 × 30	125 × 125 × 50
Power supply	220VAC/50Hz	
Control system	Stores up to several user-defined process curves, 40 segments programmable for each curve.	

Model: HAMiLab-DS

- Maximum temperature: 1600°C
- Microwave power: 0.2 ~ 1.45kW variable
- Cooling system: Water-cooled
- Overall dimensions (mm): 650 × 650 × 550 (L × W × H)
- Effective heating zone (mm): 105 × 105 × 50 (L × W × H)
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SYNOTHERM Corporation was founded in 2002 to accelerate the introduction of high temperature microwave technology into the ceramics and metals industries. Since then, it has grown to produce technologically advanced microwave systems for a variety of industries that include mining, waste remediation, implantology, and PM, as well as technical and daily-use ceramics and metals. The company has distinguished leadership and a diverse staff that are all vested in enhancing the state of the art in microwave technology. The direct investment of academic knowledge from Microwave Processing & Engineering Center (MPEC) and the tireless work of so many researchers throughout the world, is raising microwave manufacturing science above radiant heating; and this noble work has manifest into a company with one vision: "To give breath to the New Flame of Creation."

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Synthesis and sintering of functional materials and devices
Sintering of powder metallurgy parts
Synthesis and calcining of chemical materials
Roasting of various metals and non-metallic minerals

HAMiLab-V3 Microwave System

HAMiLab-VI500 Microwave System

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