

## **Green Processing of Materials with Microwaves**

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### **Abstract**

Currently industries are looking for green processes to minimise carbon emission by reducing their energy consumption to make the products compete with the international standards in all respects. Microwave processing is one of the prominent processes which is green and offers many advantages like time & energy saving, rapid, volumetric, uniform & selective heating, better product quality, no warm-up & cool-down time required, etc. Realising these advantages, Pradeep Metals Limited (PML) established an Industrial Microwave Research Center (IMRC) for developing novel technologies with microwaves and convert potential applications to commercially viable, energy efficient, green processes for different industries. Some of the efforts are discussed in this paper.

**Keywords:** *microwave, materials processing, heating, sintering, green technology, powder metallurgy*

### **1.0 Introduction**

Microwave technology is relatively new and emerging fast as a cleaner route for materials processing and is capable of meeting growing demands for better performing and cheaper products in a variety of processing industries [1, 2]. Microwave heating is fundamentally different from conventional heating. During conventional heating, traditional heat sources like oil, gas or electrical resistance heating, heat material in two stages; during first stage, thermal energy is transferred to the exterior surface of the object via radiation and in the second stage, this energy gets transferred into the interior of the object through conduction or convection. This results in higher surface temperature than the interior until thermal equilibrium is achieved after soaking it for a long duration. On the other hand, in microwave heating, the heat is generated internally within the material due to the interaction of microwave photons at an atomic level with the atoms present in the material by different mechanisms. This

results in highly efficient and instantaneous uniform, internal, volumetric, and selective generation of heat within the material. It may be noted that microwaves are not a form of heat, but rather a form of energy that gets manifested into heat through their interaction with materials [3]. Various advantages of microwave processing reported in literature are:

- Precise, controlled, and selective heating of the objects,
- Cost saving due to lesser time and energy consumption,
- Better product uniformity, faster production throughput, due to instantaneous heating,
- Less floor place, reduction in wasteful heating of surrounding cavity,
- Cheaper and greener process,

These advantages attracted worldwide attention from academia and industries to exploit these advantages for varied applications. To introduce microwave technology to the Indian industry and understand their requirements, PML organized a workshop on “Microwave Technology for Materials Processing: A Promising Option for Tomorrow,” under the aegis of ASM International, India Chapter and Indo-US Science and Technology Forum, New Delhi, at Mumbai in February 2006. A total of 20 eminent experts from U.S. UK, China, and India, were invited to present Invited Papers. Based on the discussions and feedback from participants, IMRC decided to develop a few new processes required by industries. The list of technologies developed by IMRC are divided in two areas, ‘Low temperature processing’ (< 2000C), and ‘High temperature processing’ (200-18000C).

Low temperature technologies developed are:

- Baking of metal-matrix composites like friction pads used as brake liners or clutch liners,
- Rapid curing of resin bonded grinding wheels,
- Rapid curing of concrete pre-casts like paver blocks,
- Activated carbon from natural waste such as coconut shells,
- Pre-treatment of coal where moisture was selectively removed to the desired levels as well as improvement

in its grindability, calorific value, with favorable alterations in their ash characteristics fusion temperature),

- Mag-Microwave process for petrochemical plants for improving yield, and properties of diesel & kerosene which are being produced from Heavy Vacuum Gas Oil fractions.
- A novel process for disinfestation of food grains (rice, wheat)

High temperature technologies developed are:

- Sintering of Ceramic plates used as electrodes in solid oxide fuel cells
- Sintering of metallic and ceramic composites
- Sintering of powder metal base components made of SS, refractory metals (Tungsten), Cu+Cr etc.
- Transparent Ceramics - Preparation and sintering of ALON (Al-Oxy Nitride) and Spinals (Al-Mg Oxides)
- Sintering of porous ceramic filters for molten metals prior to casting
- Smelting-reduction of iron ore
- Purification of lead recovered from scrapped lead batteries

Some of these developed technologies are novel and offer unique advantages over conventional process. These inventions are patented within India as well as in few select international countries. Please refer **Table 1** for details:

**Table 1.** Patents from IMRC-PML

Patent title	Status
Batch or Continuous Process for Pre-treatment of Heavy Petro Bottom Stocks	Granted in India (No. 277603)
Microwave assisted reduction of iron ore fines to manufacture sponge iron	Granted in India (No. 309420)
Rapid curing of resin bonded grinding wheels	Granted in US US9873185, Japan JP6407149 China No. CN 104661795 B India No. 307720
Continuous process for baking of cured friction material using electromagnetic energy	Granted in India (No. 433437)
Processing of Goethite ore using microwave	Granted in India (No. 388468)
Microwave Composite Heating Furnace (Jointly with Chubu University, Japan)	Granted in Ukraine (No. 119264), Russia (No.2705701), Japan (No. JP2016-539843) Australia (No. 2015300579)

	and under examination in India
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More details about two technologies developed are discussed in the next part of the paper.

## 2.0 Pre-treatment of Coal

Coals are extremely diverse and heterogeneous and generally contain significant amounts of moisture, ash, sulfur, and mineral impurities, all of which detract from its current potential to be an efficient and clean-burning fuel. The presence of significant moisture in many coals results in inefficient burning, leading to the burning of more coal and the consequences of increased emissions of green-house gases. More importance is given for the reduction of nitrogen, sulfur and phosphorus from coal and relatively little emphasis has been placed on coal drying which directly results in burning lower amount of coal to efficiently generate the desired amount of energy with reduced emissions [4, 5]. Realizing this shortcoming, drying and upgrading of coal is gaining momentum globally and in particular in Asia-Pacific countries, due to the ever-increasing energy demand. IMRC decided to undertake this process development as one of their projects.

The initial aim was only to reduce moisture content in coal lumps from ~30% levels to about 10% which was simple. But, after analysing the microwave processed samples carefully, we realised that the process developed with microwaves offers much more benefits than only moisture elimination. It improved the quality of coal by retaining valuable volatile matters and converting some adverse mineral matter constituents present in the coal, into friendly (catalytic) ones due to their selective and differential interactions with the microwaves, thereby improving the ash characteristics. The table below (Table 2) indicates how the characteristic of coal altered by exposing it to microwaves at 800C for 15 minutes.

**Table 2.** Effect of microwave exposure on coal moisture and other fuel properties (3 to 50mm fraction)

Type of analysis	As received coal	MW treatment @80°C (15 min.)
Total moisture content	15.8	5.2
GCV (kCal/kg)	5575	6242
<b>Proximate analysis</b>		
Fixed carbon (FC) (%)	42.8	51.6
<b>Grindability Index</b>		
Hard Grove Index (HGI)	48	50
<b>Ash Characteristics</b>		

Initial deformation temperature ( $^{\circ}\text{C}$ )	1100	1160-1220
Softening Temperature ( $^{\circ}\text{C}$ )	1160	1230-1310
Ash fusion temperature ( $^{\circ}\text{C}$ )	1300	1480-1490

The study confirmed that microwave process is unique in this respect for treating high moisture coals with varied size fractions. It is interesting to note that the combustible constituents present in the coal are transparent to microwave radiations and hence, unaffected, while the heat sink components, moisture and ash are highly amenable to microwave radiations (due to their dielectric properties). The results confirm improvement in properties of coal like decrease in the moisture content and increase in the gross calorific value (GCV) and HGI of the coal. The increase in the ash fusion temperature is the most important finding of this study which will considerably ease the problem of deposition and melting of ash on burner tips facilitating smooth and better combustion in thermal power plant heat exchangers.

### 3.0 Powder Metal sintering through Microwaves

The most recent applications of microwave technology have been reported in processing of metallic materials specially in sintering, brazing/joining and melting. The earliest work of microwave interaction with metallic powders is reported by Nishitani [6] who reported that by adding few percent of electrically conducting powders such as aluminium, the heating rates of the refractory ceramics is considerably enhanced. Whittaker and Mingos [7] used the high exothermic reaction rates of metal powders with sulfur for the microwave-induced synthesis of metal sulphides. But in all these studies no sintering of pure metal or alloy powders was reported. It was only in 1998, in the Penn State laboratory, that the first attempt of microwave sintering of powder metals was attempted [8, 9]. Many commercial powder-metal components of various alloys and different shapes prepared by using different sintering techniques have been reported by researchers including microwave.

#### 3.1 Sintering of SS316 Powder

A successful attempt was made by IMRC for sintering of SS316 powder for converting it in to one of the commercial product of PML made by hot forging. The readymade SS316 powder was brought from Hoganas India Pvt. Ltd. This powder was mixed with binders and pressed into a complex shape. SS316 sintering was done

under different atmospheres for studying the effect of atmosphere on final properties. The results are listed below in Table 3.

The study indicated that metal powders like SS are sintered easily and effectively using microwave technique and can replace conventional hot metal forging in few areas thereby reducing process steps and generation of waste considerably [10]. However, the main constraint here is the price of the powder suitable for powder metallurgy.

**Table 3.** Effect of sintering atmosphere on density

	N <sub>2</sub> (92%) + H <sub>2</sub> (8%)	N <sub>2</sub>	Ar	H <sub>2</sub>
Green density (g/cm <sup>3</sup> )	6.64	6.89	6.79	6.76
Sintered density (g/cm <sup>3</sup> )	7.50	7.23	7.15	6.98
Radial Shrinkage (%)	0.66	1.32	Nil	0.38
Axial Shrinkage (%)	2.38	2.38	Nil	0.83
Hardness (R <sub>B</sub> )	74	67	63	65
Theoretical density 7.9 g/cm <sup>3</sup>				

#### 3.2 Sintering of Tungsten powder

Apart from SS, tungsten metal powder sintering was done for DMRL, Hyderabad [11]. In our joint study, as-received tungsten and activated tungsten powder were sintered in microwave system. The powder was isostatically compacted at 250 MPa into cylindrical compacts of 40 mm dia. and 40 mm height. Activation of powder was done by reducing its particle size using high-energy planetary mill. Both compacts received from DMRL were sintered in a 3 kW microwave furnace with multimode cavity to obtain uniform heating over the compacts. Microwave sintering was carried out in a reducing atmosphere (5% H<sub>2</sub> and 95% N<sub>2</sub>) between 1600-18000C. The sintered density of the sample prepared from as received powder was measured to be 15.8 to 16.4 g/cm<sup>3</sup> which is 82 to 85% of the theoretical density of tungsten. However, sample prepared from activated powder indicated higher density i.e. 17.5 to 18.0 g/cm<sup>3</sup> (90 to 93% of the theoretical density of tungsten). It is reported that for tungsten sintering conventionally high temperatures of about 25000C is used.

Both the above studies showed clearly that the metal powders respond to microwave very well. Using this technique, intricate shapes from powder metal can be

made easily by employing this technique at much lower temperature and with better properties.

#### 4.0 Concluding Remarks

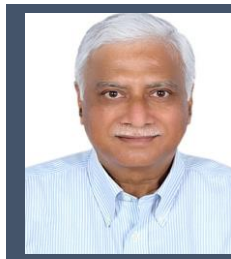
Above studies showed unique advantages of microwave processing for different industrial applications. Due to the fast and relatively uniform heating profiles, microwave heating promises an ideal means to heat bulk materials and shapes to consolidate particulate materials through uniform heat transfer. There are few more very interesting technologies developed by IMRC-PML using microwave processing, such as disinfestation of food grains, rapid curing of resin bonded grinding wheels and a green process of direct reduction of iron ore or industrial wastes to pig iron with minimum amount of coal and without use of coke thereby reducing greenhouse gas emission by almost 50%. These technologies will be discussed in detail in the next few issues of this journal.

It may be noted that “Blind application of microwave may lead to disappointment; However, wise application may have greater advantages than had been anticipated.”

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### **Comments**

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