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Microwave Processing of Metallic Materials

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Abstract

Microwave processing of materials is an emerging technology that offers a number of advantages to technocrats for processing a variety of materials. It is a new, powerful and significantly different tool to material processors to achieve new goals that may not be attainable by conventional means of processing. The most recent application of microwave technology has 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 have been reported by international researchers. The study indicated a variety of possible microstructures that can be obtained by microwave sintering of hard metals and metal based composites due to specific heating mechanisms. This presentation presents an overview of international activities carried out specifically in powder metal processing.

Introduction

Powder Metallurgy is a highly developed technique for manufacturing precision and complicated metal parts. This technique is relatively recent compared to casting and forging, but is well established and well understood. The process involves atomic motion via material diffusion driven by thermal energy. The material diffusion is a function of temperature and duration of the sintering process. The properties of the final sintered product are greatly dependent upon the microstructure, densification, grain size, phase composition and nature of the porosity present. The advantage of this process is its ability to produce large volume of highly toleranced metal components, cost effectively.

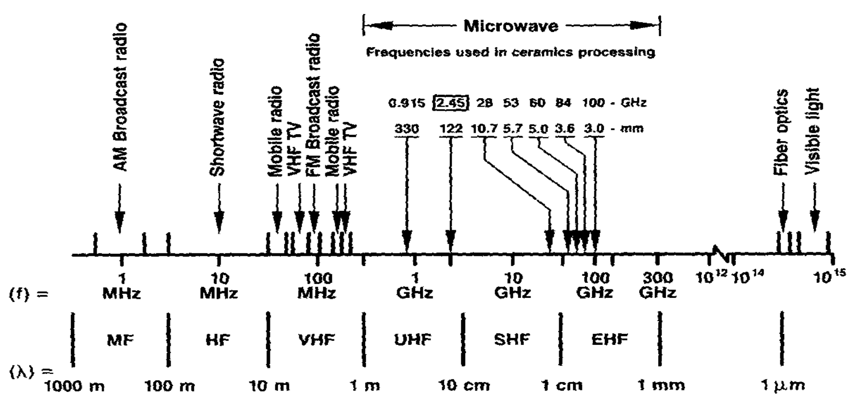
Traditional sintering processes for metallic materials consume considerable power. In many conventional powder consolidation processes, a major problem is that the time required for consolidation at high temperatures is quite long, and as a result, undesirable coarse microstructures with grain boundary impurity phases are invariably formed. Some components require post sintering treatments (such as HIP) and machining, which add to the processing costs. Finer microstructures and near theoretical densities in metallic components are still elusive and challenging. Manufacturers are looking for newer technologies and processes to meet these demands. For improving properties, new forming techniques are being introduced such as injection molding, HIPing, Spray forming etc. But still the ongoing debate has always centered on properties of the final products such as strength and size. As these properties are primarily based on the grain size and microstructure, the conventional sintering process is unable to deliver better product due to its inherent limitations. Microwave

Heating Technique is found to be much more useful at this stage due to its several anticipated advantages, namely: a) the process produces fine microstructures and improved properties due to the enhanced diffusion processes with concomitantly reduced sintering times, reduced energy consumption, no environmental hazards. It can practically be considered as environment friendly process. Another most important benefit is reduction in risk of thermal cracking as the parts undergo simultaneous volumetric heating and uniform shrinkage, and expectedly improved product performance.

As discussed above, the microwave heating is a fundamentally different type of heating process compared to the traditional heating processes adopted in industry. In conventional heating process, the material is heated with external source of heating, such as oil, gas or electrical resistance heating etc. where energy is transferred to samples mainly by radiation, and then by conduction. Whereas, in microwave processing, the heat is generated internally, within the material. This is due to interaction of microwaves at an atomic level in the material. This results in uniform, internal, volumetric and selective heating of the material. Although, Microwave heating was conceived over 50 years ago, it was commercially employed in low temperature applications such as drying of food and in rubber industry for vulcanization. But, during the past two decades, its high temperature applications gained momentum in ceramics and refractory in drying, binder-burnout and sintering etc. However, all these applications are at the verge of commercialization. Its application in metallurgy is fairly recent one.

Basics of Microwave

Microwaves are electromagnetic waves having frequencies in the range of 0.3 to 300 GHz corresponding wavelength ranging between 1m to 1 mm (Fig.1).



Electromagnetic spectrum and frequrncies used in microwave processing of ceramic materials

Figure 1: Electro magnetic spectrum and frequencies used in microwave processing of ceramic materials

The major application of microwaves is in communication. To avoid interference, only few select bands are allocated by Federal Communications Commission for Industrial, Scientific and Medical applications known as ISM band. Frequency bands allocated are 896, 915, 2450, 5800, 28000, 60000 and 84000 Hz. Out of these, 2450 Hz is used worldwide while others used by few selected nations.

In contrast with visible light, except for lasers, microwaves are coherent and polarized. They obey the law of optics and can be transmitted, absorbed or reflected, depending on the material type *[W.H.Sutton, Am. Ceram. Soc. Bull., 68 (1989)376, D.E.Clark, W.H.Sutton, "Microwave Theory and Applications in Material Processing, IV, vol.80, Eds. David Clark, Jon Binner, David Lewis, Am. Ceram. Soc., 1997,61, John Brooske, Reid Cooper, Samual Freeman, Kirill Raybakov and Vladimir Semenov, "Microwave Theory and Applications in Material Processing, IV, vol.80, Eds. David Clark, Jon Binner, David Lewis, Am. Ceram. Soc., 1997,143]*. Microwave interaction of materials could be broadly categorized in four different types.

i) Conductors i.e. metals having free electrons in bulk form are 'opaque' to microwaves and thus good reflectors.

ii) Insulators, where electrons do not flow freely are electrically non-conducting type materials such as ceramics (viz. Al_2O_3, MgO, SiO_2, glasses etc.) are 'transparent' to microwaves. But, electronic reorientation or distortions of induced or paramanent dipoles can give rise to heating.

iii) Dielectric materials possess the properties ranging from conductors to insulators. These materials are polarizable and have dipoles that reorient rapidly in response to changing electric field strength are amenable to microwave heating at low temperatures. Example of such ceramic materials are Co_2O_3, MnO_2, NiO, CuO, ZrO_2 etc. which are good 'absorbers' of microwaves at room temperature.

iv) In other materials such as composite materials the absorption of microwaves can be enhanced by adding conducting or magnetic materials. These additives absorb microwaves more rapidly than the matrix and hence material will be heated more rapidly and selectively.

Microwave heating

Microwaves are not forms of heat but rather forms of energy that are manifested as heat through their interaction with materials [*Robert Schiffmann, "Microwave Theory and Applications in Material Processing, IV, vol.80, Eds. David Clark, Jon Binner, David Lewis, Am. Ceram. Soc., 1997,41*]. As microwaves are electromagnetic waves the energy content and amplitude changes as it travels through a medium. They consist of electrical (E) and magnetic (H) components mutually perpendicular to each other in the direction of travel. A plane monochromatic electro-magnetic wave (microwave) is schematically presented in figure 2

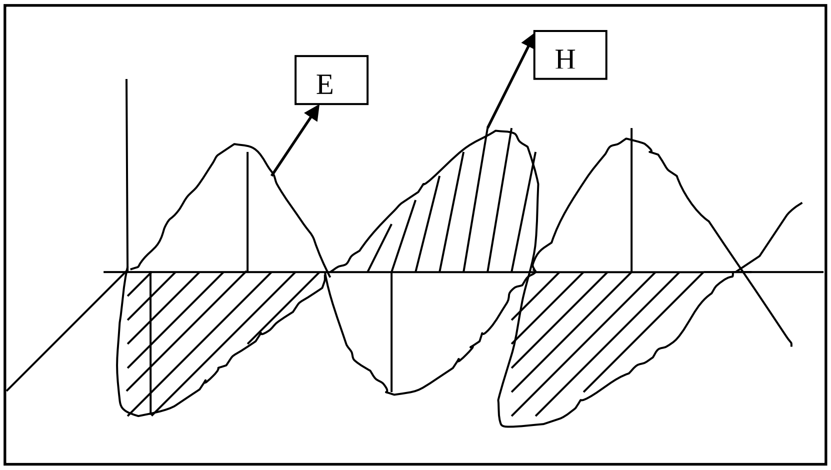


Fig. 2 : A schematic representation of an electromagnetic wave
(the electrical component (E), the magnetic component (H))

It can be seen (Fig. 2) that E component at some point is zero; then it builds up to a maximum value, decays to zero, and again builds up to maximum value with the opposite polarity before again decaying to zero. This periodic flip-flopping of the wave polarity (in both electric 'E" and magnetic 'H' component) and decay through zero causes stress in the ions, atoms, and molecules and the energy is lost within the material in the form of heat. Greater the field strength, the greater will be the effect. At the macroscopic level, the dielectric properties control the microwave processibility of a wide range of nonmagnetic materials.

Many mechanisms are reported in the literature explaining the conversion of microwaves into heat. Two important mechanisms are taken into consideration viz. conduction and polarization while heating of material. Polarization involves the short-range displacement of charge through the formation and rotation of dielectric dipoles (or magnetic dipoles if present) and is dominant at higher frequencies. Conduction requires the long-range (compared to rotation) transport of charge and is dominant at low frequencies [*D.E.Clark, W.H.Sutton, "Microwave Theory and Applications in Material Processing, IV, vol.80, Eds. David Clark, Jon Binner, David Lewis, Am. Ceram. Soc., 1997,61*].

The power dissipated in multimode systems is thus governed by the equation 1:

$$P = \omega\,(\varepsilon_0\,\varepsilon_{eff}''\,E^2 + \mu_0\,\mu''\,H^2) \qquad (1)$$

Where, P = power absorbed (W/m^3), ω = angular frequency = 2 πf where f is operating frequency, ε_0 = permittivity of free space (8.85 x 10^{-12} F/m), ε_{eff}'' = effective dielectric loss factor, E = internal electric field strength (V/m), μ_0 = permeability of free space, μ'' = magnetic loss factor, H = intensity of magnetic field.

Microwave system

A typical microwave system consists of three main components, a microwave generator, wave guides and applicator. Different types of microwave generators are used in system which depends on the frequency of microwaves. Types of generators are, Transmitting Power Grid (0.15- 3 GHz), Magnetrons (0.915-2.45 GHz), Klystrons (3-10 GHz), Gyrotron (28-140 GHz). Microwaves carry an equal amount of the electric and magnetic energy and the wave impedance remains constant in the direction of propagation. Microwaves can be divided into three types, first the transverse electromagnetic (TEM) wave, where all fields are transverse i.e. wave propagates between two parallel plates or in a coaxial line. Second type is the transverse electric (TE or H) wave where z component of the electric field is missing. Third is transverse magnetic (TM or E) wave where z component of the magnetic field is missing. These waves are carried to the applicator through waveguides, which are typically hollow having either a rectangular or a circular cross section and coated inside with conducting martial. Microwave applicators are devices that are designed to heat material by exposing to microwave field. These are designed to achieve a controlled interaction between microwaves and material under safe, reliable, repeatable and economically operating conditions.

Microwave Heating and its interaction with metallic materials

Microwave processing of materials was confined to ceramics, food, rubber and other inorganic materials till 2000. Metals or metallic materials were totally overlooked mainly due to the misconception that all the metals or metallic materials reflect microwaves and/or cause spark, plasma due to which only surface heating is possible.
Heating of metallic materials is the most recent and surprising development in the field of science & technology. This is being exploited in sintering various powder metal components ranging from small cylinders, rods, and automotive components and also in welding, joining, and brazing of metals, metal-ceramics.

This observation is evident from the conventional view shown in a plot (Figure 3) between microwave absorption in solid materials and electrical conductivity [B.P. Barnsley, "Microwave Processing of Materials," Metals and Materials, 5(11), 633, (1989)].

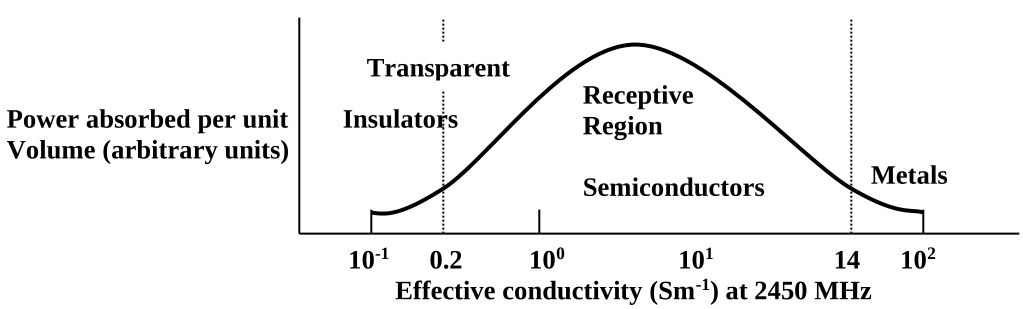


Figure 3: Microwave Energy absorption is a function of electrical conductivity .

It is evident from this plot that at room temperature only semiconductors should be good microwave absorbers, while ceramics/insulators should be transparent in microwave and the metals should reflect microwaves. Reflection/arcing/plasma phenomenon occur in the bulk metals at room temperature, due to attenuation of microwave radiation in a conductive medium that arises from the creation large potential difference in macroscopically large metal forms. This gives rise to discharges when the potential difference exceeds the break down electric resistance of the medium that separates the metal particles and the metal object [Metaxas, A.C. and Meredith R.J., Industrial. Microwave Heating, Peter Peregrinus Ltd., 1988]. Alternately, the electromagnetic field generated under influence of microwaves, allow loosely bond electrons to move and concentrate at surface, edges and points. This results in discharge of energy in the form of arcing. However, the researchers did not notice that this relation is valid only for sintered or bulk materials at room temperature, and not for powdered materials and/or for bulk at higher temperatures. The reason behind this is small particles do not generate sufficiently large potential differences for the above phenomenon to occur at room temperature. Now it has been proved that all metallic materials in powder form do absorb microwaves efficiently and effectively.

A depth at which the incident microwave power reduced by one half (due to attenuation of electric field generated within material by interaction with microwaves) is commonly known as skin depth or penetration depth. It is observed at 2.45 GHz that the skin dept in the bulk metals is very low (of the order of few microns), and hence very little penetration of microwaves takes place. Comparative values reported by T.Gerdes and M. Willert Porada [T.Gerdes and M. Willert Porada, Mat. Res. Soc. Symp. Proc., vol. 347, 531-537, 1994] at 2.4 GHz are listed below:

Table 1: Skin depth for 2.4 GHz microwave radiation

Material	Skin Depth (μm	Resistivity σ (μΩ cm)
Al	1.7	2.7
Co	2.5	6.3
WC	4.7	22
TiC	8.2	68
WC-6%Co Hard metal	4.3	19
Al2O3	4×10^7	10^{20}

However, in case of fine metal powders, rapid heating in the material as a whole can occur. A theoretical model developed recently predicted that if the metal powder particle size is less than 100μm, it will absorb microwaves at 2.45 GHz, and the maximum absorption occurs around 10-20 μm. It was further observed that the degree of microwaves absorption depends upon the electrical conductivity, temperature and the frequency. In magnetic materials other manifestations of the microwave coupling include hysteresis losses, dimensional resonances, and magnetic resonances from precession of magnetic moments of unpaired electrons [R. E. Newnham , S.J. Jang, M. Xu and F. Jones, Ceram. Trans. 21, 51, 1991].

International scenario in metal powder processing

The earliest work of microwave interaction with metallic powders is reported by Nishitani [T. Nishitani, "Method for sintering refractories and an apparatus therefor," US Patent # 4,147,911 Apr. 3, 1979] who reported that by adding few percent of electrically conducting powders such as aluminum, the heating rates of the refractory ceramics is considerably enhanced. Walkiewicz et al [J. W. Walkiewicz, Kazonich, G., and McGill, S. L., "Microwave heating characteristics of selected minerals and compounds," *Min. Metall. Processing*, 39-42, 1988] likewise simply exposed a range of materials, including six metals to a 2.4 GHz field, and reported modest heating and not sintering in the range from 120°C (Mg) to 768°C (Fe). Whittaker and Mingos [G. Whittaker, Mingos, D. M., "Microwave-assisted solid-state reactions involving metal powders," *J. Chem. Soc. Dalton Trans*. 2073-2079, 1995] 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 this laboratory that the first attempt of microwave **sintering** of powder metals (15). This was a radical discovery and now has opened up entirely new avenues of research and processing of metallic materials including sintering, melting, brazing, joining, and coatings etc._The mechanism of microwave-matter interaction is unexplained so far. It is also not determined if microwave sintering will work on all metals, alloys, and intermetallics or if there will be selective heating, as in the case of many ceramics.

Microwave Processing and Engineering Center of The Pennsylvania State University , USA reported that virtually all metals, alloys and intermetallics couple and heat efficiently and effectively in microwave field, and their green parts produce highly sintered bodies with improved mechanical properties. They reported sintering of commercial steel compositions such as FN208 and FC208 in 90 min. when sintered between 1100 to 1300 deg. C and soaking time 5-30 min. The products were highly sintered bodies with increase in mechanical properties (in FN 208, the MOR higher by 60%) [R.Roy D. Agrawal, J. Cheng, and S. Gedevanishvili, "Full sintering of powdered metals parts in microwaves", Nature**,** 399, 664 (June 17, 1999), R. M. Ankalekar, D.K. Agrawal, and R. Roy, "Microwave Sintering and Mechanical Properties of P/M Steel", Powder Metal. Vol. 44[4], 355-362 (2001), D.Agrwal, Proc. Powd. Metallurgy World Congress, Kyoto, Japan, 797-800, 2000].

Microwave sintered commercial powder metal parts by Microwave Processing and Engineering Center of The Pennsylvania State University, USA.

Researchers of University of Edinburgh, UK, studied the ability of metal powders in synthesis of metal chalcogenides and metal cluster compound using microwave technique. They reported efficient coupling of microwaves without arcing and selective nature of heating that resulted into range of solid state reactions [A.Gavin Whittaker and Michael Mingos, J. Chem. Soc.Dalton Trans., 2073-2079 (1995)].

Utilization of microwave sintering technique for pressureless fast sintering of metal based composites was reported by University of Dortmund,Germany. They reported sintering of tungsten carbide-cobalt based hard metal based composites150 deg. C lower than conventional sintering process. The composites indicated much higher mechanical properties due to reduction in closed and open porosity (<1%) at the sintering temperature of 1150 deg. C compared to conventionally sintered samples at 1300 deg. C (>5 closed and 25% open porosity respectively). They interpreted that microwave processing influences the mechanism of liquid phase sintering. The mechanism reported is local formation of eutectic liquid due to thermal non-equilibrium during microwave sintering, or an accelerated distribution of carbon, Co by evaporation and condensation throughout the sintered body due to ohmic and local plasma heating, followed by dissolution of carbon and W within parent crystals [T.Gerdes, M.Willert Porada, K. Rodiger, Mat. Res. Soc. Proc., vol.430, 45-50, 1996].

Edward B. Ripley and Jason Oberhaus of BWXT Y-12 National Security Complex, Oakridge, USA reported melting and heat treating of metals using microwave heating. Metals that have been melted and cast include steels, titanium, zirconium, uranium, copper, brass, bronze, aluminum, and many alloy systems. Melt size range up to 350 kg. They also reported three distinct methods to heat treat metals using microwave furnace: molten saltbath processing, granular suscepting media and fluidized bed process. Based on their results, it was concluded that microwaves can successfully heat treat metal components without any negative effects on the metal. Hardness values and microstructures were similar to those obtained using conventional heat treatment process.

Recently, seven SAIC (Science Applications International Cooperation, USA) personnel were recognized for their work in developing a new, safer, and cost-saving technology that uses a microwave furnace for casting enriched uranium. They received Award of Excellence "for demonstrating the technical superiority of microwave melting technology," according to the citation. All work was performed at the Y-12 National Security Complex in Oak Ridge, Tenn.. Its mission includes forming uranium parts for nuclear warheads and for fuel for the U.S. Navy's nuclear-powered submarine fleet.

Just as a conventional microwave oven combines quicker cooking times with lower energy use, Y-12's microwave furnaces offered a significant power savings over conventional induction furnaces, among several other advantages. The challenge was to take advantage of the efficiency of microwaves without the normal arcing problems. The other objective was not just to melt and cast metal but also "to see the effect of microwave melting on the quality of the molten metal. The analytical results indicated that the process significantly reduced the amounts of several key impurities. Aside from increasing purity, compared to conventional induction furnaces, microwave melting was found to be cleaner and safer for the workers because it reduces their risk of radiation exposure. It is also more economical because it requires significantly less power to generate higher temperatures. In addition, a microwave furnace takes up significantly less space than the more conventional furnaces . A crucible is "the actual melting pot for the metal used in the microwave process was a special microwave-susceptible materials, and unique-design crucibles and molds to melt and cast the metal. The crucible absorbs the microwaves rather than reflecting them, the crucible heats up, and melts the metal. The large amount of microwaves never reach the metal to cause arcing.

Concluding remarks

Based on the experience of international scientists, following benefits of microwave processing could be summarized:

1) Cost savings due to dramatic reduction in processing time and energy.
2) Precise, controlled, instantaneous and selective heating.
3) Volumetric and uniform heating.
4) Improved quality and properties.
5) Environment friendly process as it is clean, and quiet process that reduces hazardous emissions,
6) Synthesis of new materials is possible
7) Processing of materials not possible with conventional means can be processed.

Considerable work and investment has been made over many years for developing microwave processing systems for a range of products. But, most of them are by small industrial the microwave companies working closely with the user company where the user gets a proprietary process and microwave company gets exclusive manufacturing rights. As a result, much of the technology that has developed is not freely available, and the ‘wheel’ has to get reinvented over and over again. Other significant reasons responsible for slower growth are :The complexity and cost of the equipment, Limited applicability, The inherent inefficiency of conversion of electric power, Need to perform expensive feasibility studies,

To overcome these, it is essential to apply multidisciplinary approach in which different fields such as chemistry, physics, electrical & mechanical engineering, processes engineering, material science etc. are intermingled properly. One must recognize that any sample cannot be heated efficiently and uniformly by simply exposing in microwaves without considering specific microwave-materials interaction. To obtain proper results, it is essential to understand

a) process requirements
b) thermo-chemical properties of material
c) type of equipment required for desired application
d) designing of applicators to suitable for application
e) interaction of material and microwave field with in cavity
f) process economics.

In one sentence, the real key to success is to understand the basics with respect to microwave heating.

Please Note:

A research facility "INDUSTRIAL MICROWAVE RESEARCH CENTER" has been established by M/s Pradeep Metals Ltd. at Navi-Mumbai, to carry out applied research for real life requirements for Indian industry and to convert potential applications to reality. The center has established a laboratory set up where around 300 g sample can be treated up to 1100 deg. C using microwave technique for preliminary exploratory trials. A pilot-scale Microwave system with 3 KW power is also installed for conducting scale up trials (~2 kg) up to 1600 deg. C under normal/controlled atmosphere. We welcome you to visit us and discuss in details about your ideas. We also undertake designing and supply of microwave system as per client's requirements.