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MICROWAVE SINTERING, BRAZING AND MELTING OF METALLIC MATERIALS

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Abstract

Microwave energy has been in use for variety of applications for over 50 years. These applications include communication, food processing, wood drying, rubber vulcanization, medical therapy, polymers, etc. In the last two decades microwave heating has been also applied very effectively and efficiently to heat and sinter ceramic materials. Microwave heating is recognized for its various advantages, such as: time and energy saving, very rapid heating rates, considerably reduced processing cycle time and temperature, fine microstructures and improved mechanical properties, better product performance, etc. The most recent application of microwaves has been in the field of metallic materials for sintering, brazing/joining and melting. Several common steel compositions, pure metals and refractory metals have been sintered in microwaves to nearly full density with improved mechanical properties. Many commercial powder-metal components of various alloy compositions including iron and steel, copper, aluminum, nickel, Mo, Co, Ti, W, Sn, etc., and their alloys have also been sintered in microwaves producing better properties than their conventional counterparts by using a 2.45 GHz multimode microwave system. This work has been further extended to join and braze bulk metal pieces, especially super alloy based turbine blades. Further, in a specially designed microwave cavity, even the bulk metals can be made to couple with the microwave field and melted. The implications of these findings are obvious in the field of powder metal technology.

Introduction

Microwave energy has been in use for over 50 years in a variety of applications such as communications, food processing, rubber vulcanization, textile and wood products, and drying of ceramic powders. Widespread use of microwave home ovens has in fact revolutionized home-cooking. The use of the electromagnetic spectrum in the "microwave" region for many energy-intensive technologies has been led by the consumer acceptance of microwave home ovens. Use of microwave technology in material science and processing is not rather new. The areas where it has been applied include: process control, drying of ceramic sanitary wares, calcination, and decomposition of gaseous species by microwave plasma, powder synthesis, and sintering of oxide ceramics and some non-oxide systems [1-4]. Microwave technology is attractive because it has many obvious advantages when compared with conventional methods, such as: very short cycle time resulting in energy savings as high as 90% over conventional methods, rapid heating rates, finer microstructures, and hence, improved mechanical properties and environmental friendliness [4].

Researchers in academia and industry have been working in the area of microwave processing of a variety of materials for many years. Most of this work is confined mainly in the area of ceramics. However, now it has been shown that the microwave energy can, in fact, be used to sinter virtually all powdered metals as efficiently and effectively as in the ceramic systems. This has opened up an entirely new research area to investigate the advantages of microwaves for metallic materials to meet the challenging and growing needs in many metallurgical applications. This paper describes the latest developments in the area of microwave processing of metallic material, especially at the Penn State University in the last few years.

In many conventional methods involving resistant/radiation/convection heating, the thermal energy is absorbed on the surface of the work-piece and then it is transferred towards the inside via thermal conductivity; so there is an energy transfer through the thermal conductivity mechanism in these methods, and therefore the process is slow. Such methods are not very energy efficient. On the other hand in case of microwave (and RF-induction) heating, the electromagnetic energy is absorbed by the material as a whole (also known as volumetric heating) due to microwave-matter coupling and deep penetration, and then is converted in to heat through dielectric (in case of ceramics), magnetic permittivity/eddy currents (metals) loss mechanisms. Since there is an energy conversion and no thermal conductivity mechanism involved, the heating is very rapid, uniform and highly energy efficient. These two processes are fundamentally different in their heating mechanisms, and hence often result in a vastly different product.

Due to the internal heating in the microwave processing, it is possible to sinter many materials at a much lower temperature and shorter time than required in conventional methods. The use of microwave processing reduces typical sintering times by a factor of 10 or more in many cases, thereby minimizing grain growth. Thus, it is possible to produce fine microstructure in microwave sintered metal parts.

Microwaves are electromagnetic radiation with wavelengths ranging from about 1 mm to 1 m in free space and frequencies between 300 GHz to 300 MHz, respectively. However, only very few frequency bands in this range are allowed for research and industrial applications to avoid interference with communication. The most common microwave frequency used for research is 2.45 GHz ($\lambda \sim 12.25$ cm), the same as for the domestic microwave ovens; the other allowable frequencies are 915 MHz ($\lambda \sim 32.8$ cm), 30 GHz ($\lambda \sim 1$ cm) and 83 GHz for some specific applications.

The use of microwaves in the sintering of ceramic materials is relatively new. The first reported use of microwave in ceramics goes as far back as in 1968 [5], however, the real activity in the field picked up momentum only in the late 1970s and continued with great enthusiasm and excitement in the 1980s. Several earlier excellent reviews by Clark and Sutton [1], Schiffman [2], Katz [3], Sutton [4, 6], have summarized the status of microwave processing research till 1996. During this period, besides the full commercialization of microwave drying and food processing, the microwave heating and sintering of traditional and special/advanced ceramics, composites, and glass ceramics: alumina, uranium oxides, silica, zeolites, barium titanates, ferrites, glass-ceramics, hydroxyapatite, etc., were investigated. Two recent reviews [7, 8] have summarized the latest developments in the field of microwave sintering and synthesis of inorganic solids. Some of these latest developments include microwave sintering of cemented tungsten carbide [9-13], development of transparent ceramics [13, 14], and the sintering of metallic materials [15, 16]. This paper briefly summarizes the work on metallic materials performed at the Penn State University.

Microwave Processing of Metallic Materials

Microwave processing of materials was mostly confined until 2000 to ceramics, semiconductors, inorganic and polymeric materials. There have been very few detailed reports on microwave processing of metals. The main reason for this lack of work in microwave heating/sintering of metals was due to the misconception that all metals reflect microwave and/or cause plasma formation, and hence cannot be heated, except exhibiting surface heating due to limited penetration of the microwave radiation. This observation is evident from the conventional view shown in a plot (Figure 1) between microwave absorption in solid materials and electrical conductivity [17]. It is evident from this plot that only semiconductors are good microwave absorbers, ceramics/insulators are transparent in microwave, and the metals should reflect microwaves. 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 at higher temperatures. Now it has been proved that all metallic materials in powder form do absorb microwaves. The cause for this phenomenon is not yet very well explained.

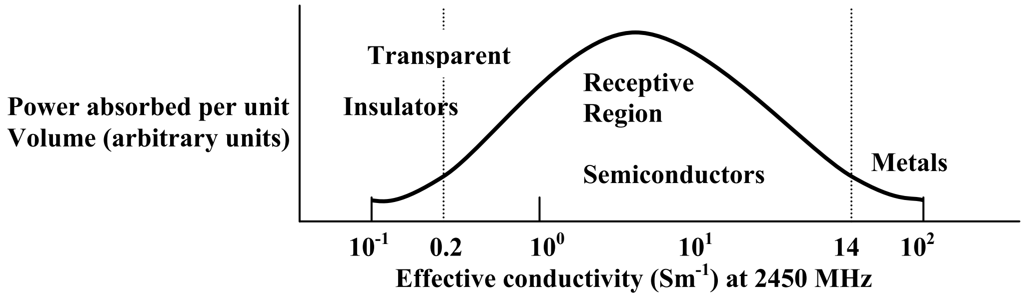

Figure 1. Microwave Energy absorption is a function of electrical conductivity

At 2.45 GHz it is observed that the skin depth in the bulk metals is very low (of the order of a few microns), and hence very little penetration of microwaves takes place. However, in the case of fine metal powders rapid heating can occur. A theoretical model developed predicted that if the metal powder particle size is less than 100μm, it will absorb microwaves at 2.45 GHz. It was further observed that the degree of microwave 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 [18].

The earliest work of microwave interaction with metallic powders is reported by Nishitani [19], who reported that by adding a few percent of electrically conducting powders such as aluminum, the heating rates of the refractory ceramics is considerably enhanced. Walkiewicz et al. [20] likewise simply exposed a range of materials, including six metals to a 2.4 GHz field, and reported modest heating (but not sintering) in the range from 120°C (Mg) to 768°C (Fe). Whittaker and Mingos [21] used the high exothermic reaction rates of metal powders with sulfur for the microwave-induced synthesis of metal sulphides. Sheinberg et al. [22] heated Cu powders coated with CuO to 650°C but did not report any sintering of them. Narsimhan et al. [23] succeeded in heating Fe alloys in a microwave oven only up to 370°C in 30 minutes. 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 at microwave **sintering** of powder metals took place [15], and since then many other researchers have reported successful sintering of many metallic materials [24-26].

Experimental

Microwave processing of various metallic materials was carried out in multi-mode cavities operating at 2.45 GHz. Figure 2 and 3 show two systems which have been used in this study: 6 kW batch process and 2 kW tubular microwave furnace.

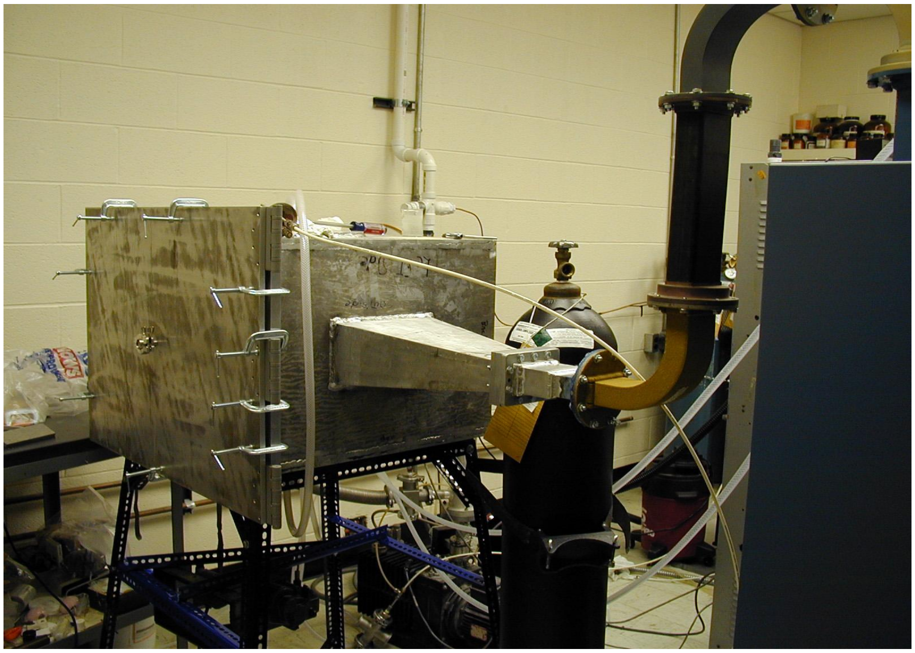

Figure 2. Multimode, 6 kW and 2.45 GHz microwave sintering system for large metal samples

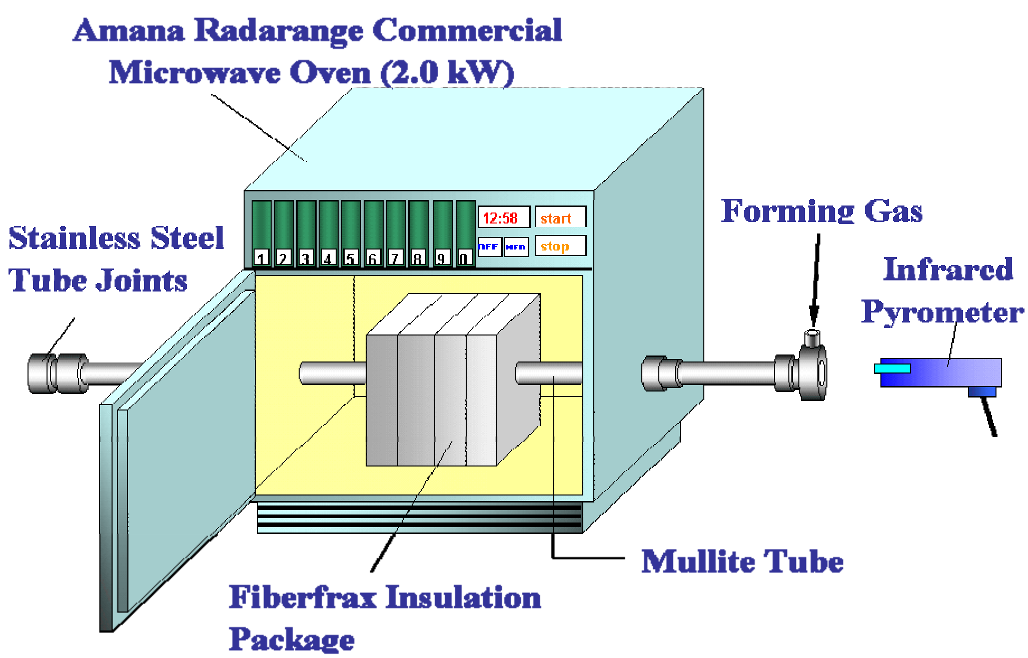

Figure 3. Schematic of a multimode, 2 kW and 2.45 GHz microwave sintering system for small metal samples

Results and Discussion

We have not so far found a single metal, as long as it is in powder form, that does not heat up in a microwave field at room temperature. It has been observed that microwave sintering of metal powders produces a superior product. The steel commercial parts of FC208 and FN208 have been sintered to near net shape. Figure 4 shows some commercial steel products sintered in a microwave. Many commercial powder-metal components of various alloy compositions including iron and steel, Cu, Al, Ni, Mo, Co, Ti, W, WC, Sn, etc. and their alloys have also been sintered in microwaves, producing nearly fully dense bodies.

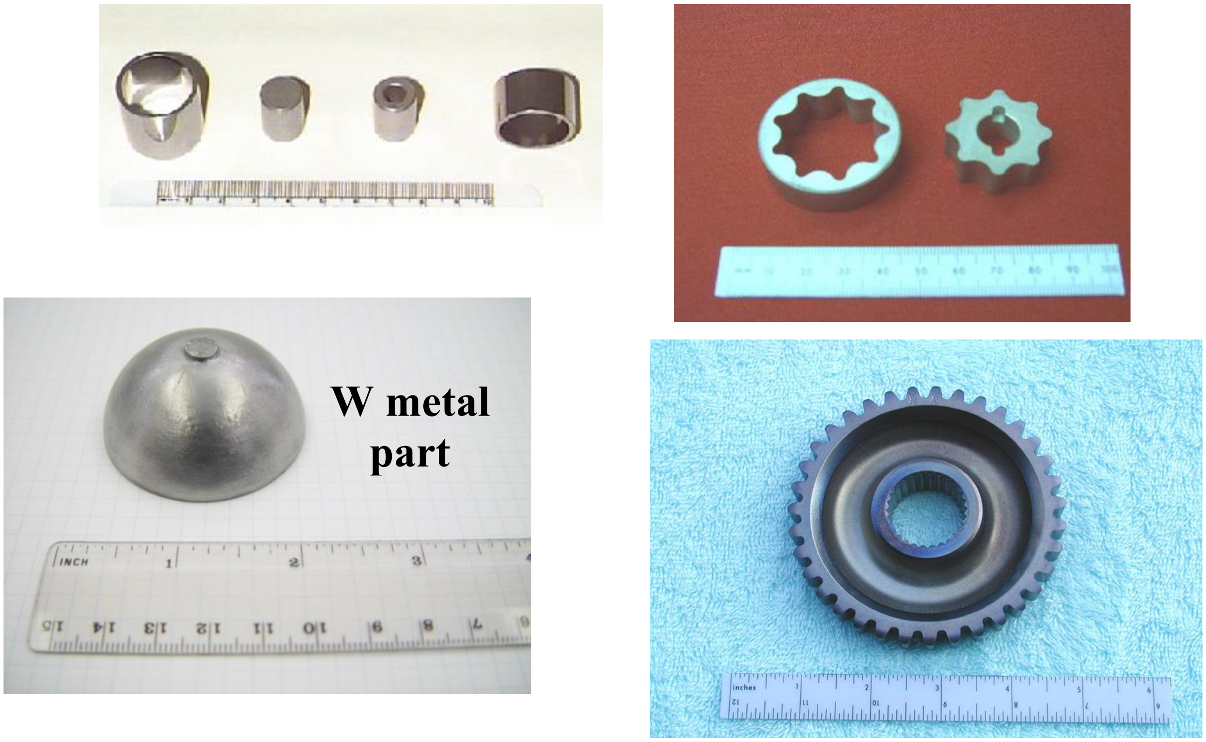

Figure 4. Various metal/steel parts sintered by microwave process.

The microwave sintering of PM green bodies comprising various metals, steels and metal alloys produced highly sintered bodies in a very short period of time [26]. Typically the total cycle time was about 90 minutes, sintering temperature ranges between 1100°C to 1300°C and soaking time of 5 to 60 minutes. The mechanical properties such as the modulus of rupture (MOR) and hardness of microwave-processed samples were much higher than the conventional samples. As an example, copper steel (MPIF FC-0208 composition) was successfully sintered by microwave technique to produce good sintered density, hardness, flexural strength, and near net dimensions, thus yielding equivalent or even sometimes superior mechanical properties than conventional sintering. In this material the Rockwell B hardness (HRB) as high as 82$\pm$2 was obtained for microwave processed samples sintered at 1260^{o}C for 5 min. soaking in flowing forming gas atmosphere. The maximum flexural strength of 1077$\pm$10 MPa was obtained for microwave sintered samples at 1140^{o}C for 20 minutes.

An examination of the microstructures and porosity distributions of the conventional and microwave sintered samples, reveals that microwave sample has more uniform microstructure than the conventional sample in which the core had more pores than the surface. Though some microwave processed samples exhibited a slightly denser core than the surface. This is typical of most ceramics sintered in a microwave, indicating that heat transport is from inside out, and the interior of the material may be hotter than the surface. However, using a modified microwave sintering system, sintered samples with both uniformly dense core and edges could be obtained. An important distinction in the microstructures of conventional and microwave-sintered samples was noticed: the pores in the microwave-sintered samples had more rounded edges than the conventional sample. It is commonly known that a sintered product exhibits a higher ductility and strength when the pore shape is more spherical. This was proven by conducting a standard test for measuring ductility and toughness of hollow cylindrical samples of FC208. Figure 5 shows the result of this test. It was found that conventional parts failed at a load of 320 lbs and microwave part at 430 lbs, indicating an increase of about 30% in the strength. But a more important feature was the manner in which the parts failed after applying the maximum load. The conventional part broke in to 4 curved pieces, which is very typical of the standard PM parts. On the other hand, the microwave processed part broke into two flat pieces, indicating a higher ductility. An explanation of this distinguishing feature was sought by analyzing the microstructures of the two samples. It was revealed that the pores in the microwave-sintered samples did have rounded edges (Fig. 5C) in contrast to the sharp edged porosity (Fig.5D) in the conventionally sintered samples [27]. A comparative study of the sintering behavior of Cu-12Sn bronze system [25] reported that bronze was microwave sintered in significantly less time resulting into higher density and more uniform microstructure. Also hardness of the microwave sintered samples compacted at 300 MPa was 50 % higher than the conventionally sintered samples.

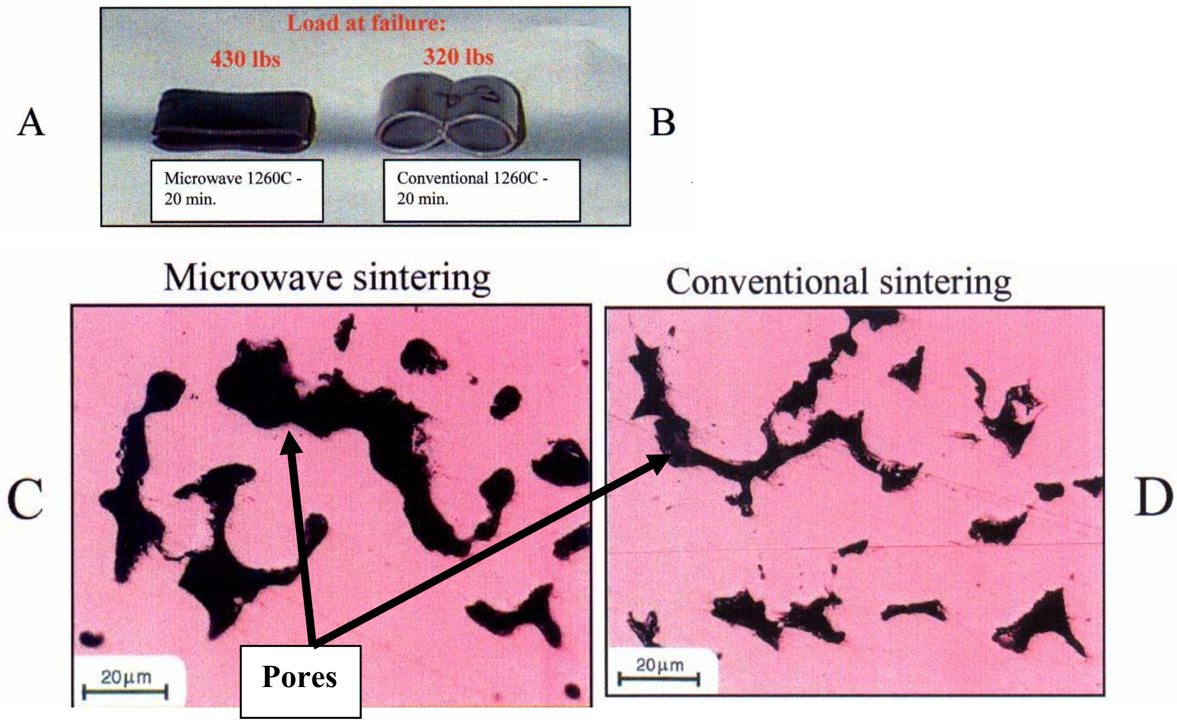

Figure 5: Ductility test results performed on FC-208 steel sample, (A) Microwave sintered (B) Conventionally sintered, Pore shape in microwave sample (C) and in conventionally sintered sample (D).

Some refractory metals such as W and Mo were also microwave sintered at much lower temperatures and sintering times than normally used in a conventional process. Figure 6 exhibits microstructures of microwave sintered nano-W powders which have been doped either with Y_2O_3 or with HfO_2 as grain growth inhibitors. It is to be noted that microwave sintering at 1400^{o}C for 20 minutes produced submicron size microstructures in the samples and densities in the order of 95+%.

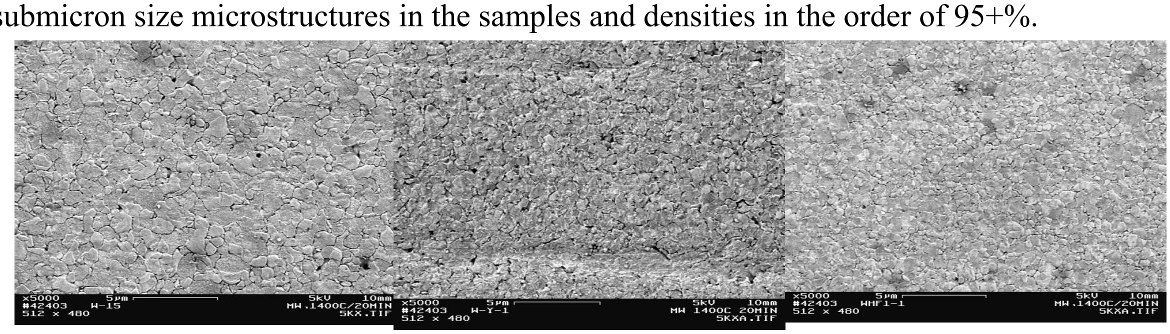

Figure 6. Microwave sintered nano-W powders at 1400^{o}C/20 minutes (a) un-doped (1-3 µm), (b) doped with Y_2O_3 (0.5-2 µm) and (c) doped with HfO_2 (0.5-0.75 µm)

Figure 7 shows a typical microstructure of Mo sample sintered in microwave at 1600°C for 1 minute. The average grain size in this sample is also submicron and density about 98%.

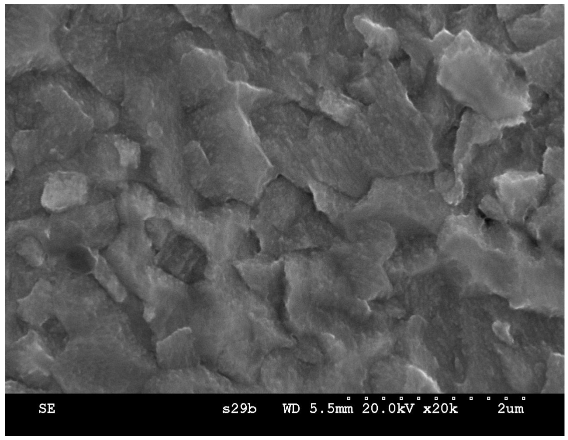

Figure 7. Typical microstructure of microwave sintered nano-Mo powder (1600°C for 1 minute, H_2) showing average grain size of ~0.55 μm

The application of microwaves to metallic materials has been extended from sintering to melting, brazing and joining of bulk metals [28-30]. Figure 8 shows some bulk metals that have been melted in a microwave field using a special insulation package with susceptors. Now at Oak Ridge National Lab's Y-12 complex, this technology has been scaled up for commercial use for metal melting and casting into useful products including recycling of alumina cans.

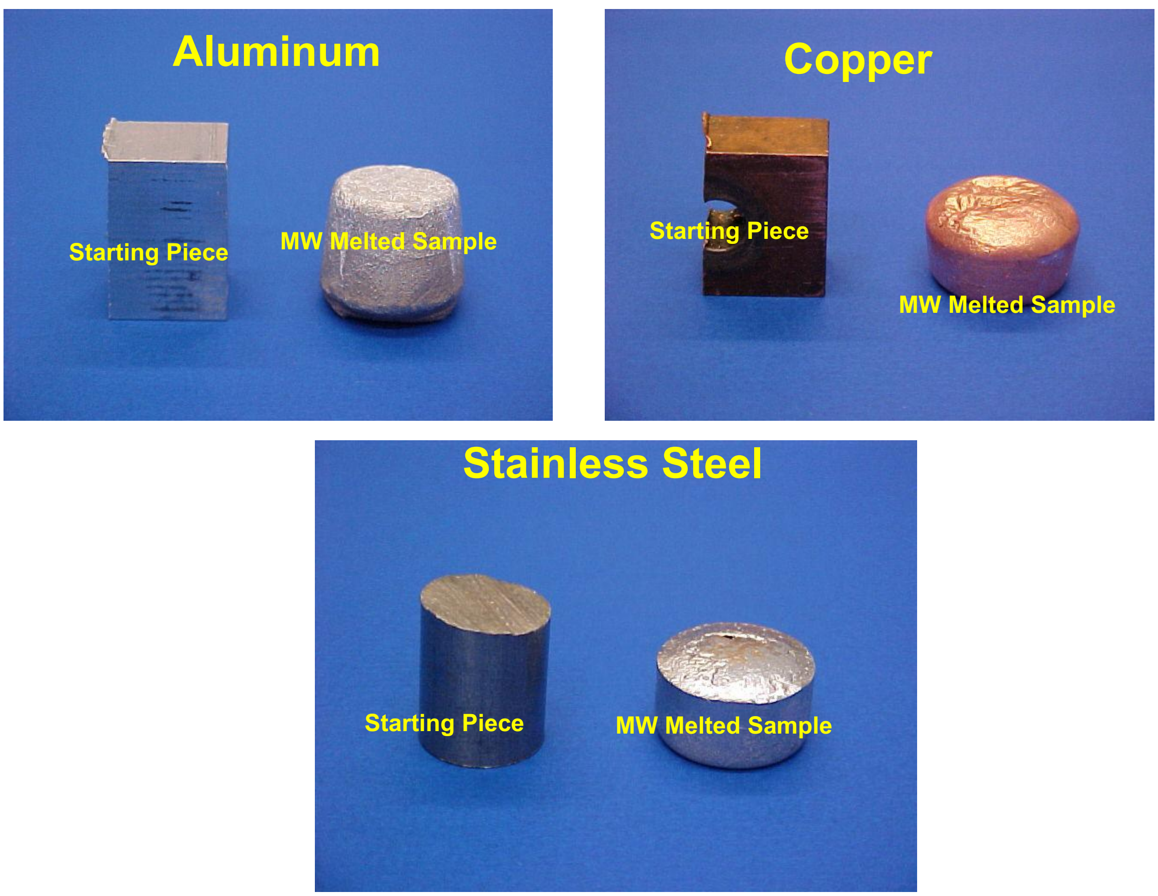

Figure 8. Some typical bulk metals before and after melting in microwave.

As mentioned earlier, microwave selectively heats powder metals and reflects bulk metals at room temperature. This feature has been exploited to braze and join bulk metals using powdered metal/alloy braze materials. We have successfully joined steels, W bulk metals, and also brazed super alloy components. An example is shown in Figure 9 in which regular steel and cast iron parts have been joined in the microwave field in 2-3 minutes using a braze powder. The joint is almost perfect, as indicated by the microstructural examination of the sample. This work can be extended to join metal to a ceramic, and also to develop ceramic coatings on steels or metal coatings on ceramics.

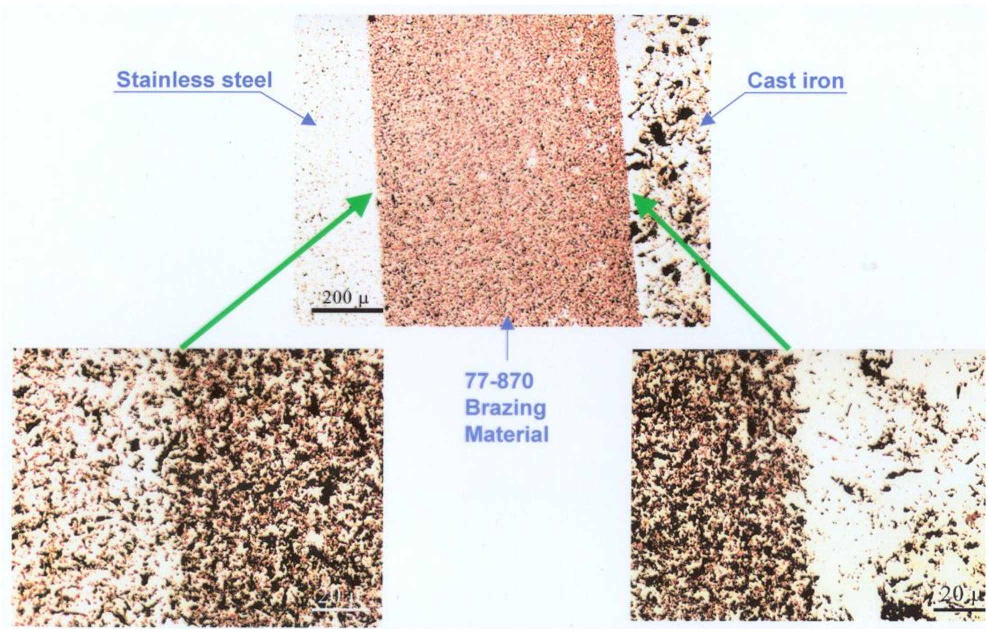

Figure 9. Microwave joining of stainless steel and cast iron using braze powder

Conclusions

The implications of the application of microwave energy and its advantages to process metallic materials as shown above are obvious in the field of metallurgy. Metal powders are used in industry for diversity of products and applications. The challenging demands for new and improved processes and materials of high integrity for advanced engineering applications require innovation and newer technologies. Finer microstructures and near theoretical densities in special PM components are still elusive and challenging. Increasing cost is also a concern of the industry. Researchers are looking for newer technologies and processes to meet these demands. The developments as reported herein using microwave processing may offer a new method to meet these demands of producing better microstructures and properties in the powder metal products.

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