Technologies **2015**, *3*, 1-18; doi:10.3390/technologies3010001

OPEN ACCESS

technologies

ISSN 2227-7080

www.mdpi.com/journal/technologies

Review

Using Microwave Energy to Synthesize Light Weight/Energy Saving Magnesium Based Materials: A Review

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Academic Editor: Luciano Feo

Received: 30 September 2014 / Accepted: 7 January 2015 / Published: 20 January 2015

Abstract: Microwave energy can be used for the processing of a wide variety of materials. It is used most commonly for the heating of food and has been increasingly applied for processing of polymers; ceramics; metals; minerals and composites. The use of microwave energy allows rapid and volumetric heating where heat is generated from within the material instead of via radiative heat transfer from external heating elements. This paper aims to provide a review on the use of energy efficient and environment friendly microwave energy route to synthesize magnesium based materials reinforced with various types of metallic and ceramic reinforcements. Magnesium composites are extremely attractive for weight critical applications in automotive; aerospace; electronics and transportation sectors. The magnesium composites were prepared using blend—compact—microwave sintering—extrusion methodology. Microwave sintering allowed a significant reduction of 80% in both processing time and energy consumption over conventional sintering without any detrimental effect on the properties of the synthesized magnesium composites. Physical; microstructure and mechanical properties of microwave sintered magnesium composites will also be discussed and compared with magnesium composites processed by conventional liquid and solid processing techniques.

Keywords: microwave sintering; magnesium composites; microstructure; mechanical properties

1. Introduction

1.1. Brief History and Applications of Microwave Heating

Microwaves are part of the electromagnetic spectrum with frequencies ranging from 300 MHz to 300 GHz and corresponding wavelengths between 1 m and 1 mm respectively. Microwave technology developed rapidly during Second World War due to the demand for better radar technology for detection of enemy aircraft and submarines. During this period, the applications of microwaves were mainly restricted to radar and communications with limited studies on the use of microwaves for heating of tissues in therapeutic applications [1,2]. Interest in microwave heating applications started towards the end of World War II when major manufacturers of microwave tubes, such as Westinghouse, General Electric and Raytheon, started to look into alternative uses for microwave tubes, and filed many patents to make use of microwave heating for industrial applications, such as drying of tires, textiles, and wood; commercial processing; and treatment of food.

In 1945, Dr. Percy Spencer from Raytheon filed a patent for using microwaves to process food leading to the introduction of commercial microwave ovens in 1947 but found limited usage in industries due to its large size and high cost. In 1960s, a smaller and cheaper domestic microwave oven was introduced and the sale of microwave ovens grew rapidly in 1970s and has since become a common kitchen appliance in many households.

Microwave heating is used most commonly for the heating of food due to the good microwave susceptibility of water molecules in the food and has been increasingly applied for processing of polymers, ceramics, metals, minerals, chemicals, composites and biological subjects. For food processing, microwave heating has been applied to drying of potato chips and pasta, meat tempering, cooking of bacon, blanching of fruits and vegetables, *etc.* [1]. In ceramics, microwave heating has been used for drying, sintering, synthesis, joining and heat treatment. Microwave heating is used mainly for curing and polymerization of polymers, and in analytical chemistry and biochemistry due to the ability for rapid synthesis of reaction products and novel materials. Biomedical applications of microwave heating are found in diathermy, hyperthermia, ablation, thawing and sterilization. The US military has also introduced microwaves as non-lethal weaponry in microwave bombs, which create a short and intense electromagnetic pulse to disable electronic devices, and Active Denial System (ADS), which heats the skin of human subjects for security and crowd control. In addition, there are also promising applications in using microwave heating for mineral processing, waste remediation and recycling.

1.2. Fundamentals of Microwave Heating

One of the earliest description of the characteristics of microwave heating was in a Scientific American article in 1943 where it was mentioned that heat was generated from within the object and involves no transfer of heat to it [1,3]. This is fundamentally different from conventional heating where heat is usually transferred to an object via conduction, convection or radiation. In conventional heating

using an oven or furnace, the heat source (usually resistance heating elements) has to heat up the entire volume of air and walls of the container first before thermal energy is transferred from the surface of the object to the interior through conduction. Due to the penetrative nature of microwaves, heat is generated from within the object through the absorption of microwave energy directly by the object and do not require substantial heating of the environment. Therefore a temperature gradient exists in both conventional and microwave heating as a result of the way heat is transferred/generated in the object as shown in Figure 1. To circumvent the problem of temperature gradient in the object, hybrid-heating comprising of both microwaves and conventional radiant heating has been employed to ensure uniform heating of the object.

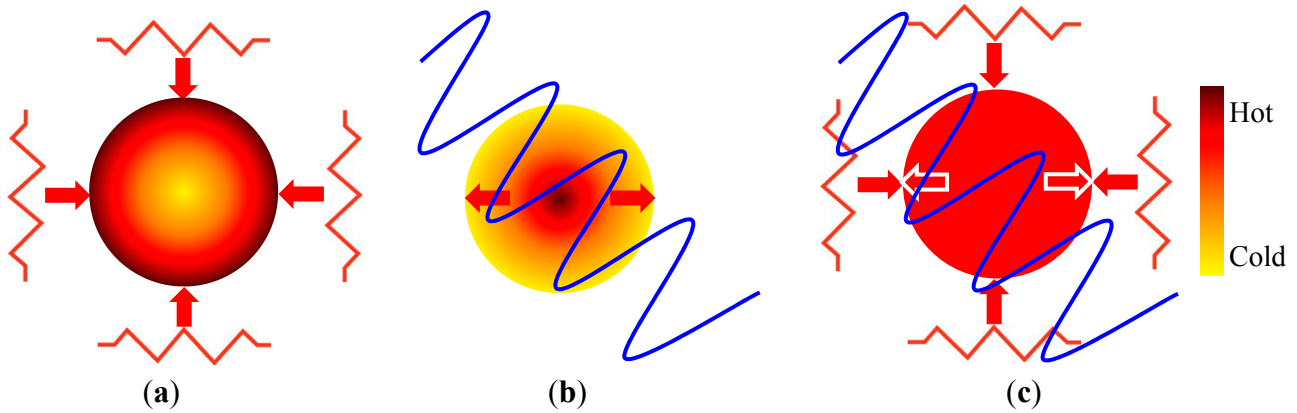

Figure 1. Heat distribution in an object during (**a**) conventional resistance heating, (**b**) microwave heating, and (**c**) hybrid heating using microwaves and radiant heating.

1.3. Microwave Sintering of Metallic Composites

Existing theories and mechanisms for microwave heating of materials focused on the interaction of dielectric materials to an applied electric field or magnetic field using properties, such as dielectric constant, loss factor, loss tangent, permeability and resistivity, to determine the power absorbed and penetration depth of microwaves for a material [1]. Dielectric materials are usually non-metals and include gases, liquids and solids [4]. For metallic conductors, the penetration depth, d of the magnetic field can be computed using Equation (1):

$$d = \sqrt{\frac{\rho}{\pi f \mu_0 \mu_r'}} \quad (1)$$

where ρ is the resistivity of metal, f is the frequency of the electromagnetic wave, μ_0 is the permeability of free space and μ_r' is the relative permeability of the material (≈1 for most metals). The resistivity for most metals are low, therefore the penetration depth for metals is usually in the micrometer range at common microwave frequencies of 915 MHz and 2.45 GHz and is more than a hundred times lower than for dielectric materials [1]. Due to their low penetration depth and good reflection of microwaves, metals are used as waveguides and cavity walls for containing microwaves. However, the behavior for bulk metals and metallic powders/particles is entirely different. While bulk metals reflect microwaves and display arching phenomenon when exposed to microwaves, metallic powders and particles are able to absorb microwaves and get heated rapidly.

In 1988, Walkiewicz *et al.* first reported the rapid heating of various metal powders when exposed to microwaves [5]. In 1999, researchers from Penn State University demonstrated the superior physical and mechanical properties of microwave sintered steel alloys over conventional heating [6]. Various metal systems such as copper, aluminum, tungsten, titanium and hardmetals have also been successfully sintered using microwaves [1,7,8].

Magnesium based materials synthesized using a hybrid microwave sintering technique was first reported by the authors in 2005 [9]. Magnesium is the lightest structural metal and possesses similar melting temperature and yield strength to aluminum. With its high strength to weight ratio, magnesium has tremendous potential for applications in the aerospace, automotive, biomedical, electronics and military sectors. However, the limited ductility due to its hexagonal close-packed structure, low elastic modulus and melting temperature limits the use of pure magnesium for load bearing applications. These limitations are being resolved through the development of new magnesium alloys and composites and innovation in processing techniques. This review aims to provide an update on the research work on magnesium based materials reinforced with different types of ceramic, metallic and amorphous reinforcements synthesized using hybrid microwave sintering.

2. Experimental Section

Elemental magnesium powder of 98.5% purity with a size range of 60–300 μm obtained from Merck KGaA, Germany was used as the matrix material. Various types of metallic and ceramic reinforcements of varying sizes were added to the matrix as shown in Table 1. $Ni_{60}Nb_{40}$ amorphous reinforcement was prepared by mechanically alloying powder mixture of elemental Ni and Nb metals at room temperature in air for 87 h using a planetary ball mill with a ball to powder ratio of 3:1 and milling speed of 200 rpm [10].

Table 1. Properties of matrix and reinforcements.

Element	Size	Supplier
Magnesium	60–300 μm	Merck KGaA, Germany
SiC	25 μm	Amet
β-SiC	45–55 nm	Nanostructured & Amorphous Materials, USA
Cu	50 nm	Argonide Corporation, USA
Ni	20 nm	Nanostructured & Amorphous Materials, USA
Al_2O_3	0.3 μm	Baikowski, USA
Al_2O_3	50 nm	Baikowski, USA
Y_2O_3	30–50 nm	Inframat Advanced Materials, USA
ZrO_2	51–65 nm	Nanostructured & Amorphous Materials, USA
$Ni_{60}Nb_{40}$	-	Prepared by mechanically alloying element Ni and Nb metals

Pure magnesium powder and reinforcements were weighed carefully and mixed in a RETSCH PM-400 mechanical alloying machine using a speed of 200 rpm for 1 h. No balls or process control agent was added during the blending step. The blended powders were uniaxially compacted using a pressure of 97 bar (~50 tons) to billets (40 mm height with 35 mm diameter) in a 100-ton press. The compacted billets were sintered using an innovative microwave assisted hybrid sintering technique for a specific duration to the desired temperature in a 900 W, 2.45 GHz SHARP microwave oven using SiC as the microwave susceptor material. Loosely packed SiC powder (contained within a microwave transparent

ceramic crucible) absorbs microwave energy readily at room temperature and is heated up rapidly, providing the radiant heat to heat the billet externally while the compacted billet absorbs microwaves and is heated from within. This hybrid heating method results in a more uniform temperature gradient within the billet and circumvents the disadvantage of heating using either conventional heating or microwaves only. The exterior of the ceramic crucible was thermally insulated using alumino-silicate fiber insulation board to reduce heat loss from the surface. All magnesium compacts were then sintered under ambient atmospheric condition without the presence of inert gas atmosphere. The schematic diagram of the experimental setup can be found in previous studies [9,11]. Temperature calibration of the sintering setup was performed beforehand using a sheathed K-type thermocouple in order to determine the appropriate sintering duration for the billets. The heating rate for the setup can be varied by changing the amount of SiC susceptor used. A higher heating rate can be realized by using a larger amount of SiC susceptor [12,13]. Pure magnesium was compacted using the same pressure as the composite formulations and sintered in microwave oven under identical conditions. All magnesium samples were heated to a temperature near the melting point of pure magnesium (approximately 640 °C) except for Mg-amorphous alloy composites which were sintered at a temperature of 550 °C.

Conventional sintering was carried out using a Carbolite tube furnace in an argon-controlled environment for comparison purpose. A heating rate of 10 °C per minute was used to heat pure magnesium metal compact to a sintering temperature of 0.85 of the melting temperature for pure magnesium, which corresponds to 785 K (512 °C), and held at this temperature for 120 min before cooling. The cooling rate was a constant 25 °C per minute. The sintered billets of pure magnesium and its composite formulations were subsequently hot extruded at a temperature of 350 °C using a 150 ton hydraulic press to produce an extruded rod ranging between 7 to 10 mm in diameter depending on the extrusion dies used. The extruded rods are sectioned and machined for various physical, microstructural and mechanical testing.

The densities of the extruded samples were determined using Archimedes principle. Polished samples taken from various sections of the extruded rods were weighed in air and when immersed in distilled water using an electronic balance with an accuracy of ±0.0001 g.

Microstructural characterization studies were conducted on polished specimens of pure metals and its composite formulations to investigate the presence of porosity, reinforcement distribution, matrix-reinforcement interfacial integrity grain size and grain morphology.

X-ray diffraction analysis was carried out on the polished samples using an automated Shimadzu XRD-6000 diffractometer. The samples were exposed to CuK_{α} radiation ($\lambda = 1.54056$ Å) at a scanning speed of 2 deg/min. The Bragg angle and the values of the interplanar spacing, d obtained were subsequently matched with the standard values for Mg and other related phases.

Microhardness measurements were performed on the polished samples using a Matsuzawa MXT 50 automatic digital microhardness tester. The microhardness test was performed using a Vickers indenter under a test load of 25 gf and a dwell time of 15 s in accordance with the ASTM standard E384-99. Tensile properties of the extruded samples were determined in accordance with ASTM standard E8M-01. The tensile tests were conducted on round tension test specimens of 5 mm in diameter and 25 mm gauge length using an automated servohydraulic testing machine (MTS 810) with a crosshead speed set at 0.254 mm/min.

Fracture surfaces of the pure magnesium and composite specimens were investigated to provide an insight into the fracture mechanisms operating during the tensile loading of samples.

3. Results and Discussion

3.1. Hybrid Microwave Sintering Technique

Hybrid microwave sintering significantly reduces the sintering time for magnesium compacts with a reduction of 80% to 90% in processing time due to a faster heating rate, higher sintering temperature and the elimination of holding time as shown in Figure 2. A rapid heating rate and high sintering temperature will enhance densification and better activate the bulk transport processes [14]. Rapid heating minimizes grain growth and can produce materials with high sintered densities. Minimizing the grain growth also enhances the mechanical properties of materials. A higher sintering temperature will (i) increase the microwave penetration depth due to an increase in the resistivity and aid in the absorption of microwave energy by the metal compact [12,15] and (ii) increase the number of active atoms and available sites for diffusion. From Equation (1), the penetration depth is a function of resistivity and the resistivity of metals generally increases linearly with increasing temperature due to the reduced mobility of electrons at elevated temperature [16]. Researchers have also observed enhanced microwave absorption by near-molten metals in a highly nonlinear fashion that is contrary to the general linear relationship between resistivity and temperature [15]. Therefore the high sintering temperature near the melting point of the metal will enhance the absorption of microwaves by the metal. The number of vacant atomic sites and the number of atoms with sufficient energy to move into the vacant sites are related with an Arrhenius equation as shown in Equation (2):

$$\frac{N}{N_0} = \exp\left(-\frac{Q}{RT}\right) \tag{2}$$

where N/N_0 is the ratio of available sites or activated atoms to total atoms, Q is the activation energy, R is the gas constant and T is the absolute temperature. Since sintering is a thermally activated process, higher sintering temperatures can significantly shorten the sintering time. The combination of rapid heating and higher sintering temperature allows the elimination of the holding time leading to the significant reduction in sintering time for magnesium, which can also be extended to other metallic systems.

Also, the volumetric heating of hybrid microwave heating has been demonstrated by the researchers for the heating of aluminum compacts [12]. The *in-situ* temperature measurements were conducted by drilling 5 mm diameter holes to depths of 3 and 20 mm for temperature measurements at the surface and in the center of the compacts respectively using a shielded thermocouple. It can be observed that the difference in temperature between the center and the surface is minimal as shown in Figure 3, indicating volumetric heating of the compact. Also at higher temperature above 350 °C, the center of the compact indicates a slightly higher temperature than the temperature taken near the surface of the compact indicating the absorption of microwave energy by the compact. It may be noted that the *in-situ* temperature measurements were carried out on separate green compacts due to the difference in absorption of microwaves by the metal after sintering. A higher heating rate can be observed in metal compact that is sintered for the first time. On reheating a previously sintered aluminum compact, the

heating rate is significantly reduced by approximately half that of the unsintered compact as shown in Figure 4. Previously sintered metal compacts will react as a bulk material with limited penetration depth therefore reflecting most of the microwave energy when exposed to microwaves again. Heating occurs mainly by the external susceptors and hence requires a longer time to be heated to the same temperature again [12].

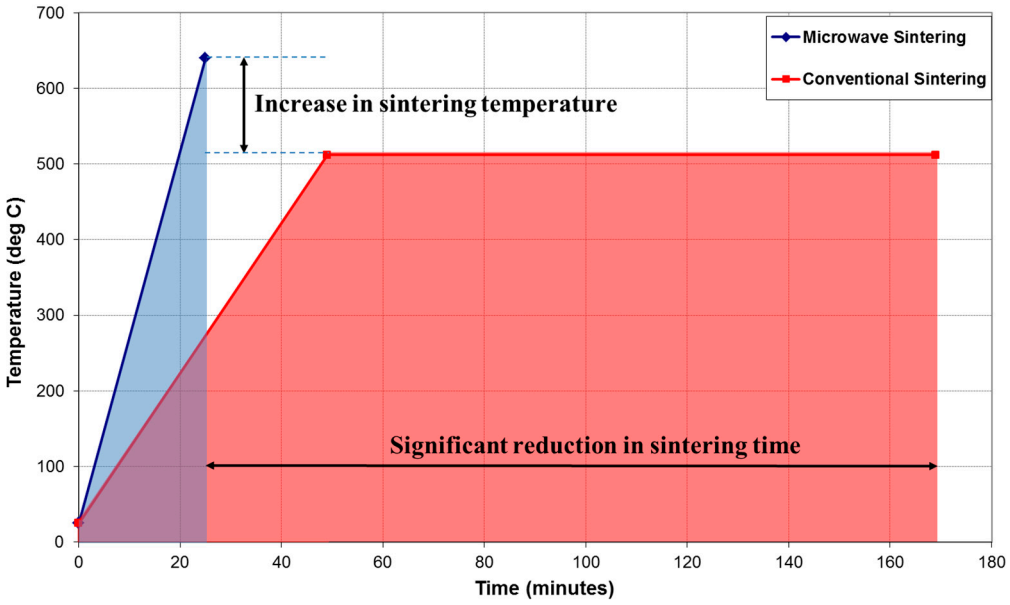

Figure 2. Comparison of sintering profile between conventional and microwave sintering for magnesium.

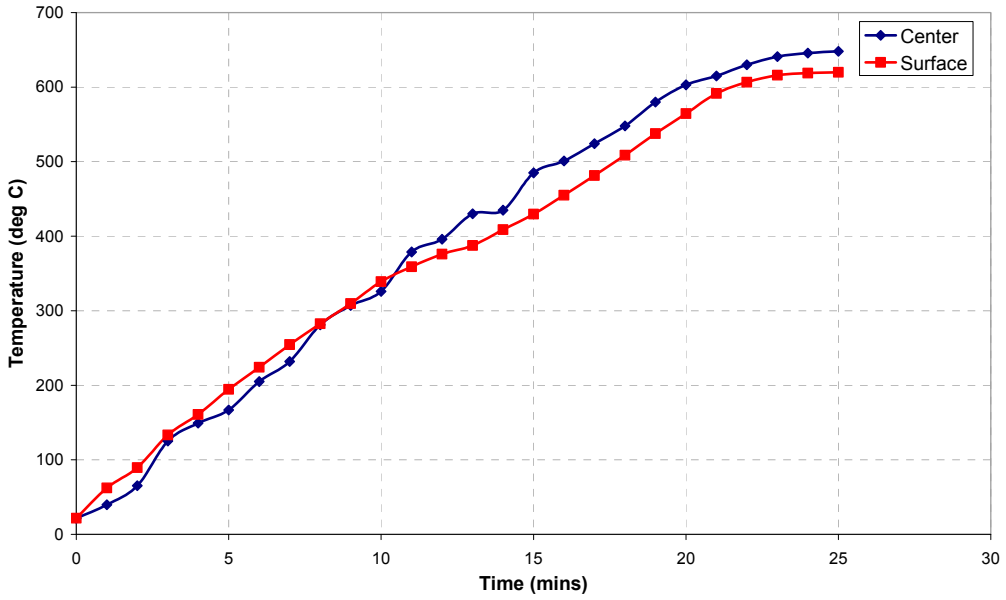

Figure 3. *In-situ* temperature measurements in the center and on the surface of an aluminum compact [12].

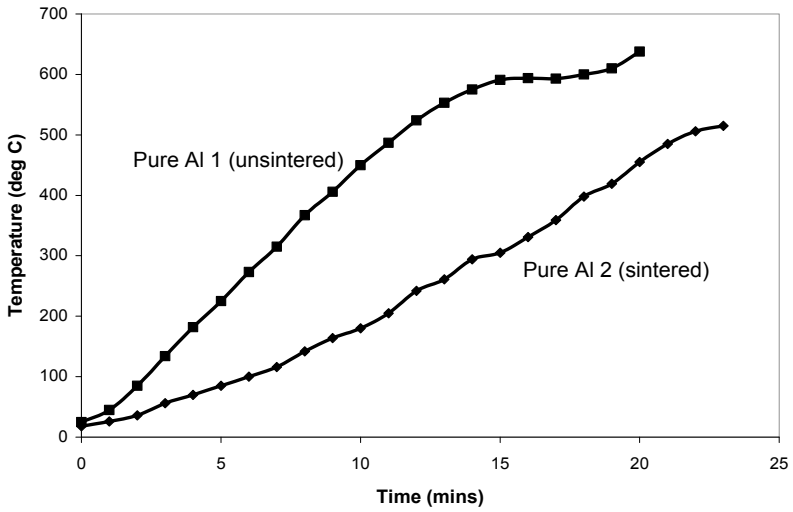

Figure 4. Differences in heating behavior between sintered and unsintered compacts [12].

For conventional heating, a slower heating rate and long isothermal hold at sintering temperature are required to minimize temperature differences in the material due to the effect of distances from furnace walls and poor heat transfer through the porous structure which promotes higher rate of grain growth [14]. In addition, the heating rate is often limited by the slow rate of increase in temperature of the resistive heating elements and the need to heat up the entire surroundings inside the furnace before heat is transferred to the material.

For hybrid microwave sintering, the heating rate can be varied by controlling the amount of SiC susceptor added and the size of SiC particles used. A higher heating rate is achieved by increasing the amount of SiC used and by decreasing the size of the SiC particles as shown in Figure 5.

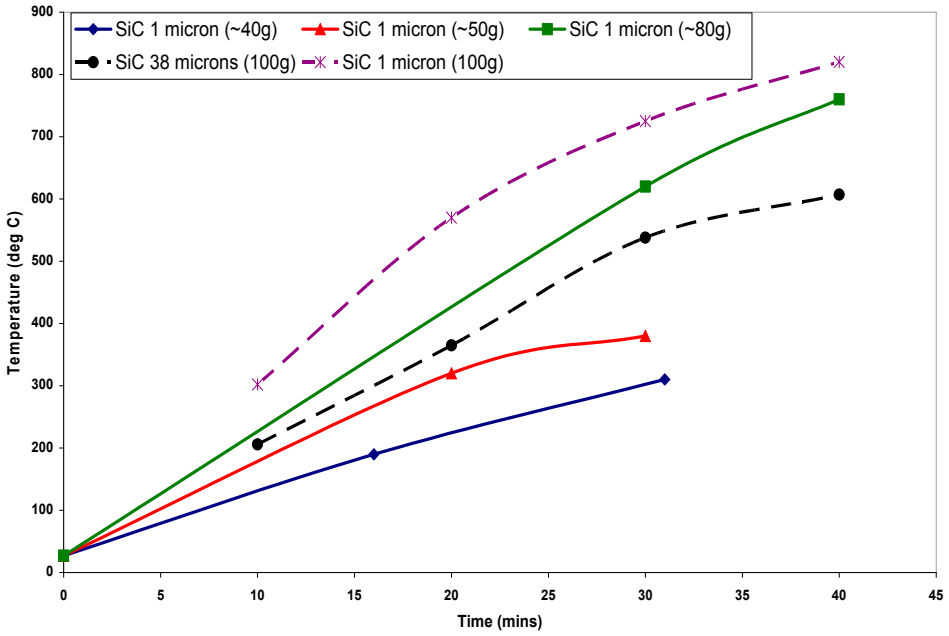

Figure 5. Effect of amount and particle size of SiC susceptor on the heating rate [12].

Based on the various magnesium based formulations investigated by the authors, the hybrid microwave sintering technique provides a simple and economical way to sinter highly reactive

magnesium metal at high temperature without the need for an inert protective atmosphere unlike in conventional sintering where an inert atmosphere is required. Most importantly, the end properties of hybrid microwave sintered magnesium (which will be discussed in later sections) are not affected by the absence of an inert atmosphere during sintering and XRD results of the specimen did not shown the presence of oxide peaks as shown in Figure 6. This is also supported by a study conducted by Takayama *et al.* [17] comparing copper compacts sintered in air using microwaves and conventional heating. EDX analysis from the study revealed a significantly lower level of oxygen content in microwave sintered copper compact when compared to conventionally sintered compact in air. It was reported that the level of oxygen content in the microwave-sintered sample was comparable to that of conventionally sintered compacts in an inert atmosphere.

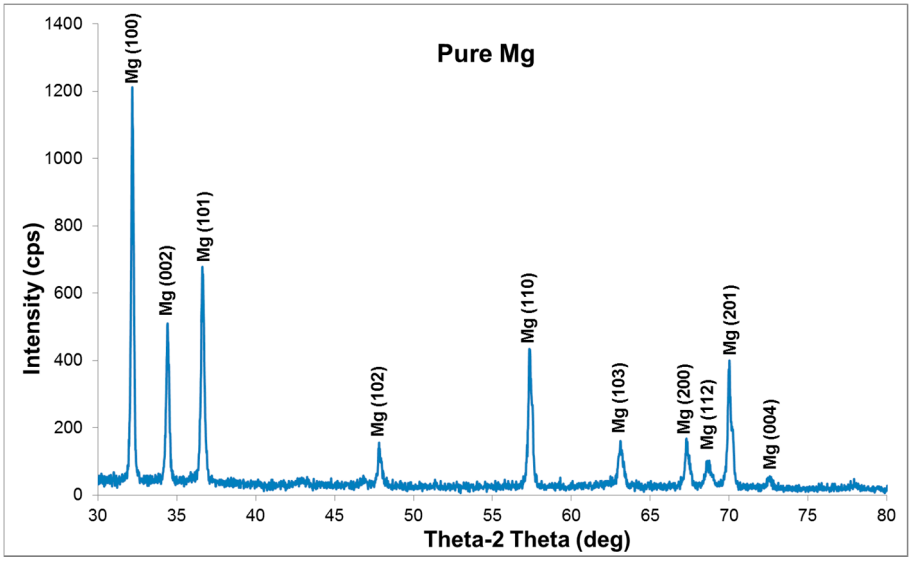

Figure 6. X-ray diffractogram of pure magnesium after microwave sintering.

Another advantage of microwave sintering is the significant energy efficiency over conventional sintering. A simple calculation of the energy consumption for the sintering of magnesium or aluminum using a conventional tube furnace and a microwave oven during the heating phase is shown in Table 2. It can be observed that the use of microwave sintering can lead to an impressive energy savings of up to 86%, which is economically viable for industries and environmentally friendly in the reduction of CO_2 emissions.

Table 2. Analysis of energy consumption between conventional and microwave sintering during heating.

Material	Heating Unit	Power kW	Time h (mins)	Energy Consumption kWh	Energy Savings %
Magnesium	Tube furnace (Carbolite CTF15/75)	6	0.82 (49)	4.92	86
	Microwave (Sharp magnetron)	1.6 *	0.42 (25)	0.67	

* Based on AC power required for operation of the magnetron.

3.2. Physical Properties

Near theoretical density can be realized for hybrid microwave sintered magnesium after extrusion. The density and porosity of microwave-sintered magnesium is in close comparison with conventionally sintered magnesium in spite of the significant reduction in sintering time as shown in Table 3 [12,18–26]. The results of porosity measurements amongst the pure magnesium samples indicate that higher heating rate leads to an improvement in the densification of magnesium.

Table 3. Results of density and porosity measurements.

Materials	Theoretical ρ (g/cm^3)	Experimental ρ (g/cm^3)	Porosity (%)	Grain Size (μm)	Aspect Ratio
Mg Conv	1.740	1.737 ± 0.002	0.15	33 ± 8	1.5 ± 0.3
Mg MW (32 min)		1.734 ± 0.002	0.33	36 ± 9	1.4 ± 0.3
Mg MW (25 min)		1.737 ± 0.001	0.17	27 ± 7	1.6 ± 0.4
Mg MW (13 min)		1.738 ± 0.007	0.13	20 ± 3	1.4 ± 0.1
Composites containing microwave susceptors reinforcement					
Mg 10 SiC (38 μm) *	1.888	1.865 ± 0.004	1.22	-	-
Mg 0.35 β-SiC (45–55 nm) *	1.745	1.735 ± 0.003	0.58	-	-
Mg 0.5 βSiC (45–55 nm) *	1.747	1.739 ± 0.002	0.48	-	-
Mg 1.0 β-SiC (45–55 nm) *	1.755	1.753 ± 0.007	0.11	-	-
Composites containing microwave transparent reinforcements					
Mg 0.3 Al_2O_3 (50 nm) *	1.747	1.741 ± 0.004	0.32	-	-
Mg 0.6 Al_2O_3 (50 nm) *	1.753	1.742 ± 0.008	0.67	24 ± 4	1.5 ± 0.4
Mg 1.0 Al_2O_3 (50 nm) *	1.762	1.748 ± 0.010	0.83	15 ± 3	1.5 ± 0.3
Mg 0.17 Y_2O_3 (40 nm) *	1.746	1.73 ± 0.01	0.87	19 ± 3	1.4 ± 0.2
Mg 0.7 Y_2O_3 (40 nm) *	1.763	1.757 ± 0.006	0.35	18 ± 3	1.4 ± 0.2
Mg 0.3 ZrO_2 (51–65 nm) *	-	-	-	24 ± 7	-
Mg 1.0 ZrO_2 (51–65 nm) *	-	-	-	25 ± 4	-
Composites containing metallic reinforcements					
Mg 0.3 Cu (50 nm) *	1.762	1.758 ± 0.002	0.19	17 ± 5	1.5 ± 0.3
Mg 0.6 Cu (50 nm) *	1.783	1.776 ± 0.006	0.41	15 ± 4	1.5 ± 0.3
Mg 1.0 Cu (50 nm) *	1.812	1.809 ± 0.007	0.13	15 ± 4	1.5 ± 0.3
Composites containing hybrid reinforcements					
Mg 0.3 ZrO_2 0.7 Cu	-	-	-	9 ± 2	-
Mg 0.7 Y_2O_3 0.3 Cu	1.784	1.775 ± 0.001	0.45	9 ± 5	1.6 ± 0.4
Mg 0.7 Y_2O_3 0.6 Cu	1.806	1.792 ± 0.004	0.77	9 ± 4	1.5 ± 0.3
Mg 0.7 Y_2O_3 0.3 Ni	1.785	1.778 ± 0.002	0.34	9 ± 3	1.4 ± 0.3
Mg 0.7 Y_2O_3 0.6 Ni	1.806	1.802 ± 0.002	0.21	6 ± 2	1.4 ± 0.3
Mg 0.7 Y_2O_3 1.0 Ni	1.835	1.829 ± 0.002	0.30	5 ± 2	1.5 ± 0.3

* For composites, numbers in parenthesis represent size of reinforcement particles.

Magnesium composite formulations can also be sintered to near-theoretical density. Comparing magnesium composite reinforced with micron-size SiC and nano-size β-SiC particles, it can be observed that Mg 10 SiC composite exhibit higher porosity than Mg/β-SiC composite formulations. Due to the large size difference between nano-size β-SiC and Mg particles, the nano-size β-SiC particles are able to coat around the larger magnesium particles during the mixing process, resulting in the network

distribution of nano-size reinforcements which can be observed in microstructure studies of the Mg nanocomposite (refer to Figure 4). The better distribution of nano-size reinforcements promoted more even heating (through the absorption of microwave energy) throughout the compact during sintering which may lead to the lower porosity. Also, the smaller powder sizes have more surface energy that provides a larger driving force for sintering.

The amount of porosity amongst the magnesium nanocomposites is dependent on the type of reinforcement particles and its ability to couple with microwaves. In this review, three different types of reinforcements were highlighted for discussion; (1) silicon carbide (microwave susceptor); (2) alumina, yttria and zirconia (microwave transparent); and (3) copper and nickel (metallic conductor). Silicon carbide absorbs microwaves readily at room temperature and can be heated rapidly to high temperatures. Therefore, MgSiC composite formulations were able to achieve the lowest porosity and a reduction in the porosity with increasing additions of reinforcement. Alumina is microwave transparent at room temperature and cannot be easily heated using 2.45 GHz microwaves, hence it was observed that the amount of porosity increases with increasing volume fraction of alumina reinforcement added. MgY_2O_3 composites also displayed similar porosity levels with $MgAl_2O_3$ composites. Bulk copper metal being a metallic conductor will reflect most of the incident microwaves due to limited penetration depth and cannot be heated easily. However, copper powder has been shown to absorb microwaves readily and can be heated to high temperatures in both the electric and magnetic field components of microwaves [27]. For MgCu composites, no correlation was observed between volume fraction of reinforcement and porosity level. This may be attributed to the agglomeration of nano-size Cu particles with increasing volume fraction and the formation of molten Mg_2Cu phase at temperature above 485°C due to reaction between copper and magnesium. Hybrid composites also displayed low porosity values with the maximum porosity of 0.77% exhibited by Mg 0.7 Y_2O_3 0.6 Cu. No significant trend in porosity values was observed with increasing volume fraction of metallic reinforcement added.

3.3. Microstructure

Microstructural characterization studies revealed a finer microstructure in hybrid microwave sintered magnesium when compared to its conventionally sintered counterpart with a reduction in average grain size except for the sample that was microwave sintered for 32 min as shown in Table 2. The reduction in grain size may be attributed to the higher sintering temperature and reduction in sintering time. For microwave-sintered samples, the grain size decreases with an increase in heating rate from an average of 36 to 20 μm.

Microstructural characterization on the grain size of nanocomposite samples revealed an equiaxed structure and a reduction in grain size of the matrix with the addition of higher volume fraction of reinforcements as shown in Table 2. The reduction in grain size of the matrix can be attributed to: (i) the ability of nano-size particulates to nucleate magnesium grains during recrystallization and (ii) the restricted growth of the magnesium grains due to grain boundary pinning.

As mentioned earlier, nano-sized particulates are found decorating the particle boundaries of the magnesium matrix in a network fashion as the nano-particulates are usually found on the surface of the matrix powder after mixing and sintering process will not redistribute the particulates into the grain as

shown in Figure 7. The observation is similar with other researchers that synthesized metallic composites reinforced with nanoparticles using powder metallurgy technique [28,29].

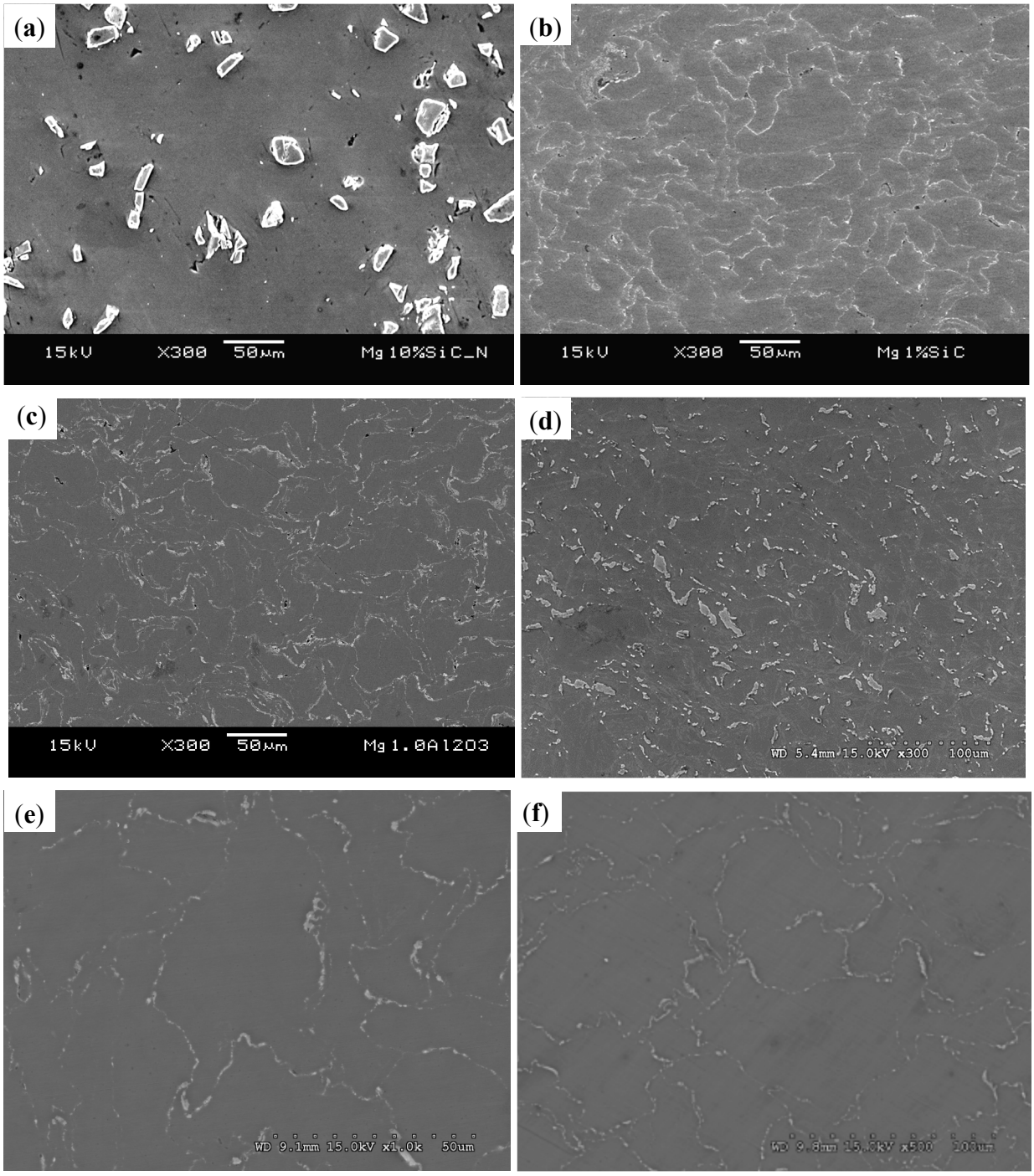

Figure 7. Representative SEM micrographs showing the distribution of reinforcements for (**a**) Mg 10 SiC; (**b**) Mg 1.0 β-SiC [20]; (**c**) Mg 1.0 Al_2O_3; (**d**) Mg 1.0 Cu; (**e**) Mg 0.7 Y_2O_3 [18]; and (**f**) Mg 1.0 ZrO_2 [23].

3.4. Mechanical Properties

An improvement in microhardness was observed in microwave sintered pure magnesium when compared to conventionally sintered counterpart. This can be attributed to the finer microstructure of the

microwave sintered samples which is consistent with the findings of other investigators where it has been shown that microwave sintered metal compacts have higher hardness than their conventionally sintered counterparts [6,30].

The addition of nano-size and amorphous reinforcements leads to an increase in the hardness of the magnesium matrix as shown in Table 4. The largest increase was observed in Mg reinforced by 10% of $Ni_{60}Nb_{40}$ amorphous reinforcement. The increase in microhardness can be attributed to the presence of harder ceramic, metallic or amorphous particulates as reinforcements, which acts as a constraint to localized matrix deformation during indentation.

Table 4. Selected mechanical properties of magnesium nanocomposites.

Materials	Processing Method	Microhardness HV	0.2% YS (MPa)	UTS (MPa)	Failure Strain (%)	Ref.
Tensile properties						
Mg Conv	Tube furnace	37 ± 1	105 ± 0	150 ± 1	5.0 ± 0.7	
Mg MW (32 min)		36 ± 2	116 ± 17	186 ± 21	11.3 ± 1.0	[18]
Mg MW (25 min)		40 ± 1	121 ± 2	176 ± 2	5.4 ± 0.7	
Mg MW (13 min)		47 ± 2	134 ± 7	193 ± 1	6.9 ± 2.5	
Mg/0.3 Al_2O_3		48 ± 3	119 ± 7	175 ± 8	7.5 ± 0.2	
Mg/0.6 Al_2O_3		54 ± 3	130 ± 5	180 ± 7	7.4 ± 0.3	[21]
Mg/1.0 Al_2O_3		60 ± 4	154 ± 5	213 ± 12	6.3 ± 0.4	
Mg/0.17 Y_2O_3		38 ± 0	144 ± 2	214 ± 4	8.0 ± 2.8	[22]
Mg/0.7 Y_2O_3		45 ± 2	157 ± 10	244 ± 1	8.6 ± 1.2	
Mg/0.3 ZrO_2		40 ± 1	85 ± 8	139 ± 8	8.1 ± 1.6	
Mg/0.6 ZrO_2		42 ± 2	117 ± 11	182 ± 14	9.4 ± 2.7	[23]
Mg/1.0 ZrO_2	Hybrid microwave sintering	42 ± 2	98 ± 6	158 ± 12	8.6 ± 2.2	
Mg 10 SiC		44 ± 1	140 ± 2	165 ± 2	1.5 ± 0.8	[12]
Mg/0.3 β-SiC		40 ± 1	132 ± 14	194 ± 11	6.3 ± 1.0	
Mg/0.5 β-SiC		42 ± 1	144 ± 12	194 ± 10	7.0 ± 2.0	[20]
Mg/1.0 β-SiC		43 ± 2	157 ± 22	203 ± 22	7.6 ± 1.5	
Mg/0.3 Cu		49 ± 1	188 ± 13	218 ± 11	5.9 ± 1.1	
Mg/0.6 Cu		52 ± 2	237 ± 24	286 ± 8	5.4 ± 1.2	[24]
Mg/1.0 Cu		60 ± 3	194 ± 17	221 ± 17	2.9 ± 0.4	
Mg (0.3 ZrO_2 + 0.7 Cu)		48 ± 1	196 ± 16	249 ± 8	8.2 ± 1.1	[23]
Mg (0.6 ZrO_2 + 0.4 Cu)		50 ± 1	139 ± 22	193 ± 21	11.4 ± 2.9	
Mg (0.7 Y_2O_3 + 0.3 Ni)		54 ± 4	221 ± 7	244 ± 1	9.0 ± 0.9	
Mg (0.7 Y_2O_3 + 0.6 Ni)		60 ± 4	232 ± 8	262 ± 6	9.5 ± 0.9	[26]
Mg (0.7 Y_2O_3 + 1.0Ni)		63 ± 4	228 ± 8	272 ± 2	5.5 ± 0.7	

Table 4. *Cont.*

Materials	Processing Method	Microhardness HV	0.2% YS (MPa)	UTS (MPa)	Failure Strain (%)	Ref.
Compressive properties						
Pure Mg	Hybrid microwave sintering	-	109 ± 4	284 ± 11	23 ± 3	[23]
Mg/0.3 ZrO_2		40 ± 1	109 ± 6	273 ± 13	19 ± 1	
Mg/1.0 ZrO_2		42 ± 2	109 ± 5	262 ± 18	19 ± 4	
Mg/(0.3 ZrO_2 + 0.7 Cu)		48 ± 1	124 ± 7	352 ± 18	12 ± 3	
Pure Mg		43 ± 2	70 ± 6	265 ± 8	16.2 ± 0.8	[10]
Mg/3 $Ni_{60}Nb_{40}$		62 ± 4	85 ± 4	283 ± 10	17.6 ± 1.1	
Mg/5 $Ni_{60}Nb_{40}$		84 ± 5	130 ± 11	320 ± 11	18.4 ± 1.3	
Mg/10 $Ni_{60}Nb_{40}$		95 ± 5	90 ± 7	322 ± 10	17.2 ± 1.6	
Tensile properties						
Pure Mg	PM + hot extrusion	-	215	230	7.0	[28]
Mg/3.0 SiC (mixed)		-	180	220	3.0	
Mg/3.0 SiC (milled)		-	220	280	2.0	
Pure Mg	PM + hot extrusion	-	134 ± 11	190 ± 10	4.6 ± 0.6	[31]
Mg/0.5 Al		-	218 ± 16	271 ± 11	6.2 ± 0.9	
Mg/1.0 Al		-	185 ± 9	226 ± 12	3.3 ± 1.0	

For hybrid microwave sintered nanocomposites, all the nanocomposites reinforced with ceramic oxides and carbides displayed an improvement in strength except for $MgZrO_2$ nanocomposites due to clustering of ZrO_2 particulates. Improvements in ductility were observed for all Mg nanocomposites reinforced with ceramic oxides and carbides reinforcements. The largest improvement in strength was observed with the addition of metallic Cu nanoparticles with an improvement of 104% in 0.2% YS and 70% in UTS in Mg/0.6 Cu nanocomposites but ductility was reduced.

Addition of metallic reinforcements increases strength but reduces ductility of composites while ceramic reinforcement increases ductility but strength improvement is less than that of metallic reinforcement. Hence, hybrid (Ceramic + Metallic) Mg nanocomposites were investigated and found to provide an optimum combination of strength and ductility surpassing that of conventional Mg + Ceramic or Mg + Metallic nanocomposites [23,26]. In particular, Mg (0.3 ZrO_2 + 0.7 Cu) and Mg (0.7 Y_2O_3 + 0.6 Ni) hybrid nanocomposites were able to achieve significant improvement in both strength and ductility over $MgZrO_2$ and MgY_2O_3 nanocomposites.

The improvement in 0.2% YS and UTS can be attributed to Orowan strengthening, grain refinement and internal strain and thermal stresses between reinforcements and matrix. Orowan strengthening occurs due to the presence of nano-size reinforcements, which hinders the movement of dislocations. Grain refinement arises due to the presence of reinforcing particles, which acts as nucleation sites during solidification or recrystallization and the pinning of grain boundaries resulting in limited grain growth. The strain and CTE mismatch between the matrix and the nano-size particles contributes to an increase in dislocations around the interfacial region between the matrix and the particles thus strengthening the material.

The increase in failure strain for ceramic oxides and carbides may be due to the activation of non-basal slip systems [32] but is dependent on the distribution of nano-particulates and volume fraction

of reinforcements added. For metallic reinforcements, the reduction in ductility with larger volume fraction of reinforcement is due to the increasing presence of harder metallic reinforcements and the formation of intermetallic phases.

A comparison of the tensile properties of magnesium composites synthesized using hybrid microwave sintering in this study with other magnesium composites by powder metallurgy indicates that comparable properties can be achieved even with a significant reduction in processing time.

Similar compressive yield strength was observed between pure Mg and Mg/ZrO_2 composites while average ultimate compressive strength was marginally reduced in case of Mg/ZrO_2 composites when compared to pure Mg. This may be due to the similar grain sizes between pure Mg and $MgZrO_2$ composites. It is well known that yielding in magnesium materials is mainly due to twinning and compressive yield strength of magnesium can be increased by reducing the twinning activity through grain refinement [23]. For $Ni_{60}Nb_{40}$ amorphous particle reinforced composites, the optimum improvement is observed with addition of 5% volume fraction of reinforcement leading to an improvement of ~85% in compressive yield strength, ~30% in ultimate strength and ~14% in ductility.

3.5. Fractography

The results of fracture surface analysis revealed a predominantly brittle fracture in the case of Mg samples (see Figure 8a). This can be attributed to the HCP crystal structure of magnesium that restricts the plastic deformation to $\{0001\}\langle 11\bar{2}0\rangle$ basal slip and $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ pyramidal twining at temperature below 498 K [33]. The presence of cleavage steps and microscopically rough fracture surface indicates the inability of magnesium to deform significantly under uniaxial tensile loading. For the Mg composite formulations, the fracture surface revealed a predominantly brittle fracture with refined fracture features (see Figure 8b–e)

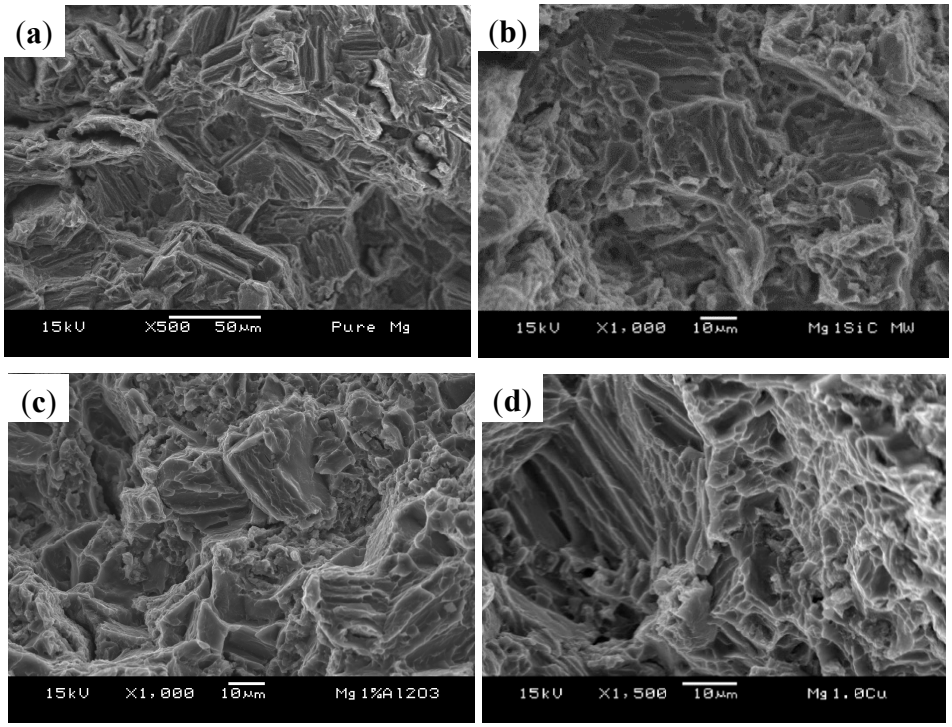

Figure 8. *Cont.*

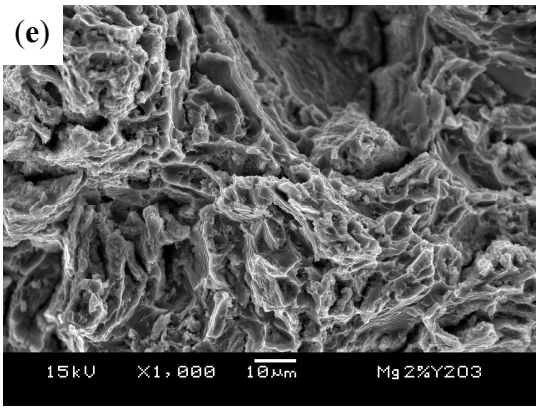

Figure 8. Representative SEM micrographs of the tensile fracture surface for: (**a**) monolithic Mg; (**b**) Mg 1.0 SiC; (**c**) Mg 0.6 Al_2O_3; (**d**) Mg 1.0 Cu; and (**e**) Mg 0.7 Y_2O_3.

4. Conclusions

(1) It has been shown that pure magnesium and magnesium composites can be synthesized using a hybrid microwave sintering technique that utilizes microwave energy and heat from external susceptors for sintering.
(2) Significant reduction in sintering time was achieved through rapid heating, higher sintering temperature and the elimination of holding time without any detrimental effect on the end properties of the sintered magnesium materials.
(3) Potential cost savings can be realized with the reduction in sintering time and sintering under atmospheric condition without the need for an inert atmosphere.
(4) Microstructural characterization revealed finer microstructure for microwave-sintered magnesium when compared to conventionally sintered magnesium.
(5) The nano-size reinforcements formed a continuous network along the grain boundaries of the matrix.
(6) Mechanical characterization revealed an increase in hardness, 0.2% YS and UTS of magnesium with the addition of nano-size reinforcements. Failure strain was improved with the addition of SiC and Al_2O_3 ceramic reinforcements but displayed the opposite trend with the addition of metallic copper as reinforcement.

Acknowledgments

Manoj Gupta would like to acknowledge MOE Tier 1 grant R265 000 496 112.

Author Contributions

Both authors contribute equally to the preparation of the review article and have read and approved the final manuscript.

Conflicts of Interest

The authors declare no conflict of interest.

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