

**Progress in aluminum and magnesium matrix composites obtained by spark plasma,  
microwave and induction sintering**

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## **Abstract**

Aluminum and magnesium matrix composites are attractive lightweight materials with strength surpassing that of the corresponding matrix metals. Recent years have seen extensive research aimed at finding ways to control the structure of metal matrix composites and obtaining materials with new sets of properties. This review aims to show that field-assisted sintering is a promising approach for the powder metallurgy processing of aluminum and magnesium matrix composites. The review focuses on the achievements in the compositional and microstructural design and properties of aluminum and magnesium matrix composites obtained by spark plasma sintering, microwave sintering, and induction sintering. Issues related to the process scale-up in the field-assisted sintering technologies are also addressed.

**Keywords:** metal matrix composites; aluminum; magnesium; alloy; spark plasma sintering; induction sintering; microwave sintering.

## **Introduction**

The weight of structural elements is an important factor for automotive, aerospace, marine, and medical applications. In recent years, extensive research has been conducted to develop materials capable of providing the desired strength while keeping the weight of a structure as low as possible. The innovative car concepts have adopted the multi-material design idea, the essence of which is to use the “best” material available for each function [1].

Aluminum and magnesium matrix composites are attractive lightweight materials with strength surpassing that of the corresponding matrix metals. The composites can be based not only on aluminum or magnesium matrices but also on Al- and Mg-based alloys. Along with light weight and high strength, the attractive properties of aluminum alloys are high corrosion resistance, good ductility, thermal and electrical conductivity, good machinability, and a variety of possible finishes. German [2] marks light metals (titanium, aluminum and magnesium) as one of the future prospects of the sintering science and technology. The powder metallurgy approach is selected to make materials with refined microstructures compared with those produced by ingot metallurgy. The market for sintered aluminum parts has included business machines (with the greatest variety of products), automotive components, aerospace components, power tools, appliances, and structural parts. Sintered aluminum-based materials are used for the fabrication of gears, levers, components for office equipment, medical equipment, and casings of small electrical motors.

For composites, higher microstructure versatility is a feature of the powder metallurgy processing as compared with casting or infiltration methods. Tailored microstructures often lead to improved mechanical performance and corrosion resistance. In order to be used as structural materials, aluminum alloys should possess strength above 500 MPa at room temperature and tensile elongation of 5% (high-strength alloys) [3]. Sintered aluminum alloy parts can be made competitive with Al-based cast products, Al-based extruded materials and other sintered metallic materials.

Magnesium and its alloys possess attractive properties such as low density, good damping performance, biocompatibility, recyclability, and large hydrogen storage capacity, which give them prospects in aerospace, transportation, biomedical, and energy sectors [4]. The difficulties that need to be overcome include low strength, insufficient plasticity and low corrosion resistance of magnesium alloys. The application of magnesium alloys is limited by lower ductility and lower mechanical strength as compared with aluminum alloys. Generally, aluminum alloys are less expensive than magnesium alloys [5].

Recent reviews covering certain areas of field-assisted sintering of metal matrix composites indicate a growing interest to the possibilities of scientific and technological upgrade of the conventional processing of these materials [6, 7, 8, 9, 10]. The present review aims to highlight the features of field-assisted sintering of lightweight MMCs – composites with Al/Al alloy and Mg/Mg alloy matrices – and mainly covers the literature published in the past decade. The review discusses the principles of the microstructure design, the structure formation and mechanical and functional properties of the Al/Al alloy and Mg/Mg alloy matrix composites obtained by field-assisted techniques – spark plasma, microwave and induction sintering.

## **Physical principles of spark plasma, microwave and induction sintering and their application to aluminum and magnesium**

### *Spark plasma sintering*

Spark Plasma Sintering (SPS) is a modern sintering technique, in which the sample/tooling assembly is heated by pulsed direct current [11, 12, 13, 14, 15, 16, 17, 18, 19]. The main feature of SPS is the simultaneous application of electric current and uniaxial pressure to the sample (Figure 1). The die and the punches are usually made of graphite, so the electric current passes through the punches and the die and also through the sample in the case of sintering of conductive materials. A historical overview of the development of the field-assisted sintering methods, including SPS, can be found in monograph [18]. In the SPS, the electric

current is applied in the form of pulses, usually 3 ms long. In many laboratory facilities, the pulse sequence is such that 12 pulses are followed by an “off” period having a duration of two pulses. The voltage in the SPS facilities does not usually exceed 10 V.

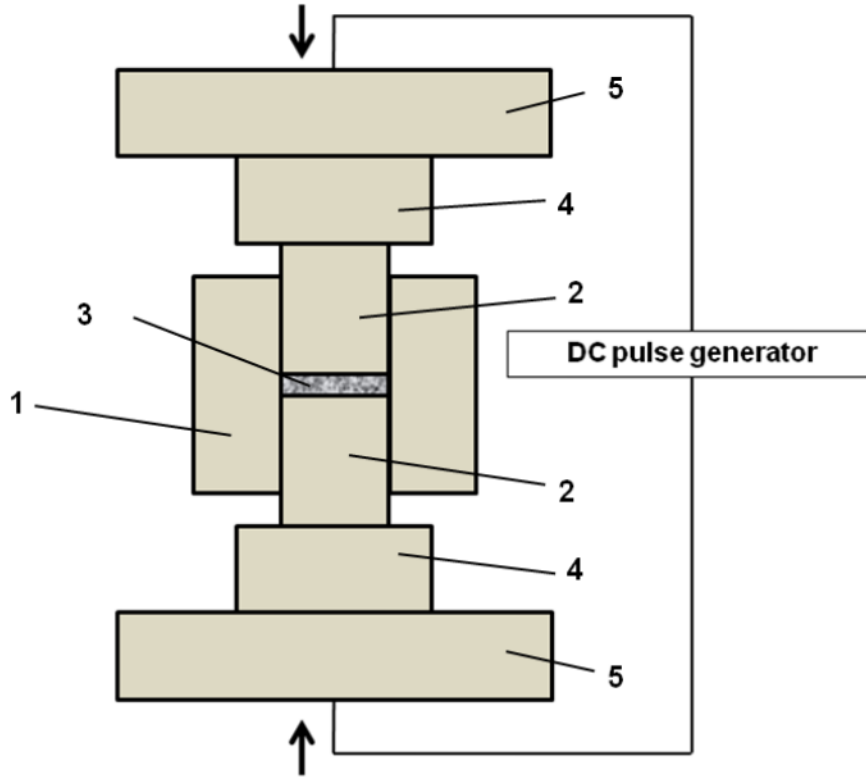


**Figure 1.** Schematic representation of the tooling/specimen assembly utilized in the SPS processing: 1 – graphite die, 2 – graphite punches, 3 – sample, 4, 5 – graphite spacers.

Apart from Joule heating, electric-current effects discussed in the context of SPS are electromigration [20], electroplasticity [21], and plasma formation [11]. The electric field effect on mass transport can be taken into account by applying the electromigration theory [22]. The flux  $J_i$  of the diffusing species can be expressed as

$$J_i = -\frac{D_i C_i}{RT} \left[ \frac{RT}{\partial x} \frac{\partial \ln C_i}{\partial x} + F z^* E \right] \quad (1)$$

where  $D_i$  is the diffusivity of the  $i$ th species,  $C_i$  is the concentration of the species,  $F$  is Faraday's constant,  $z^*$  is the effective charge on the diffusing species,  $E$  is the field strength,  $R$  is the gas constant and  $T$  is the absolute temperature. Effective charge  $z^*$  is a parameter, with which the strength of the electromigration effect is measured. It can be expressed as a sum of  $z_{wd}$ , a contribution from the electron wind, and  $z_{el}$ , an electrostatic component:

$$z^* = z_{wd} + z_{el} \quad (2)$$

In some investigations, the possibility of plasma formation was considered as a mechanism of fast densification of metals and metal-containing mixtures [23, 24]. It should be noted that Hulbert et al. [25] found no evidence of the plasma state of matter in the normal SPS runs. However, at voltages higher than those usually used in the SPS furnaces, evaporation of metals and transition of the gaseous phase into an ionized gas is possible, as demonstrated by Saunders et al. [26].

With sintering acceleration observed in the SPS processes relative to conventional sintering and hot pressing, the role of overheating of the inter-particle contacts is of great importance. This question has been studied theoretically and experimentally [27, 28, 29]. Kuz'mov et al. [28] showed that the contact temperature may be substantially higher than the average macroscopic temperature of the compact such that melting of the metal can take place in the vicinity of the contact.

An important question for the SPS processing of metallic powders is the possibility of removal/disruption of oxide films present on the surface of the particles. For oxides that are easily reduced by carbon under conditions of SPS, direct contact with graphite foil or tooling was shown to be critical for surface cleaning and reduction of the oxygen content in the sintered material [30]. For oxides, such as  $Al_2O_3$ , which cannot be reduced by carbon at temperatures used for sintering of the metal (Al), a loss of integrity of the oxide film can occur, the concentration of the oxide in the material remaining unchanged. An interesting work was

reported by Li et al. [31], who found that the heating rate in the SPS influences the concentration of the oxides in the sintered material. An  $\text{Al}_{86}\text{Ni}_6\text{Y}_{4.5}\text{Co}_2\text{La}_{1.5}$  glassy alloy in the form of powder was subjected to SPS using two different heating rates ( $5\text{ }^{\circ}\text{C min}^{-1}$  and  $40\text{ }^{\circ}\text{C min}^{-1}$ ). The concentration of oxygen in the material was significantly reduced after SPS conducted at the higher heating rate. Efficient densification of the powder at the higher heating rate was attributed to the removal of the oxide films under the conditions of passing electric current. The evidence of more efficient densification at  $40\text{ }^{\circ}\text{C min}^{-1}$  is shown in the corresponding micrographs of the sintered compacts (Figure 2). Xu et al. [32] indicated that a more significant volume expansion of the metal relative to that of the oxide upon heating caused the disruption of  $\text{Al}_2\text{O}_3$  films facilitating the formation of metallurgical bonds between the Al particles. Melting of aluminum can also contribute to the inter-particle bonding quality. The above described examples show that surface cleaning may play a significant role in the formation of bulk metals during SPS; however, it cannot be regarded as a phenomenon always present in the SPS processing of metallic powders. The possibility of surface cleaning and the underlying physical/chemical mechanisms of the oxide film removal depend on many factors including the chemical nature of the oxide, the thickness of the film, and the crystal structure of the film and its mechanical properties.

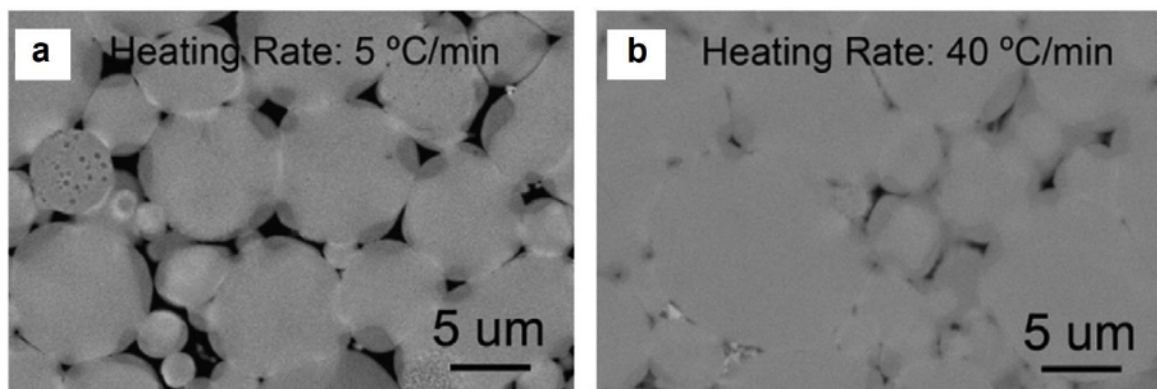


**Figure 2.** Microstructure of compacts obtained from an  $\text{Al}_{86}\text{Ni}_6\text{Y}_{4.5}\text{Co}_2\text{La}_{1.5}$  glassy powder by SPS at  $248.5\text{ }^{\circ}\text{C}$  at different heating rates: (a)  $5\text{ }^{\circ}\text{C min}^{-1}$ , (b)  $40\text{ }^{\circ}\text{C min}^{-1}$ . Reprinted from [31], Copyright (2013), with permission from Elsevier.

### *Microwave sintering*

Microwave sintering uses the energy of microwave electromagnetic field, which is converted into the thermal energy [33, 34, 35, 36, 37]. An important advantage of microwave heating is volumetric energy absorption in many materials. Since the power generated by the microwave source can be fully converted into the thermal energy to heat the sample (without the need to heat the furnace elements), microwave sintering offers much higher heating rates in comparison with conventional processes. While bulk metals are opaque to microwaves, metals in the powder form are good microwave absorbers [36, 37]. The susceptor material can be used when the powder to be sintered poorly absorbs the microwave radiation. In composite systems, one of the components can play the role of a susceptor.

In a microwave sintering set-up (Figure 3), the susceptor material (silicon carbide, SiC) is rapidly heated and provides heat to the specimen. The pre-compacted specimen also absorbs heat. In this manner, hybrid microwave heating occurs ensuring a more uniform heating of the specimen and eliminating the disadvantages of conventional heating or heating by microwaves only [38, 39]. Due to rapid heating and short cycles, microwave sintering of metal-based compacts can be carried out in air without causing deterioration of the mechanical properties of the sintered materials.

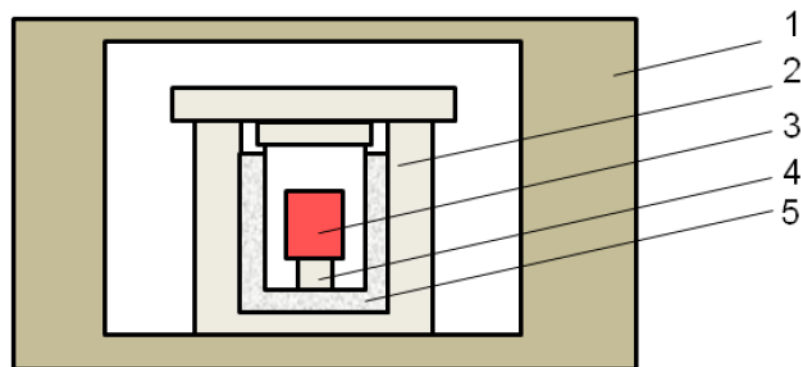


**Figure 3.** Schematic representation of a microwave sintering set-up: 1 – microwave furnace, 2 – crucible, 3 – specimen, 4 – support, 5 – microwave susceptor (SiC powder).

### *Induction sintering*

Induction sintering is based on the use of eddy currents induced in an electrically conductive material [40, 41]. The penetration depth of the eddy currents  $h$  is determined as

$$h = \frac{1}{\sqrt{\pi f \mu \sigma}}, \quad (3)$$

where  $f$  is the frequency,  $\mu$  is the material magnetic permeability and  $\sigma$  is the material electrical conductivity. Induction sintering units are smaller and easier to maintain than conventional sintering furnaces. A conductive container or a die is usually used as a susceptor and is directly heated by the eddy currents (Figure 4). In this case, the powder is heated through radiation and thermal conduction. Alternatively, eddy currents can be induced directly in the pre-consolidated compacts to be sintered. Both schemes of induction sintering ensure fast heating, reduced sintering times, and high energy efficiency.

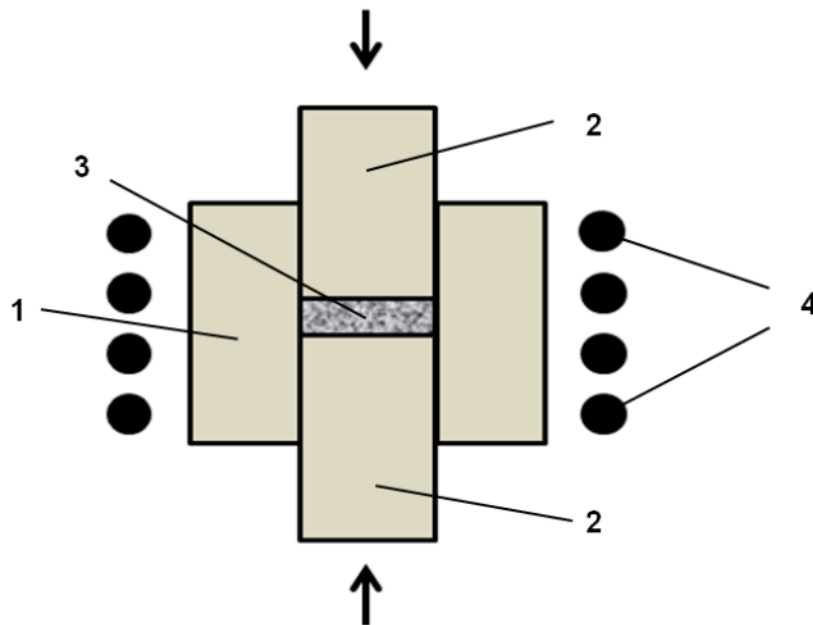


**Figure 4.** Schematic representation of the susceptor (die)/specimen assembly in induction sintering: 1 – die, 2 – punches, 3 – sample, 4 – induction coil.

### *Field-assisted sintering: advantages over conventional sintering*

The advantages of the field-assisted sintering methods over conventional sintering span high heating rates, densification within shorter times, and lower energy consumption. For composites, a much wider range of reinforcements is possible, as, by conventional sintering, it may be rather difficult to preserve certain reinforcing phases in the material. The availability of powder consolidation methods capable of preserving the inner structure of the composite particles in terms of phase composition and phase distribution significantly widens the range of achievable properties of the composites. Normally, in the SPS, induction and microwave sintering processes, the major fraction of the material remains in the solid state. Only in the high-temperature regions developing locally, melting of the material can occur. Therefore, the initial microstructure of the powder particles is of great importance, as it can, to a great extent, be inherited by the sintered material.

### *Field-assisted sintering of unreinforced aluminum and magnesium: mechanisms and resulting materials*

Investigations of field-assisted sintering of Al and Mg powders shed light on the mass transport mechanisms that operate during the processing of Al- and Mg-based composite mixtures. In this section, the sintering behavior of Al and Mg powders is discussed. The only reinforcing element in the sintered metals is the oxide phase originating from the native oxide film unavoidable in the commercial and laboratory-produced powders and powder mixtures.

Olevsky & Froyen developed a constitutive model for SPS that takes into account different transport mechanisms [20]. In that model, the contributions of surface tension, external load and electromigration to shrinkage were jointly analyzed. It was found that the electromigration-related material flux can be a significant component of electric current-accelerated diffusion. The model allowed constructing maps of sintering mechanisms operating during SPS. Such a map for an aluminum powder is shown in Figure 5. The map indicates three

porosity/grain size domains; in each domain, the applied load, surface tension or electromigration dominates as a driving factor of material transport. The power-law creep induced by an external stress dominates at high porosities. At low porosities, the electromigration can become the dominant mechanism. For small particle sizes and low porosity, surface tension was found to be the main driving factor for densification. For very small porosities in the electromigration-dominating zone, the elimination of voids may require externally applied loads. The results of modeling agreed satisfactorily with the experimental data on the shrinkage kinetics of aluminum.

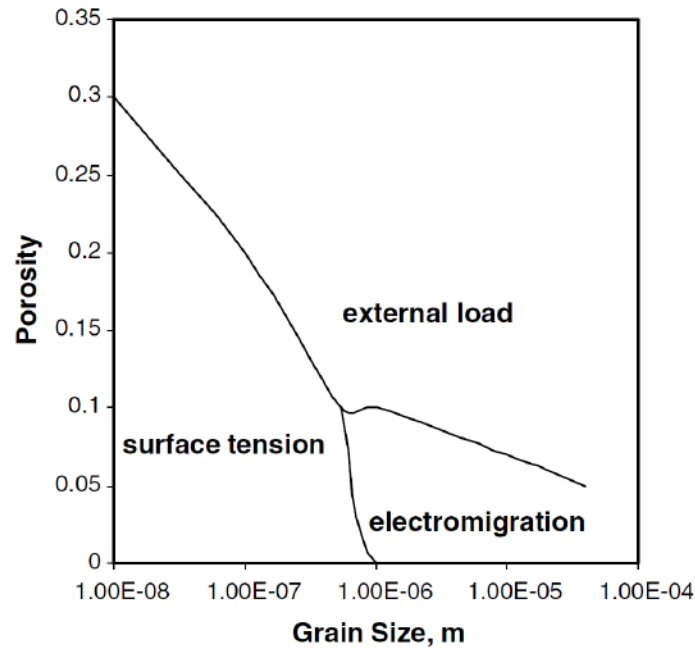


**Figure 5.** Densification map for aluminum,  $U/I=417 \text{ V m}^{-1}$ ,  $T=673 \text{ K}$ ,  $\bar{\sigma}_x=283 \text{ MPa}$ .  $\bar{\sigma}_x$  is effective external stress in  $x$ -direction. Reprinted from [20], Copyright (2006), with permission from Elsevier.

According to Xie et al. [42], there are two types of interfaces in aluminum obtained by SPS of a commercially available powder: metal/metal and metal/oxide/metal. The authors of that work saw the main cause of the destruction of the oxide films present on the surface of the powder in the plastic deformation of the particles of the metal; the deformation was more

significant at a higher temperature. In the bulk material obtained by SPS (600 °C, 1 h) of a ball-milled Al powder having 38 nm crystallites, a bimodal grain size distribution was observed [43]. An electron backscatter diffraction orientation image map of the sintered aluminum is shown in Figure 6. The authors attributed the presence of large grains to the formation of high-temperature regions in the sample during SPS. Virtually all grain boundaries in the sintered material were of high angle (with misorientations  $>15^\circ$ ).

During SPS, the response of the inter-particle contacts, Joule heating and plastic deformation may play significant roles varying with the particle size. Cheng et al. [44] found that smaller magnesium particles showed a less significant shape change and a smaller degree of plastic deformation than larger particles. This was explained by the hindering effect of grain boundaries. Thermal softening, localized melting and filling of the pores by the melt were considered as mechanisms contributing to densification.

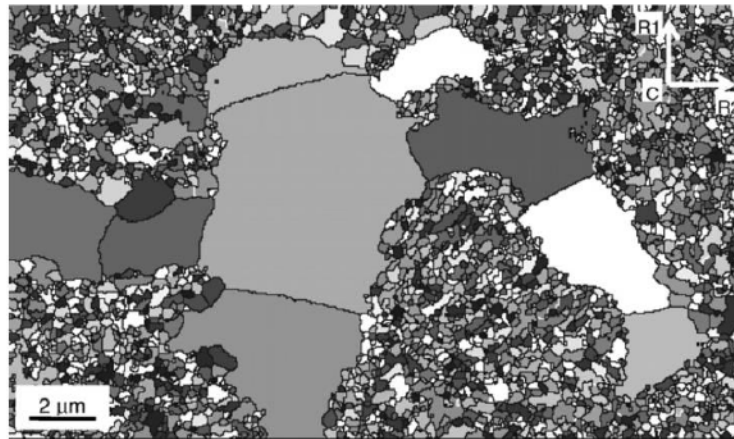


**Figure 6.** Euler contrast orientation image map of bulk aluminum obtained by SPS (600 °C, 1 h) of the ball-milled powder, stearic acid added as a process control agent. Black lines represent misorientations  $>15^\circ$ . C represents the compression direction, and R1 and R2 are two orthogonal directions arbitrarily defined in the compression plane. Reprinted from [43], Copyright (2013), with permission from Elsevier.

When a Mg powder was sintered under a pressure of 60 MPa at 525–585 °C, compacts with relative densities greater than 99% were obtained [23]. Along with grain boundaries, the initial particle boundaries could be observed (Figure 7). It was suggested that the latter were due to oxides still present along the particle boundaries. The boundaries became less visible in samples sintered at higher temperatures. The oxide films remaining in the compacts influenced the fracture behavior of the material: facets corresponding to the initial Mg powder particles were observed on the fracture surface of the compacts sintered at 525 °C.

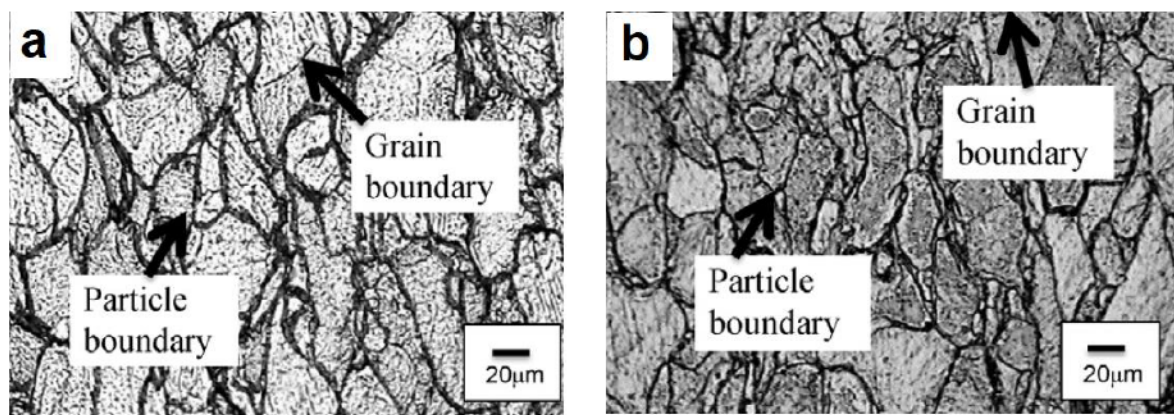


**Figure 7.** Microstructure of magnesium obtained by SPS, 60 MPa, 5 min: (a) 525 °C; (b) 585 °C. Reprinted from [23], Copyright (2011), with permission from Elsevier.

For aluminum and magnesium, microwave sintering can lead to 82–85% reduction in sintering time and energy savings of 96% compared with conventional sintering [38]. In order to demonstrate the result of the hybrid microwave process in terms of achieving uniform heating, hardness of aluminum and magnesium sintered cylindrical specimens was measured along their radius [38]. The hardness of the hybrid microwave sintered compacts did not change along the radius of the specimen, while the compacts obtained by conventional sintering were softer in the center.

The possibility of sintering of aluminum powders by induction heating was shown in ref. [45]: a mechanically milled aluminum powder was sintered using high-frequency induction

heating. The powder was cold-compacted before sintering. The induction sintering process lasted only 3 min and, for simplification, was conducted in air. No new phases were detected in the sintered material, which showed that induction sintering of aluminum is possible in air without any significant oxidation-related effects.

In the processing of composites, the above mentioned mass transport mechanisms are supplemented by those related to the direct interaction of the reinforcing elements with the electric current/electromagnetic radiation and chemical interactions at the matrix/reinforcement interfaces, as discussed in the following sections.

### **Recent trends in the selection and design of reinforcements for aluminum and magnesium matrix composites**

In this section, traditional and novel reinforcement options for aluminum and magnesium matrix composites are presented. All these reinforcements can be used in composites processed by SPS, induction, and microwave sintering. Traditional reinforcements in MMCs are ceramic particles, whiskers and fibers [46]. Sharp corners of the reinforcing elements limit the ductility of these composites [47]. There is a possibility that local overheating can lead to rounding of the sharp corners of the particles of the hard phases during a field-assisted sintering process. The structure formation of ceramic particle-reinforced aluminum matrix composites obtained by field-assisted sintering is the subject of recent publications [48, 49, 50, 51, 52, 53, 54, 55, 56, 57, 58]. Poor wettability between the metal matrix and the ceramic reinforcement leads to the formation of difficult-to-eliminate porosity at the interfaces. The porosity can also concentrate within the agglomerates or clusters of the reinforcing particles. Sweet et al. [54] produced aluminum matrix composites reinforced with particles of SiC, AlN or Si<sub>3</sub>N<sub>4</sub> by SPS. They found that full densification of composites containing fine ceramic particles was problematic due to the presence of ceramic clusters. The extent, to which aluminum could be forced into those (cluster) regions, determined the residual porosity of the composite. The clusters were, therefore, the

principal source of residual porosity of the sintered composites. After sintering at low temperatures, pores were present between the ceramic particles in a cluster, as aluminum did not penetrate the cluster. As the sintering temperature was increased, partial or full penetration of the metal into the clusters was observed.

As seen from examples of ref. [54], ceramic particles in a metal matrix, if clustered, do not sinter with each other under consolidation conditions selected based on the properties of the metal matrix. Those clusters, therefore, comprise a disproportionate part of the total porosity of the composite. In order to fully eliminate the porosity, it is desirable to design composites in such a manner that the particles of the matrix material and those of the reinforcement sinter within the same temperature range. The idea behind the use of metals or metallic alloys as reinforcements is based on the introduction of a harder and stronger metal into matrices of softer metals. In the case of reactive metals, intermetallic layers can form between the particle of the harder metal and the matrix [59, 60]. The reinforcement will thus be represented by particles with a core-shell structure.

A rapidly developing trend in the area of MMCs with Al/Al alloy and Mg/Mg alloy matrices is to use metallic glass as a reinforcement. Above their glass transition temperatures, metallic glasses enter a technologically attractive Newtonian flow in the supercooled liquid region  $\Delta T_x$  between the glass transition temperature  $T_g$  and the crystallization temperature  $T_x$ . The advantage of metallic glass is its softness and deformability within the supercooled liquid region  $\Delta T_x$  and its very high strength at ambient temperatures [61]. Similar to oxide glasses, in the supercooled liquid region  $\Delta T_x$  above  $T_g$ , the viscosity of metallic glasses drops drastically and they become liquid-like. Owing to their excellent mechanical properties at room temperature, metallic glasses present a highly promising alternative to ceramic reinforcements, being particularly suitable for strengthening low-melting temperature metal matrices, such as Al and Mg and their alloys. The composite materials should be designed in such a way that the chosen sintering temperature of the composite powders is within the supercooled liquid region  $\Delta T_x$  of

the glass while being close to the solidus temperature of the matrix alloy. Figure 8 illustrates this approach and schematically shows a drop in the yield stress in metallic glasses and Mg alloys (as possible matrix materials) with the temperature [62]. After compaction to full density and cooling to below  $T_g$  and further down to room temperature, the glassy particles become the hard reinforcement phase. Consolidation of the metallic glass-matrix metal (alloy) powder mixtures at temperatures below  $T_g$  is also possible, if high pressures are used and a temperature-related viscosity drop of the metallic glass is not necessary for eliminating the porosity.

The limitations imposed by the need to preserve a metastable phase may, in some cases, prevent high-quality sintering of the composite as a whole. As an alternative to metallic glasses, high-entropy alloys [63] are suggested for the metal reinforcing purposes. These alloys are thermally stable and possess enhanced mechanical properties relative to conventional alloys. Owing to inherent high hardness and strength [64], quasicrystalline alloys can also be used as reinforcing phases to strengthen softer metallic matrices.

Extensive research has been carried out to find the possibilities of incorporating carbon nanotubes (CNTs) and graphene into Al and Mg matrices [65, 66, 67, 68]. The use of hybrid reinforcements (two or more reinforcing phases in the same composite) is justified when each reinforcing phase plays a specific role [69, 70, 71]. Kwon et al. [69] obtained aluminum matrix composites reinforced with CNTs and nanosized SiC particles. The presence of SiC facilitated mixing of aluminum and CNTs during ball milling, so the particles of SiC could be considered as a mixing agent. At the same time, CNTs acted as a lubricant during milling. In the sintered material, particles of SiC reduced the contact area between the Al matrix and CNTs and amount of  $Al_4C_3$  formed at the interface.

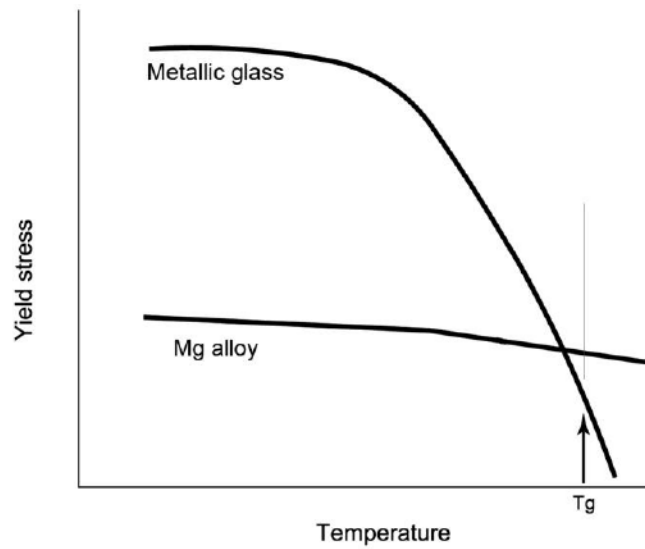


**Figure 8.** Schematic illustration of the change in the yield stress of a metallic glass and a Mg alloy with increasing temperature.  $T_g$  is the glass transition temperature of the metallic glass.

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### **Features of the in-situ synthesis of reinforcements during field-assisted sintering**

Synthesizing new phases and consolidating the powder within the same field-assisted process offers, along with a technological advantage of reducing the number of processing stages, a possibility of influencing the chemical reactivity of the materials under a specific action of electric field. Historically designed for solid-state sintering, SPS has emerged into a method of solid-state synthesis under applied electric current and mechanical pressure [72]. SPS dies can be used as chemical reactors for conducting the syntheses of single-phase materials and composites. As, in real SPS processes, the heated and compressed sample is, in most cases, a mixture of powders, an increase in the chemical reactivity under the intrinsic action of electric current is not always easy to quantify. In the reaction mixtures, the contact conditions between the particles of the reactants influence the current density and the temperature in the vicinity of the inter-particle contacts.

During reactive SPS, Kirkendall effect can cause the formation of pores. Zhang et al. [59] studied the formation mechanism of tungsten aluminides in 80 at.% Al-20 at.% W mixtures

subjected to SPS. The aluminum matrix composites were obtained through solid-state diffusion. The particle size of the metallic powders in the initial mixture was found to influence the phase formation and the microstructure of the solid-state sintered material. The reinforcements consisted of W particles surrounded by  $\text{Al}_4\text{W}$  and  $\text{Al}_{12}\text{W}$ , when micrometer-sized particles were used for the synthesis. The material was not well densified owing to the occurrence of Kirkendall effect.

The sintering time was found to be critical for obtaining materials with improved mechanical strength by reactive SPS. Rodríguez et al. [73] produced aluminum matrix composites by adding small amounts of zirconium to aluminum (0.5 wt.%, 1 wt.% and 1.5 wt.%) and sintering the mixtures by SPS. In order to introduce zirconium into aluminum, a colloidal process based on the use of a zirconium alkoxide was applied. After 3 min of holding the alloy at 625 °C during SPS, no intermetallic particles were observed in the microstructure; however, after 1 h,  $\text{ZrAl}_3$  platelets nucleated and grew, becoming visible in the microstructure as inclusions in the Al matrix. Zirconium additions significantly decreased the mechanical strength of the composites relative to pure aluminum when sintering time was only 3 min. The Zr-containing alloys sintered for 1 h showed higher stiffness and yield strength than the unreinforced aluminum sintered under the same conditions.

In the past decades, microwave energy has been widely used for the synthesis of materials [33], including the synthesis in the combustion mode [34]. The advantage of the energy transfer by means of microwaves over conventional heating is the direct interaction of the electromagnetic waves with the reactants and intensification of the reaction processes. In both processes, heating up to the ignition temperatures is possible without a susceptor. When microwaves are used for the ignition, the target temperature is first reached in the center of the sample, and the combustion front propagates from the center through the entire volume. After the reaction is complete, the process can continue to sinter the synthesized material into a bulk

sample. Induction heating can also be used to for the synthesis and ignition of self-propagating high-temperature reactions in powder mixtures [74].

### **Development of the interfaces between the matrix and the reinforcement in the processes of rapid sintering**

Using methods of rapid sintering, it is possible to obtain matrix/reinforcement interfaces formed by interdiffusion across distances of only a few nanometers. A “non-equilibrium” interface was obtained in aluminum matrix composites reinforced with CNTs [75]. The “non-equilibrium” term was used to describe an interface that formed prior to crystallization of the carbide,  $\text{Al}_4\text{C}_3$ , layer. The layer containing Al and C was formed between the matrix and CNT due to interdiffusion of the elements. The formation of amorphous interfacial layers was possible owing to rapid cooling after SPS (at a rate of  $500\text{ }^\circ\text{C min}^{-1}$ ). Carbon contained in this layer originated from an amorphous carbon coating on the surface of CNTs formed during the powder processing before sintering. The formation of the “non-equilibrium” interface allowed improving the tensile properties of the composites.

In order to enable load transfer from an Al matrix to multi-walled CNTs (MWCNTs), certain amounts of  $\text{Al}_4\text{C}_3$  should be allowed to form [76]. The following aspects of the formation of  $\text{Al}_4\text{C}_3$  at the interface were highlighted: (1)  $\text{Al}_4\text{C}_3$  may bridge the outmost and inner walls of the MWCNT preventing peeling of the outmost wall from the inner walls; (2) nano-sized  $\text{Al}_4\text{C}_3$  enhances the interfacial shear resistance between the MWCNTs and the Al matrix, preventing the pullout of MWCNTs from the matrix. As the concentration of  $\text{Al}_4\text{C}_3$  is critical, it is important to precisely control the conditions of heat treatment of the composites. In ref. [76], an induction furnace was used to controllably heat-treat composites sintered by SPS.

The features of the interfaces developing in  $\text{Zr}_{65}\text{Cu}_{18}\text{Ni}_7\text{Al}_{10}$  metallic glass-Al 7075 composite obtained by SPS were studied by Wang et al. [77]. The composite consists of a face-centered cubic (fcc) Al matrix, an amorphous alloy reinforcement and precipitates in the matrix.

The selected-area electron diffraction (SAED) pattern of the area corresponding to Figure 9a indicates the presence of an amorphous phase and nanocrystalline fcc-Al (Figure 9c). SAED patterns taken from area 1 (metallic glass reinforcement), area 2 (fcc-Al) and the MgZn<sub>2</sub> precipitate of Figure 9a are shown in Figure 9d, e, f, respectively. The precipitate was confirmed to be the  $\eta$ -MgZn<sub>2</sub> phase (Figure 9f). The interface between the metallic glass and the fcc-Al matrix was observed using high-resolution transmission electron microscopy (HRTEM), as shown in Figure 9b. A thin layer (thickness of 2–3 nm) was formed at the interface as a result of short to medium range atomic diffusion. This thin interdiffusion layer provided good bonding between the matrix and the reinforcing phase enabling efficient load transfer from the matrix to the reinforcements upon mechanical loading. The limited diffusion between the two phases favored interfacial bonding and was due to fast consolidation of the powders at a moderate temperature (SPS at 300 °C/600 MPa/10 min).

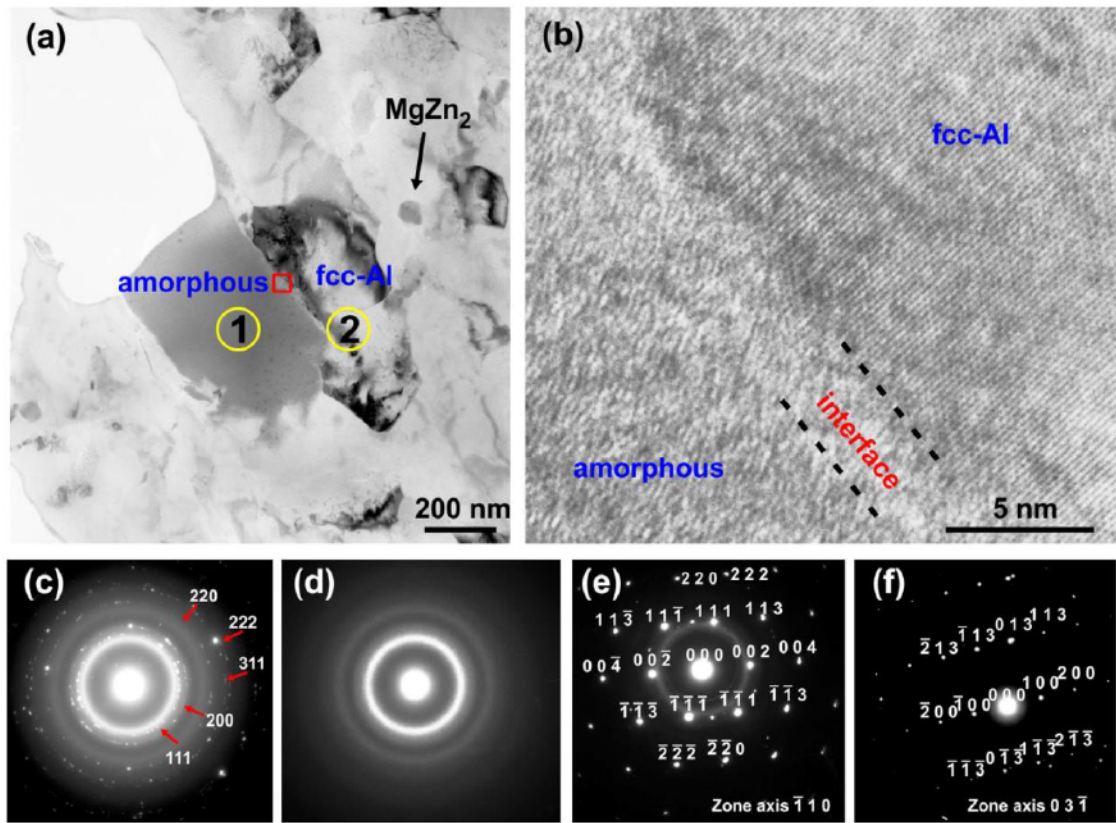


**Figure 9.** TEM/SAED microstructure of  $Zr_{65}Cu_{18}Ni_7Al_{10}$  metallic glass-Al 7075 composite obtained by SPS: (a) bright-field TEM image, (b) HRTEM image of the interface between the metallic glass and the fcc-Al matrix, (c) SAED of the area corresponding to (a), (d) SAED of area 1 indicating an amorphous structure, (e) SAED of area 2 indicating fcc-Al structure, (f) SAED of the precipitate phase indicating the  $\eta$ - $MgZn_2$  structure. Reprinted from [77], Copyright (2016), the article is available under a Creative Commons Attribution 4.0 International License, <http://creativecommons.org/licenses/by/4.0/>

## Microstructure formation of aluminum and magnesium matrix composites obtained by field-assisted sintering

### *Aluminum and magnesium matrix composites obtained by SPS*

In this section, examples of the microstructure evolution of aluminum and magnesium matrix composites during SPS are presented and discussed for systems of different compositions. The evidence of local overheating during SPS is also analyzed.

An interesting microstructure development was observed during SPS of an Al powder ultrasonically mixed with BN nanoparticles [78]. As seen in Figure 10a, the agglomerates of BN nanoparticles are located in the spaces between the Al particles after SPS at 600 °C for 5 min. When the holding time was increased to 60 min, the BN nanoparticle regions were intermixed with aluminum (Figure 10b). Intermixing was attributed to local melting of aluminum at the inter-particle contacts during SPS. The real temperature of the sample was also found to be slightly higher than the measured temperature, as, when aluminum was sintered at a temperature of 620 °C measured in the graphite die, molten aluminum was found to squeeze out of the die.

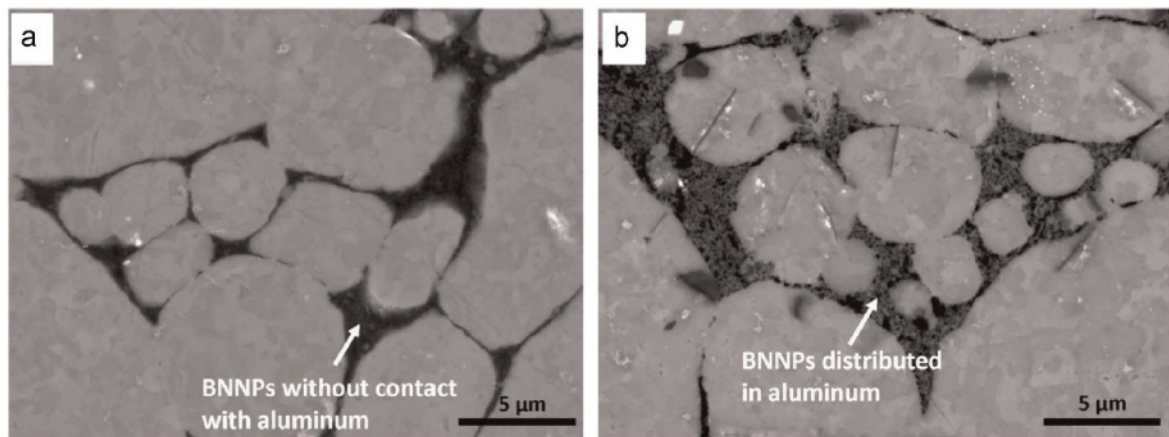


**Figure 10.** Microstructure of Al-4.5 wt%BN nanoparticle composites obtained by SPS at 600 °C: (a) holding time 5 min, (b) holding time 60 min (back-scattered electron images). Reprinted from [78], Copyright (2015), with permission from Elsevier.

Guan et al. [79] emphasized the importance of controlling the interfacial interactions between the aluminum matrix and the metallic glass reinforcement. By using SPS and hot rolling conducted at a temperature below the crystallization temperature of the metallic glass, it was possible to preserve the cores of the reinforcing particles amorphous with only outer layers crystallized and reacted with aluminum to form  $\text{FeAl}_3$  (Figure 11).

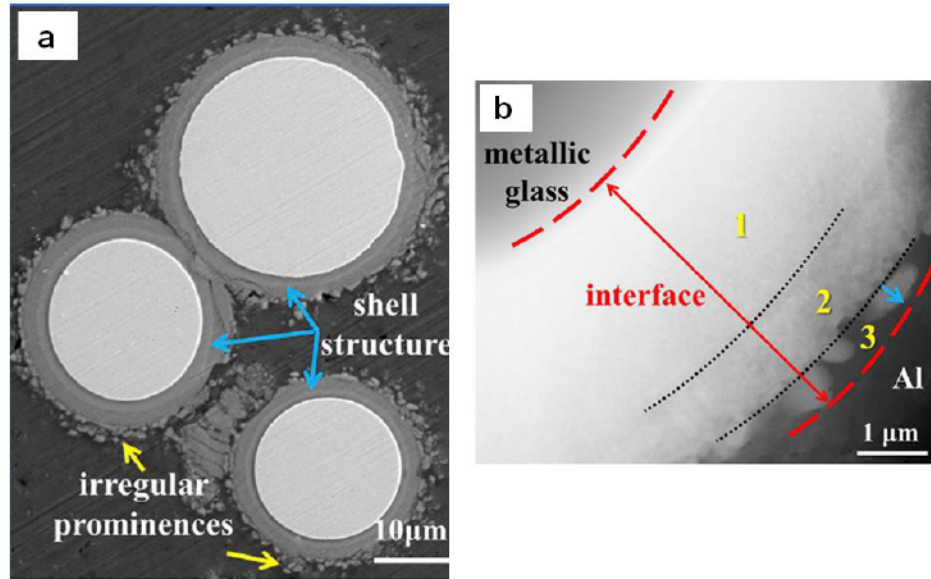


**Figure 11.** Microstructure of the composite obtained by SPS of 40 vol.%  $\text{Fe}_{50}\text{Cr}_{25}\text{Mo}_9\text{C}_{13}\text{B}_3$ -Al mixture followed by hot rolling (a) and the structure of the interface (b). Reprinted from [79], Copyright (2019), with permission from Elsevier.

During SPS at 550 °C, a diffusion layer was formed between  $\text{CoCrFeMnNi}$  high-entropy alloy particles and the Al 2024 alloy matrix [80]. The layer was serrated on the aluminum alloy side. The authors suggested that this layer formed under conditions of high local temperatures developed during SPS. The hardness of the composite material was found to be higher than that of the aluminum matrix and higher than predicted by the rule of mixtures due to the presence of the interdiffusion layer and a gradient interface microstructure beneficial for reducing the stress concentration at the interface. Liu et al. [81] showed that, in composites sintered from a mixture

of Al and AlCoCrFeNi high-entropy alloy powders, the thickness of the interfacial layer increases with the sintering temperature. This layer has an increased concentration of aluminum relative to the high-entropy alloy and presents an Al-based solid solution containing Co, Cr, Fe and Ni. The grains of the transition layer were found to be in the sub-micrometer range, while those of the Al matrix and the high-entropy alloy were 20  $\mu\text{m}$  and 4  $\mu\text{m}$ , respectively. Finer grains of the diffusion layer were explained by the formation of melt upon the passage of current followed by rapid solidification. The authors considered this layer to be favorable for the transition from the iso-stress to the iso-strain deformation mode of the composite. When the interfacial layer reached a certain thickness (after sintering at 580 °C), the hardness of the composite was close to the upper bound calculated from Voigt model.

A high-entropy  $\text{Al}_{0.6}\text{CoCrFeNi}$  alloy was used to modify the properties of an amorphous  $\text{Al}_{65}\text{Cu}_{16.5}\text{Ti}_{18.5}$  matrix [82]. The starting high-entropy alloy possessed a single-phase body-centered cubic (bcc) structure. The composite sintered by SPS at 550 °C consisted of the bcc phase, fcc phase, AlCu, AlCu<sub>2</sub>Ti and an amorphous phase (the sintering temperature was higher than  $T_x$  of the initially amorphous matrix alloy powder). The formation of the fcc phase in the composite was attributed to localized high temperatures during sintering, as the consolidation temperature was below the bcc to fcc+bcc transition of the high-entropy alloy. An interdiffusion layer formed between the reinforcing particles and the matrix. The elemental composition of this layer showed that its formation cannot be attributed to (slow) solid/solid interdiffusion or rapid crystallization of the amorphous matrix. It was, therefore, concluded that this layer formed as a result of enhanced diffusion under the effect of localized high temperatures. The authors proposed a model to calculate the temperature increase  $\Delta T$  due to the passage of electric current in different regions of the composite structure. It was concluded that the amorphous layer contacting the high-entropy particle was heated more significantly than the particle itself. The sintered composite material had a very complex microstructure and exhibited high compressive strength ( $\sim 3$  GPa).

A comparison of results of SPS of Al-Fe<sub>66</sub>Cr<sub>10</sub>Nb<sub>5</sub>B<sub>19</sub> powder mixtures with those of SPS of a pre-compacted pellet of the same composition (compaction pressure 400 MPa) suggested that local heating at the interface caused by interfacial resistance is an important factor influencing the reaction advancement at the interface and the formation of intermetallics [83].

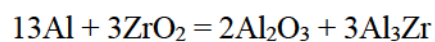
In an investigation of the formation of aluminum matrix composites reinforced with carbon fibers during SPS, Lalet et al. [84] found that the pulse parameters (the number of ON and OFF periods) influenced the interfacial reactions. With the ON/OFF pulsing ratio set at 24:1, a microstructure similar to that of composites obtained by hot pressing was observed. Under 12:2, 6:4 and 3:3 pulsing patterns, the oxide layer present on aluminum fractured and Al<sub>4</sub>C<sub>3</sub> formed at the Al/carbon fiber interface. The direct contact between aluminum and the fiber was explained by the infiltration of molten aluminum through the disrupted Al<sub>2</sub>O<sub>3</sub> layer. When the pulsing pattern was such that the ON time was short, a high current had to be applied: at 4:6 pulsing pattern, molten aluminum leaked out of the die, although the temperature measured by the thermocouple (in direct contact with the sample) at that moment was only 500 °C. Therefore, the application of a high current led to significant overheating and caused melting of aluminum. The observed crystallization of the initially amorphous Al<sub>2</sub>O<sub>3</sub> layer was also put forward as the evidence of high local temperatures.

#### *Aluminum and magnesium matrix composites obtained by microwave sintering*

In composites sintered by microwaves, the presence of a component absorbing microwaves plays an important role in the microstructure formation. The influence of susceptor contained in the composite mixture is discussed below using examples elaborated in refs. [67, 85, 86, 87]. Silicon carbide SiC is often used as a reinforcement in MMCs sintered by microwaves. Thakur et al. [85] used two-directional (hybrid) microwave sintering to fabricate Al/Ti metastable composites and Al/(Ti + SiC) composites. SiC particles contained in the powder mixtures contributed to the heating process of the compact by acting as a microwave absorbent. CNTs

added to a Mg alloy played a dual role of mechanical reinforcement and microwave susceptor [67]. It was found that the interaction between an Al matrix and WC reinforcing particles occurred at lower sample temperatures in microwave sintered composites than in conventionally sintered composites [86]. Microwave heating caused a higher temperature at the interface of Al and WC particles than the macroscopic sample temperature due to superior absorption of microwaves by the WC particles. The formation of interfaces in Al/(Ti,W)C composites prepared by microwave sintering was studied by Zheng et al. [87]. Microwave sintering at 580 °C, which is close to the solidus temperature of Al 6061 alloy, led to the formation of compacts with altered shape because of bubbling and melt squeezing out of the compact. The evidence of local melting of the matrix material was also found in the microstructure of the sample sintered at a lower temperature (560 °C): the corners of the (Ti,W)C particles in the sintered material were not as acute as in the raw powder. It was suggested that the presence of a liquid phase between Al and (Ti,W)C was caused by a higher absorbing ability of (Ti,W)C in comparison with the Al alloy and an additional heat transfer to the alloy matrix surrounding the ceramic particles. The ability of the (Ti,W)C particles to absorb the microwave energy could be enhanced by their geometrical characteristics – a small size and the presence of sharp surface features.

Treatment of powders by microwaves can also be used for realizing the combustion synthesis of MMCs. In-situ Al<sub>2</sub>O<sub>3</sub>- and Al<sub>3</sub>Zr-reinforced aluminum matrix composites were obtained by the SiC-assisted combustion synthesis [88]. The reaction mixture was composed of Al and ZrO<sub>2</sub> powders. Aluminum was taken in excess to form both the matrix and the reinforcing phases. The synthesis of the composite occurred via the following reaction:



The heating profiles of Al+ZrO<sub>2</sub> powder mixtures in a SiC susceptor-assisted microwave combustion synthesis process are shown in Figure 12. The reaction mixture with stoichiometry to fully convert aluminum into the ceramic phases (mixture C) shows a very fast temperature rise corresponding to the combustion reaction. In mixtures A and B, due to excess of aluminum, the

temperatures developed during the synthesis were lower. The microstructure of the composites was found to be finer than that of the composite prepared by annealing of the cold-pressed pellets in a vacuum furnace at 1100 °C for 10 min (Figure 13). In the microstructure of the composites, the dark background is the Al matrix, rectangular gray particles are the  $\text{Al}_3\text{Zr}$  reinforcement, fine particles represent the  $\text{Al}_2\text{O}_3$  phase.

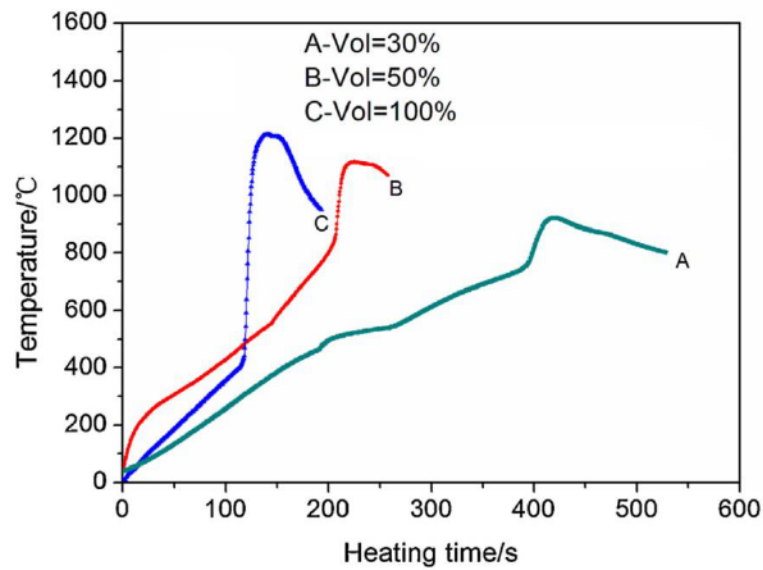


**Figure 12.** Heating profiles of Al+ZrO<sub>2</sub> powder mixtures in a SiC susceptor-assisted microwave combustion synthesis process, the compositions of the mixtures correspond to the formation of 30 vol.% (A), 50 vol% (B) and 100 vol.% (C) of  $\text{Al}_2\text{O}_3 + \text{Al}_3\text{Zr}$  reinforcing phases. Reprinted from [88], Copyright (2015), with permission from Elsevier.

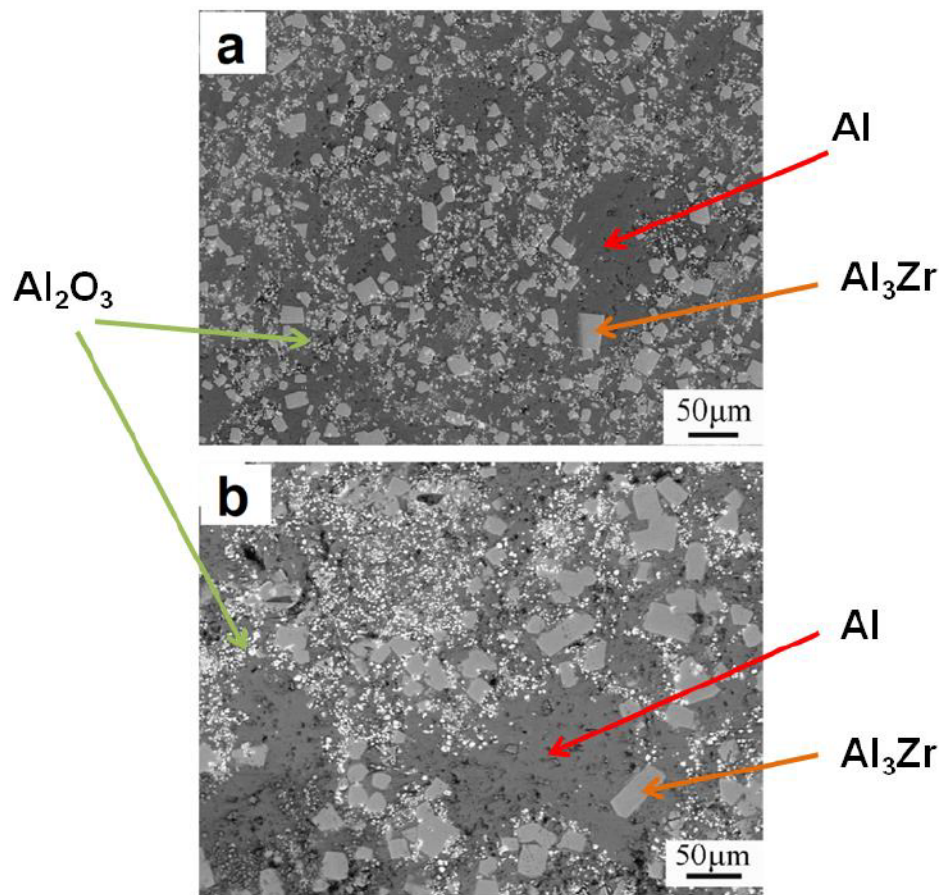


**Figure 13.** Microstructure of Al-Al<sub>2</sub>O<sub>3</sub>-Al<sub>3</sub>Zr composites obtained by microwave combustion synthesis (a) and synthesis via vacuum furnace annealing (b). Dark back ground – Al matrix, rectangular gray particles – Al<sub>3</sub>Zr, fine particles – Al<sub>2</sub>O<sub>3</sub>. Reprinted from [88], Copyright (2015), with permission from Elsevier.

#### *Aluminum and magnesium matrix composites obtained by induction sintering*

As induction sintering is not used as widely as SPS for the production of composites, the available structural and mechanical property data are rather limited. Induction heating has been successfully used for producing metallic glass-reinforced composites with Mg and Al alloy matrices [62, 89, 90, 91]. Al alloy 520.0 strengthened by Cu<sub>54</sub>Zr<sub>36</sub>Ti<sub>10</sub> metallic glass particles was obtained in ref. [89]. The composite was sintered by induction heating in a steel die under a pressure of 50 MPa at the maximum temperature of 720 K (which is within the supercooled liquid region of the Cu<sub>54</sub>Zr<sub>36</sub>Ti<sub>10</sub> metallic glass) with a holding time at this temperature of 2 min. Although the pressure during sintering was not very high and the holding time was very short,

the composite was fully dense. The material featured a uniform distribution of the glassy reinforcement particles in the matrix.

### **Comparative investigations: the influence of the sintering method on the phase composition and microstructure of the composites**

The heating method of the powder mixtures was shown to influence the phase composition of the sintered material. The Al-VC mixtures were sintered by conventional and microwave sintering; the maximum temperature was the same in those experiments (600 °C) [92]. In the microwave sintering experiment, the sample was not soaked at the maximum temperature. Under conventional sintering conditions, the mixture was held for 1 h at the maximum temperature.  $VAI_3$  intermetallic formed only in the microwave sintered material and was as a result of the interaction between VC and Al. No reaction between the phases was observed in the conventionally sintered material. The authors explained enhanced reactivity of VC towards Al under microwave treatment by high local temperatures developing due to microwave absorption by the vanadium carbide particles. Under conventional sintering, no physical basis was present for increasing the temperature in the vicinity of VC particles. In ref. [93], Al alloy matrix composites containing hybrid reinforcements were developed by microwave and conventional sintering. The microwave process was proved to be more efficient for achieving densification (at the same measured temperature) leading to increased hardness and bending strength despite a 1-h soaking in conventional sintering and the absence of any soaking in the microwave process. Higher densities demonstrated by the microwave sintered samples in comparison with conventionally sintered samples were explained by the volumetric nature of heating in the microwave sintering method. The SiC and TiC particles' acted as good absorbers of microwave energy.

Joseph et al. [94] compared the mechanical behavior of Al matrix composites containing  $\omega$ -Al-Cu-Fe particles consolidated from a mixture of metallic aluminum and i-Al-Cu-Fe

(icosahedral quasicrystalline phase) by hot isostatic pressing and SPS. In composites produced by both methods, copper diffused into the Al matrix leading to the formation of semi-coherent precipitates of  $\theta'$ -Al<sub>2</sub>Cu in the case of SPS and incoherent  $\theta$ -Al<sub>2</sub>Cu in the case of hot isostatic pressing. The difference in the structural characteristics of the precipitates was due to a much shorter holding time in the case of SPS. Composites synthesized by the SPS showed a higher proof stress  $\sigma_{0.2\%}$  than those obtained by hot isostatic pressing when measured at temperatures below 473 K. An important message of that research is the possibility of the formation of new reinforcing phases of controlled crystalline structure upon the interaction of the matrix metal with the introduced additives.

### **Mechanical properties of aluminum and magnesium matrix composites obtained by field-assisted sintering**

Tables 1–4 summarize the mechanical properties (in compression and tension) of Al-based alloys, Mg-based alloys, Al/Al alloy matrix composites and Mg/Mg alloy matrix composites obtained by SPS, microwave and induction sintering extracted from the recent literature. Some alloys and composites obtained with the use of field-assisted sintering possess attractive combinations of strength and ductility and can compete with classical cast and wrought materials.

For MMCs obtained by field-assisted sintering, due to the use of high heating rates, grain growth can be controlled such that both the matrix and the reinforcing particles can retain their fine sizes. The following strengthening mechanisms should be considered in MMCs obtained by field-assisted sintering: dispersion strengthening, grain boundary strengthening, dislocation strengthening, solution strengthening, and load transfer. Knowing the relative contribution of each mechanism can be useful for the property tailoring of the composites in the required direction.

Grain refinement is a very effective route to improve the strength according to Hall-Petch equation [95]:

$$\sigma_y = \sigma_0 + kd^{-1/2}, \quad (4)$$

where  $\sigma_y$  is the yield stress of the material with grain size  $d$ ,  $\sigma_0$  is the yield stress of the material with coarse grains and  $k$  is a chemistry- and microstructure-dependent constant.

The contribution of Orowan strengthening  $\Delta\sigma_{Or}$  can be calculated as follows [96]:

$$\Delta\sigma_{Or} = \varphi \frac{Gb}{L} \quad (5)$$

where  $\varphi$  is a constant equal to 2,  $G$  is the shear modulus of the matrix,  $b$  is the Burgers vector of the matrix and  $L$  is the inter-particle distance.

Solution strengthening can be significant in materials processed by field-assisted sintering, unless conditions of sintering are intentionally selected such that the time and temperature favor decomposition of solid solutions and the formation of precipitates [97], metastable or stable (purposeful ageing of alloys during the field-assisted sintering/treatment).

Dislocation strengthening of the matrix is determined by Taylor formula [95]:

$$\sigma = \sigma_0 + \alpha Gb\rho^{1/2}, \quad (6)$$

where  $\sigma$  is the flow stress,  $G$  is the shear modulus,  $b$  is the Burgers vector,  $\rho$  is the dislocation density and  $\alpha$  is a material-dependent constant between 0.5 and 1 [96]. In MMCs containing ceramic reinforcements, dislocations can originate from the CTE mismatch. In addition, in the case of fast consolidation of powders at relatively low temperatures, the matrix metals can retain dislocations accumulated during the powder processing stage (for example, during mechanical milling).

Similar to MMCs obtained by conventional methods, composites fabricated by field-assisted sintering can be strengthened by load transfer. As composites produced by sintering are mostly discontinuously reinforced composites, a model elaborated by Nardone and Prewo [98] can be applied. In their work, it was shown that the shear lag theory can be used to describe the strength of discontinuous SiC-reinforced aluminum matrix composites provided that the theory is modified to take into account the tensile transfer of load from the matrix to the discontinuous reinforcement. According to the model, the yield strength of a composite reinforced with whiskers or particles can be calculated as follows:

$$\sigma_{cy} = \sigma_{my} \left[ \frac{1}{2} V_r (s + 2) + V_m \right] \quad (7)$$

where  $\sigma_{cy}$  is the yield strength of the composite,  $\sigma_{my}$  is the yield strength of the unreinforced matrix,  $s$  is the aspect ratio of the reinforcement,  $s = L_r/d_r$  ( $L_r$  is the length and  $d_r$  is the diameter of the reinforcement),  $V_r$  and  $V_m$  is the volume fraction of the reinforcement and the matrix, respectively. This equation predicts a strengthening effect for reinforcement with an aspect ratio  $s = 1$ .

Interestingly, during SPS of particle-reinforced agglomerates obtained by mechanical milling, microstructures analogous to trimodal composites (as described in ref. [99]) can be obtained. The concept of trimodal architectures is based on combining three components: nano/ultrafine matrix grains, coarse matrix grains, and ceramic reinforcements. Such a combination gives opportunities for flexible engineering of the composites' microstructures and mechanical properties. The role of coarse grains is to improve the ductility of otherwise brittle materials. Yang et al. [99] obtained the trimodal composites by adding a coarse-grained unreinforced matrix particles to the cryomilled B<sub>4</sub>C-Al alloy mixture and subjecting the powder to hot isostatic pressing followed by hot extrusion. A possibility of local temperature differences leading to the formation of bimodal microstructures in metallic materials processed by SPS was considered in refs. [43, 100, 101]. If a ceramic particle-reinforced powder material is subjected

to SPS under such conditions that high-temperature regions develop locally, melting of a fraction of the matrix can take place, leading to a certain redistribution of the phases and the formation of particle-depleted areas of the matrix. Those particle-depleted areas could play a role similar to that played by the coarse matrix grains in trimodal composites.

The bimodal grain structures were found to exhibit deformation and fracture behavior similar to ductile phase-toughened brittle materials [102]. The ductile coarse grains in the ultrafine-grained matrix impede the propagation of microcracks, which leads to enhanced ductility and toughness. Bimodal-structure alloys can be obtained by field-assisted sintering methods. In the case of SPS, the bimodal structure can be obtained either by consolidating a mixture of powders with different particle sizes [103] or “in-situ” by employing the effect of local overheating.

A common problem of materials produced by powder sintering, including field-assisted sintering, is residual porosity, which can vary from fractions of a percent to several percent, unless rendered intentionally high for specific applications. Therefore, the influence of porosity on strength should be taken into account. According to Balshin [104], the tensile strength of a porous metal-ceramic material can be expressed as

$$\sigma_p = \sigma_0 \theta^m, \quad (8)$$

where  $\sigma_p$  is the strength of a material with residual porosity,  $\theta$  is the relative density,  $\sigma_0$  is the strength of the zero-porosity material and  $m$  is a constant.

**Table 1.** Sintering conditions and mechanical properties in compression of aluminum matrix composites and aluminum alloys obtained by field-assisted sintering

| Composition                                                                               | Sintering conditions                          | Strain rate, s <sup>-1</sup> | Yield strength/0.2% proof stress, MPa | Ultimate strength, MPa | Deformation at fracture, % | Reference |
|-------------------------------------------------------------------------------------------|-----------------------------------------------|------------------------------|---------------------------------------|------------------------|----------------------------|-----------|
| Al-Zn-Mg                                                                                  | SPS, 500 °C, 10 min, 80 MPa                   | 10 <sup>-3</sup>             | 610                                   | 736                    | -                          | [97]      |
| Al 2024                                                                                   | SPS, 500 °C, 10 min, 50 MPa                   | 3.7 · 10 <sup>-4</sup>       | 256                                   | -                      | -                          | [105]     |
| Al 2024+2 wt.% TiN                                                                        | SPS, 500 °C, 10 min, 50 MPa                   | 3.7 · 10 <sup>-4</sup>       | 730                                   | 831                    | 10                         | [105]     |
| Al+ ω-Al-Cu-Fe + θ'-Al <sub>2</sub> Cu                                                    | SPS, 550 °C, 2 min, 100 MPa                   | 1.4 · 10 <sup>-4</sup>       | 500                                   | -                      | 4                          | [94]      |
| Al 6082 + ω-Al-Cu-Fe + Al <sub>2</sub> Cu + i-Al-Cu-Fe + Al <sub>13</sub> Fe <sub>4</sub> | SPS, 550 °C, 5 min, 50 MPa                    | 1.8 · 10 <sup>-3</sup>       | 519                                   | 639                    | 24                         | [106]     |
| Al (air atomized, ball-milled with stearic acid)                                          | SPS, 600 °C, 1 h, 49 MPa                      | -                            | 440                                   | -                      | -                          | [107]     |
| Al (air atomized)                                                                         | SPS, 600 °C, 1 h, 49 MPa                      | -                            | 173                                   | -                      | -                          | [107]     |
| Al+5 vol% AlCoCrFeNi high-entropy alloy                                                   | SPS, 580 °C, 10 min, 6 MPa                    | 10 <sup>-3</sup>             | 137                                   | -                      | 50                         | [81]      |
| Al 7075 + 15 vol.% Zr <sub>65</sub> Cu <sub>18</sub> Ni <sub>7</sub> Al <sub>10</sub>     | Cold compaction; SPS, 300 °C, 10 min, 600 MPa | 5 · 10 <sup>-4</sup>         | 366                                   | 471                    | 25                         | [77]      |
| Al + 40 vol. % Al <sub>65</sub> Cu <sub>16.5</sub> Ti <sub>18.5</sub>                     | SPS, 500 °C, 400 MPa                          | 10 <sup>-3</sup>             | 1100                                  | 1710                   | 4.3                        | [108]     |
| Al + 5 at.% Fe                                                                            | SPS, 480 °C, 5 min, ~100 MPa or greater       | 10 <sup>-3</sup>             | 992                                   | 1045                   | 30                         | [109]     |

|                                                                                                                                            |                                                                                       |                     |     |      |      |       |
|--------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------|---------------------|-----|------|------|-------|
|                                                                                                                                            |                                                                                       |                     |     |      |      |       |
| Al 7075+15vol.%<br>Ti <sub>48</sub> Zr <sub>7.5</sub> Cu <sub>39</sub> Fe <sub>2.5</sub> Sn <sub>2</sub> Si <sub>1</sub>                   | Cold compaction; SPS,<br>300 °C, 10 min, 600 MPa                                      | $5 \cdot 10^{-4}$   | 950 | 1002 | 4    | [110] |
| Al+21vol.%(TiAl <sub>3</sub> +Al <sub>2</sub> O <sub>3</sub> )                                                                             | Accumulative roll<br>bonding, SPS.<br>550 °C, 102 MPa, 5 min;<br>700 °C, 4 MPa, 5 min | -                   | -   | 630  | 20   | [111] |
| Al+25 vol. %Ni <sub>60</sub> Nb <sub>40</sub>                                                                                              | Microwave sintering, 550<br>°C; hot extrusion, 350 °C                                 | $8.3 \cdot 10^{-4}$ | 155 | 375  | >50  | [112] |
| Al                                                                                                                                         | Microwave sintering, 550<br>°C; hot extrusion, 350 °C                                 | $8.3 \cdot 10^{-4}$ | 80  | 245  | >50  | [112] |
| Al 6061 + 30 wt.% Ti(W,C)                                                                                                                  | Microwave sintering, 560<br>°C, 45 min                                                | $3.6 \cdot 10^{-4}$ | 236 | 474  | 33.5 | [87]  |
| Al 6061 + 10 wt.% Ti(W,C)                                                                                                                  | Microwave sintering, 560<br>°C, 45 min                                                | $3.6 \cdot 10^{-4}$ | 118 | 346  | 39.6 | [87]  |
| Al 6061                                                                                                                                    | Microwave sintering, 560<br>°C, 45 min                                                | $3.6 \cdot 10^{-4}$ | 99  | -    | -    | [87]  |
| Al                                                                                                                                         | Microwave sintering, 550<br>°C; annealing 400 °C, 1 h;<br>hot extrusion, 350 °C       | $8.3 \cdot 10^{-4}$ | -   | 308  | 9.5  | [113] |
| 15 vol.% (Al-Li-Cu) + Al                                                                                                                   | Microwave sintering, 550<br>°C; annealing 400 °C, 1 h;<br>hot extrusion, 350 °C       | $8.3 \cdot 10^{-4}$ | -   | 453  | 6.4  | [113] |
| Al 6061 +15 vol.%<br>[(Fe <sub>1/2</sub> Co <sub>1/2</sub> ) <sub>75</sub> B <sub>20</sub> Si <sub>5</sub> ] <sub>96</sub> Nb <sub>4</sub> | Induction sintering, 555<br>°C, 2 min, 70 MPa                                         | $10^{-3}$           | 570 | 600  | 13   | [90]  |
| Al 520.0 + 15 vol.% Cu <sub>54</sub> Zr <sub>36</sub> Ti <sub>10</sub>                                                                     | Induction sintering, 447<br>°C, 2 min, 50 MPa                                         | $10^{-3}$           | 580 | 840  | 14   | [89]  |
| Al 2024 + 15 wt.%<br>Fe <sub>73</sub> Nb <sub>5</sub> Ge <sub>2</sub> P <sub>10</sub> C <sub>6</sub> B <sub>4</sub> metallic glass         | Induction sintering, 550<br>°C, 30 min, 400 MPa                                       | $10^{-3}$           | 403 | 660  | 12   | [91]  |

**Table 2.** Sintering conditions and mechanical properties in tension of aluminum matrix composites and aluminum alloys obtained by field-assisted sintering.

| Composition                                                                        | Sintering conditions                                                    | Strain rate, $s^{-1}$ | Yield strength/<br>0.2% proof stress, MPa | Ultimate strength, MPa | Deformation at fracture, % | Reference |
|------------------------------------------------------------------------------------|-------------------------------------------------------------------------|-----------------------|-------------------------------------------|------------------------|----------------------------|-----------|
| Al+1 vol.% CNT                                                                     | SPS, 630 °C, 30 min, 30 MPa; hot extrusion, 500 °C                      | $5 \cdot 10^{-4}$     | 279                                       | 306                    | 12                         | [75]      |
| Al+A <sub>4</sub> C <sub>3</sub> /graphene (from 0.42 wt.%graphene oxide+Al)       | SPS, 610 °C, 20 min, 50 MPa; hot extrusion                              | $1.67 \cdot 10^{-3}$  | 149                                       | 178                    | 19                         | [114]     |
| Al-4Cu + 1 wt.% reduced graphene oxide                                             | SPS, 500 °C, 10 min, 50 MPa;                                            | $3.5 \cdot 10^{-4}$   | 139                                       | 320                    | 10                         | [115]     |
| Al+4.5wt.%BN (AlN and AlB <sub>2</sub> formed in situ during ball milling and SPS) | SPS, 600 °C, 60 min, 50 MPa                                             | -                     | 250                                       | 386                    | 5                          | [116]     |
| Al (ball-milled)                                                                   | SPS, 600 °C, 60 min, 50 MPa                                             | -                     | 160                                       | 167                    | -                          | [116]     |
| Al+1wt.%BN nanosheets                                                              | SPS, 600 °C, 60 min, 50 MPa                                             | -                     | -                                         | 152                    | 6.5                        | [117]     |
| Al+4wt.%Sc                                                                         | SPS, 550 °C, 2 min, 50 MPa + T6 treatment                               | -                     | 197                                       | 226                    | 11                         | [118]     |
| Al+0.75vol.%CNT                                                                    | SPS, 630 °C, 30 min, 30 MPa; hot rolling, 550 °C                        | $2.1 \cdot 10^{-3}$   | 200                                       | 220                    | 21                         | [119]     |
| Al 2024                                                                            | SPS, 500 °C, 10 min, 50 MPa; thermo-mechanical treatment                | $5 \cdot 10^{-4}$     | 530                                       | 583                    | 16.2                       | [3]       |
| Al 6061+30 vol.% Si <sub>3</sub> N <sub>4</sub>                                    | SPS, 570 °C, 15 min, 50 MPa; heat treatment (solution treatment, aging) | -                     | 400                                       | 499                    | 1.5                        | [120]     |
| Al+25 vol. %Ni <sub>60</sub> Nb <sub>40</sub>                                      | Microwave sintering, 550 °C; hot extrusion, 350 °C                      | $8.3 \cdot 10^{-4}$   | 102                                       | 120                    | 9.5                        | [112]     |

|                          |                                                                           |                     |     |     |      |       |
|--------------------------|---------------------------------------------------------------------------|---------------------|-----|-----|------|-------|
| Al                       | Microwave sintering, 550 °C; hot extrusion, 350 °C                        | $8.3 \cdot 10^{-4}$ | 65  | 75  | 25.8 | [112] |
| Al+5 wt.% Ti             | Microwave sintering; hot extrusion, 350 °C                                | -                   | 103 | 143 | 12.0 | [85]  |
| Al                       | Microwave sintering, 550 °C; annealing 400 °C, 1 h; hot extrusion, 350 °C | $8.3 \cdot 10^{-4}$ | 105 | 119 | 12.5 | [113] |
| 15 vol.% (Al-Li-Cu) + Al | Microwave sintering, 550 °C; annealing 400 °C, 1 h; hot extrusion, 350 °C | $8.3 \cdot 10^{-4}$ | 138 | 167 | 6.4  | [113] |

**Table 3.** Sintering conditions and mechanical properties in compression of magnesium matrix composites and magnesium alloys obtained by field-assisted sintering.

| Composition                                                                                    | Sintering conditions                          | Strain rate,<br>$s^{-1}$ | Yield<br>strength/0.2%<br>proof stress, MPa | Ultimate<br>strength,<br>MPa | Deformation<br>at fracture,<br>% | Reference |
|------------------------------------------------------------------------------------------------|-----------------------------------------------|--------------------------|---------------------------------------------|------------------------------|----------------------------------|-----------|
| AZ91                                                                                           | SPS, 360 °C, 1 h                              | $1.1 \cdot 10^{-3}$      | 185                                         | 341                          | 15.2                             | [121]     |
| AZ91                                                                                           | SPS, 360 °C, 2 h; 200 °C, 2 h                 | $1.1 \cdot 10^{-3}$      | 217                                         | 374                          | 13.9                             | [121]     |
| Mg + 1Al + 0.15CNT                                                                             | SPS, 560 °C, 5 min;<br>hot extrusion, 400 °C  | $10^{-3}$                | 118                                         | 321                          | 17.9                             | [122]     |
| Mg-Gd-Zn-Zr (GZ100K)                                                                           | SPS, 400 °C, 10 min,<br>50 MPa                | $10^{-3}$                | 210                                         | 386                          | 20                               | [123]     |
| Mg + 2vol.%SiC                                                                                 | SPS, 450 °C, 5 min, 50 MPa                    | $10^{-4}$                | -                                           | 247                          | 9                                | [124]     |
| AZ80                                                                                           | SPS, 350 °C, 3 min,<br>100 MPa                | $2.5 \cdot 10^{-4}$      | 442                                         | 546                          | 4.2                              | [101]     |
| Mg AZ31                                                                                        | SPS, 400 °C, 5 min, 80 MPa                    | $2.5 \cdot 10^{-4}$      | 400                                         | 500                          | 3.6                              | [125]     |
| Mg                                                                                             | SPS, 400 °C, 5 min, 80 MPa                    | $2.5 \cdot 10^{-4}$      | 400                                         | 500                          | 3.6                              | [126]     |
| Mg + Mg-Zn alloy                                                                               | SPS, 400 °C, 5 min, 80 MPa                    | $2.5 \cdot 10^{-4}$      | 408                                         | 506                          | 6.6                              | [126]     |
| AZ91 + Zr <sub>57</sub> Nb <sub>5</sub> Cu <sub>15.4</sub> Ni <sub>12.6</sub> Al <sub>10</sub> | Induction sintering,<br>440 °C, 2 min, 50 MPa | -                        | 325                                         | 542                          | 11                               | [62]      |
| AZ61+3 vol. % CNT                                                                              | Microwave sintering,<br>500 °C, 8 min         | -                        | 117                                         | 218                          | 10.2                             | [67]      |

**Table 4.** Sintering conditions and mechanical properties in tension of magnesium matrix composites and magnesium alloys obtained by field-assisted sintering.

| Composition                             | Sintering conditions                                       | Strain rate, $s^{-1}$ | Yield strength/0.2% proof stress, MPa | Ultimate strength, MPa | Deformation at fracture, % | Reference |
|-----------------------------------------|------------------------------------------------------------|-----------------------|---------------------------------------|------------------------|----------------------------|-----------|
| Mg                                      | SPS, 585 °C, 5 min, 60 MPa                                 | $2.8 \cdot 10^{-3}$   | -                                     | 120                    | -                          | [23]      |
| Mg + 10wt.%SiC                          | SPS, 585 °C, 5 min, 60 MPa                                 | $2.8 \cdot 10^{-3}$   | -                                     | 140                    | -                          | [23]      |
| Mg + 1Al + 0.15CNT                      | SPS, 560 °C, 5 min; hot extrusion, 400 °C                  | $10^{-3}$             | 157                                   | 271                    | 8.8                        | [122]     |
| Mg + 3wt.%Ti ball-milled                | SPS, 600 °C, 30 min, 30 MPa; hot extrusion, 400 °C         | $5 \cdot 10^{-4}$     | 184                                   | 224                    | 14.9                       | [127]     |
| Mg                                      | Microwave sintering, 640 °C, 25 min; hot extrusion, 350 °C | $1.6 \cdot 10^{-4}$   | 116                                   | 168                    | 6.1                        | [128]     |
| Mg + 2wt.%Y <sub>2</sub> O <sub>3</sub> | Microwave sintering, 640 °C; hot extrusion, 350 °C         | $1.7 \cdot 10^{-4}$   | 157                                   | 244                    | 9.1                        | [129]     |

## **Functional properties of selected aluminum and magnesium matrix composites obtained by field-assisted sintering**

### *Thermal and electrical conductivity of aluminum matrix composites*

Al-diamond composites are of interest from the viewpoint of developing materials with high thermal conductivity [130, 131]. Tan et al. [132] addressed the issue of the nature of the aluminum-diamond interface and distinguished between diffusion-bonded and reaction-bonded interfaces. The diffusion-bonded interface was thought to be preferable for achieving a low thermal resistance. A low thermal conductivity of some composites produced by SPS was explained by the temperature gradients within the sample. Diamond particles located in the high-temperature regions were more likely to develop the reaction-type interfaces (with the formation of  $\text{Al}_4\text{C}_3$  as the reaction product).

Aluminum matrix composites containing silicon particles are attractive packaging materials for the microelectronic applications, as their CTE can be reduced relative to pure aluminum, their thermal conductivity remaining high. By SPS at 510 °C, nearly fully dense Si-Al composites were obtained [133]. A composite containing 45 vol.% Si showed a good combination of properties: an average CTE of  $12.7 \cdot 10^{-6} \text{ K}^{-1}$  and a thermal conductivity of  $113 \text{ W m}^{-1} \text{ K}^{-1}$ . The obtained value of CTE was lower than that of the counterpart materials. The benefit of SPS was in keeping the grains of silicon small during consolidation. A high surface area of silicon particles allowed restraining expansion of the Al phase lowering the CTE of the composite.

It is a serious challenge to fabricate high-strength Al matrix composites without sacrificing their electrical conductivity. It was shown that Al matrix composites can retain a high electrical conductivity (at the level of unreinforced aluminum) and become stronger than pure aluminum when few-layer graphene joined to the Al matrix by  $\text{Al}_4\text{C}_3$  nanorods acts as a reinforcement [114]. The electrical conductivity of the composite having a yield strength of 149 MPa and an ultimate tensile strength of 178 MPa was 59.8% of the International Annealed

Copper Standard (%IACS). The synthesized material was, therefore, recommended as a high-performance conductor.

#### *Biocompatibility and biodegradability of magnesium matrix composites*

Magnesium-based composites are suitable for biomedical applications. As microstructural refinement is beneficial for magnesium alloys designed for temporary implants, SPS is considered as a processing method for these alloys. Currently, SPS of magnesium matrix composites and Mg-based alloys is studied with the prospects of developing biodegradable materials [134, 135, 136, 137, 138, 139, 140].

Microwave sintering was used for making porous Mg-based composites as candidate biodegradable materials [141]. A  $\text{Si}_3\text{N}_4$  powder or an  $\text{Al}_2\text{O}_3$  whisker additive was introduced into the Mg matrix to modify its electrochemical performance. As  $\text{Si}_3\text{N}_4$  absorbs microwaves, higher local temperatures caused explosive evaporation and formation of larger pores compared with composites containing  $\text{Al}_2\text{O}_3$ .

#### **Scale-up of the field-assisted sintering processes**

The key problem of the scale-up of the sintering processes is to ensure the same temperature in the laboratory-size and industrial-size sintered parts. The temperature distribution in specimens differing in electrical conductivity has been addressed in ref. [142] for the SPS process in a die. It was shown that, for conductive materials, the temperature in the center of a disk-shape specimen is higher than in the rim areas. The predictive capability of modeling approaches is of great importance for the industrial implementation of SPS. The scalability of SPS was analyzed experimentally and theoretically by Olevsky et al. [143, 144]. In those studies, scalability experiments on the SPS of similarly shaped alumina specimens of the four different sizes have been conducted. The processed specimens have been characterized in terms of relative density and grain-pore structure. Overall, SPS showed good scalability potential. At the same

time, it was shown that, due to the demonstrated possibility of a significant size impact in case of high heating rates and large specimen sizes, the predictive capability of reliable modeling approaches is of great importance for the industrial implementation of the SPS technique.

In the area of metallic materials, Tokita [145] reported the commercial use of metallic sputtering targets 99–100% dense, 5–20 mm thick and 300–350 mm in diameter obtained by SPS. An advanced SPS approach enabling the scale-up fabrication of large size complex shapes and an energy efficient processing has been described in ref. [146]. A unique configuration using deformable and electrically insulated interfaces has been employed to constrain the electric current path for a significant reduction of the electric current required to heat the 40 mm specimen while imposing a gear shape onto the processed parts. A comprehensive electrothermal-mechanical simulation of the SPS process revealed that the high thermal stability observed for this SPS approach originates from different factors: the high thermal conductivity of the metal powder and the thermal confinement of this metal powder via the lateral thermal contact resistance and via the upper and lower alumina powder which has a very low thermal conductivity. That case study demonstrated the high scalability potential of the SPS technology which can combine the benefits of advanced material properties (through high pressures, high heating rates) and complex shapes. It is theoretically possible to place any number of complex shape interfaces in a large-dimension tooling set (like in a conventional furnace), improving drastically the productivity and, in turn, the scalability of the SPS technology. Such an improvement would require perfect control of the temperatures and a homogeneous displacement field of the powder.

Comparative studies of SPS and hot pressing were reported by Musa et al. [147] for Ti–38 vol.%Al<sub>2</sub>O<sub>3</sub>–11.5 vol.%TiC composites: only 5 min was needed to sinter the material by SPS and about 5 h was needed to do the same using HP. In both cases, relative densities greater than 99.9% were reached. The times given are a sum of the duration of the heating stage and the dwell time at the maximum temperature. The total energies required for the SPS and hot pressing

runs were compared to reveal that the SPS technology allowed for energy saving of 90–95%. Notably, the use of graphite felt around the die to reduce heat losses by radiation was found to lead to energy savings of 25% under the selected sintering conditions.

As Kelly & Graeve pointed out [148], high-throughput SPS systems will have a serious impact on mass production. It was emphasized that lessons learnt during the development of the hot pressing technology can be directly applied to SPS to minimize the associated risks. An ideal SPS design for manufacturing would be a fully automated process, from loading and handling of the die to sintering, cooling, and sample removal from the die. The key difference between hot pressing and SPS is the method of heating. When SPS is used instead of hot pressing, the processing times are significantly reduced; some processes require lower temperatures. The reduced times and temperatures in the SPS processes lead to a reduced energy consumption (and energy cost) per sample. As within a given time, SPS will produce a larger number of parts than hot pressing, the sintering cost (which includes the operation of the cooling system, consumables and burden costs and is taken to be the same for SPS and hot pressing in ref. [148]) per sample will be lower for the SPS technology than for hot pressing. A higher productivity and a better expandability of SPS relative to hot pressing have also been highlighted in ref. [149].

The scalability of microwave sintering is often difficult to achieve [36]. The results obtained by Manière et al. [150, 151] provide a basis for explaining the inherent instability and non-reproducibility of the experiments frequently reported in the literature for microwave sintering. The process instabilities stem from the intrinsic non-homogeneous electro-magnetic fields repartition and their sensitivity to the presence of dissipative materials, such as aluminum and magnesium matrix composites, during microwave sintering [39, 113, 128]. The direct microwave heating configuration, for large scale specimens, results in the formation of a hot spot at the center of the sample. The hot spot phenomenon increases drastically in the beginning of the process and then stabilizes when the densification occurs resulting, at the end of the heating cycle, in a significant temperature difference across the sample's volume. The microwave field

penetration and then the heating become more superficial when the densification happens; this fact explains the temperature gradients' stabilization. The sample shape is highly deformed during the densification due to the hot spot formation but tends to go back to the cylindrical shape at the end of the densification. A decrease in the sample size appears to reduce the thermal gradients and, at the same time, it resolves the problem of densification heterogeneities.

Sintering of small samples is, therefore, more stable. The temperature inhomogeneity can be balanced by the use of susceptors ("hybrid microwave heating"), which can add an external heat flux uniformizing the temperature inside the sample [150, 151]. The hybrid heating configuration succeeds in reducing the temperature gradients in the sample, allowing a more uniform and scalable overall densification of the large samples.

Induction sintering provides fast heating and cooling, dramatically shortens the sintering time and ensures high energy efficiency. The scalability of the induction sintering process can be achieved if the cross-sections of the coil and the sintered part are similar with small gaps between them and if undesirable thermal gradients are avoided by selecting optimal heating rates and penetration depths [18].

## **Summary**

The overview of the literature on the processing, microstructure and properties of Al- and Mg-based materials processed by field-assisted sintering shows that composites and alloys with high strength and high ductility can be obtained by powder metallurgy through the flexible compositional and microstructural design and a proper choice of the field-assisted sintering parameters. Traditional MMC compositions (metal matrix composites reinforced with ceramic particles) as well as composites reinforced with particles of alloys (metallic glass, high-entropy alloys, derivatives of quasicrystals) are currently of interest. MMCs with metallic glass reinforcements present a rapidly developing research direction. The very possibility of consolidating the metal matrix–metallic glass mixtures while preserving the amorphous state of

the reinforcement depends on the sintering method. SPS and microwave sintering have been successfully used for the production of functional Al- and Mg-based materials (materials with high thermal and electrical conductivity, biodegradable materials). Processes of local character (overheating, melting, infiltration, chemical reactions) play significant roles in the microstructure formation and property variation of the composites obtained by SPS and microwave sintering. The scale of locality is determined by either the initial particle size or the distance between inclusions in a matrix. The progress in the microstructural and compositional design of the composites made over recent years is a solid basis for the development of commercial products.

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## **Progress in aluminium and magnesium matrix composites obtained by spark plasma, microwave and induction sintering**

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