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Processing, microstructure, properties, and applications of $MoSi_2$-containing composites: a review

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Intermetallic molybdenum disilicide ($MoSi_2$) possesses unique physical, chemical, thermal, and mechanical properties that make it compatible with some ceramics (SiC, Al_2O_3) and metals (Cu, Al) to manufacture composite materials. Its current applications, chiefly limited to heating elements, can be expanded if its properties are judiciously combined with those of other materials like SiC or Al to produce ceramic- and metal-matrix composites with improved mechanical, thermal, functional, or even multifunctional properties. This review presents a perspective on the feasibility of manufacturing ceramic- and metallic-based $MoSi_2$ composite materials. A comprehensive discussion of the pros and cons of current liquid-state and solid-state processing routes for $MoSi_2$ metal-matrix composites and the resulting typical microstructures is presented. Although $MoSi_2$ has been studied for more than five decades, it was not until recently that industrial applications demanding high temperature and corrosion resistance started utilizing $MoSi_2$ as a bulk material and a coating. Furthermore, beyond its traditional use due to its thermal properties, the most recent applications include it as a contact material in microelectronic components or circuits and optoelectronics. The short-term global growth predicted for the $MoSi_2$ heating elements market is expected to significantly impact possible new applications, considering its potential for reuse and recyclability. A prospective assessment of the application of recycled $MoSi_2$ to composite materials is presented.

KEYWORDS

$MoSi_2$ composites, processing routes, properties, reinforcement, applications

1 Introduction

Intermetallic $MoSi_2$ is a moderate-density (6.23 g/cm^3) material that has been widely used to manufacture high-temperature heating elements due to its thermal properties. It is a material, that is, chemically stable, which makes it ideal to be applied in the development of composite materials with a ceramic or metallic matrix, either as a reinforcement/thermal/functional phase or as a matrix phase. In addition, $MoSi_2$-based composites can be manufactured through different processing routes, either by solid-state or liquid-state methods (Pech-Canul and Makhlouf, 2000; Bhattacharya et al., 2002; Houan et al., 2004; Köbel et al., 2004; Cemal et al., 2012; Khanra et al., 2012; Venkateswaran et al., 2015; Gousia et al., 2016; Contreras-Cuevas et al., 2018). The utilized processing methods include powder metallurgy, spontaneous infiltration, mechanicalalloying, thermal spray, and pressure infiltration, amongst others.

Around the 1980s, the properties of intermetallic $MoSi_2$ were studied, and it proved to be an excellent material to be applied in metal matrix composites (MMC) (Schlichting, 1978; Fitzer and Remmele, 1985). However, it was not until the mid-1990s that research on $MoSi_2$-based composites began, emphasizing the mechanical, physical, and thermal properties of the material. But, the subject of the potential applications of a $MoSi_2$-based composite has not been dealt with in-depth, whether in components for electronic packaging, heat sinks, components for aircraft engines, etc. High-temperature heating elements are the most requested application to date. Another issue that has not received the corresponding attention has to do with the recycling and reuse of components that are manufactured with $MoSi_2$ to be used as raw material in the development of composite materials.

It is expected that the market for molybdenum disilicide ($MoSi_2$) heating elements will grow significantly by 2025 with marketplace segmentation by players (Kanthal, I Squared R, Zircar, and MHI, amongst others), by types (1700°C, 1800°C, and 1900°C grades), and by end-users (Laboratory and Industrial furnaces). The confluence of these factors, together with the pinpoint spotting of the consumption market by regions (North America, Europe, Asia Pacific, Latin America, Middle East, and Africa), suggests the economic influence of this crucial intermetallic material (Global Molybdenum Disilicide, 2021).

This review provides constructive criticism of the state of the art of intermetallic $MoSi_2$ to produce composite materials, comparing its properties with those of other materials that can be combined, the different production methods, and applications.

2 Intermetallic $MoSi_2$: crystal structure, properties, and applications

Molybdenum disilicide ($MoSi_2$) is an intermetallic material of moderate density (6.23 g/cm^3) that has generated interest in various industrial fields due to its physical, mechanical, and thermal properties (Yao et al., 1999). Due to its high melting point (2030°C), its main use is for high-temperature applications such as resistors or heating elements, and as it is resistant to corrosion, it is an ideal material for corrosive environments. When subjected to high temperatures, it forms a passive layer of silicon dioxide (SiO_2), which protects it from oxidation (Cook et al., 1992). In the electrical industry, the use of $MoSi_2$-based composite materials has generated special interest, since this intermetallic alone has a low thermal expansion coefficient in the range of 20°C–1400°C (CTE = 7–10 × 10^{-6} K^{-1}) (Petrovic, 1992), a good thermal conductivity at 25°C (κ = 66.2 W/m K) (Kulczyk-Malecka et al., 2016), and a good Young's modulus (E = 440 GPa) (Nakamura et al., 1990).

Intermetallic $MoSi_2$ has two polymorphic phases: the alpha and the beta modifications. The alpha phase, the most stable polymorph up to 1900°C, has a body-centered tetragonal structure ($C11_b$-type) (Figure 1) and belongs to the space group No. 139, $I4/m2/m2/m$ ($I4/mmm$, Patterson symmetry) (Hahn, 2005), tI6 in Pearson notation. Its lattice parameters are $a = b$ = 3.203 Å and c = 7.855 Å (Lide, 2005; Cardarelli, 2008; Villars and Cenzual, 2012). According to Waghmare et al. (1998), the characteristics of $MoSi_2$ in its alpha form may favor an improvement in mechanical properties. According to Gokhale and Abbaschian (1991), $MoSi_2$ undergoes a polymorphic transformation from a low-temperature tetragonal form (α-$MoSi_2$) to a high-temperature hexagonal form (β-$MoSi_2$) at 1900°C, which corresponds to the Mo-Si equilibrium phase diagram (Figure 2) (Gokhale and Abbaschian, 1991). The beta phase has a hexagonal structure (C40-type) and belongs to the space group No. 180, $P6_222$ ($P6/mmm$, Patterson symmetry) (Hahn, 2005); its lattice parameters are a = 4.60 Å and c = 6.55 Å (Zamani et al., 2012).

It is a material soluble only in nitric acid (HNO_3) and hydrofluoric acid (HF) (Cardarelli, 2008). The most common ways of preparing $MoSi_2$ and $MoSi_2$-based composites are by sintering and plasma spraying (Yao et al., 1999), the latter being used to form dense pieces. The densification of materials aims to produce a material that can be easily handled and to increase its mechanical and physical properties.

According to Yao et al. (1999), with the plasma spray process, it is possible to achieve near-net shape composites and consolidate the material in a single operation, and under a rapid solidification process, fine-grained and chemically homogeneous microstructures are achieved. According to Koch (1998), solid-state processing routes, such as mechanical alloying and powder metallurgy, offer benefits such as relatively simple processes and the flexibility they offer when manufacturing MMCs. Due to the chemical reaction with silicon to form silicides, $MoSi_2$ has limited applicability for hardening ductile phases with metallic phases, and using ceramic phases such as SiC or ZrO_2 as reinforcing phases has a modest effect on improving and increasing plastic flow and toughness (Ito et al., 1996).

The properties of $MoSi_2$ were described for the first time in 1949 by Maxwell (1945), who argued that this material had desirable high-temperature properties such as oxidation resistance, good high-temperature fracture strength, and good thermal conductivity and ductility at high temperatures (1315°C). However, $MoSi_2$ has some drawbacks, such as susceptibility to harmful oxidation at low temperatures (400°C–600°C) (Chou and Nieh, 1993a), high creep rate above 1200°C (Yao et al., 1999), and oxygen diffusion at the grain boundaries at high temperatures (T > 900°C) (Chou and Nieh, 1993b). At 1600°C, its yield strength at 0.2% displacement is approximately 20 MPa (Sharif et al., 2001). Since it is brittle below 900°C, with low fracture toughness (2–4 MPa m$^{1/2}$) (Yao et al., 1999), its high-temperature structural applications are limited. Consequently, it is more attractive for the design of composites for low- and moderate-temperature applications. Table 1 shows the physical, mechanical, and thermal properties of $MoSi_2$.

However, using ceramic and refractory reinforcements in $MoSi_2$ composites has improved the mechanical properties and conferred better resistance to high temperatures. Some studies used $MoSi_2$ as a reinforcing phase in ceramic-matrix composites for high-temperature applications, as in the work of Grohsmeyer et al. (2019). They investigated the densification behavior and resulting microstructures of hot-pressed ZrB_2-$MoSi_2$ composites using different ZrB_2 particle sizes (3–12 um) and $MoSi_2$ contents (5–70 vol%). It has been found that $MoSi_2$ is a beneficial phase for the densification of ZrB_2 at 1750°C due to the formation of a transient liquid phase (Silvestroni et al., 2018). According to Sciti et al. (2006a) in UHTC (ultra-high temperature ceramics) ZrB_2-$MoSi_2$ composites, the microstructural analysis indicates that at least 20% vol. $MoSi_2$ is needed to obtain a dense material. In addition, at temperatures above 1200°C, the oxidation of $MoSi_2$

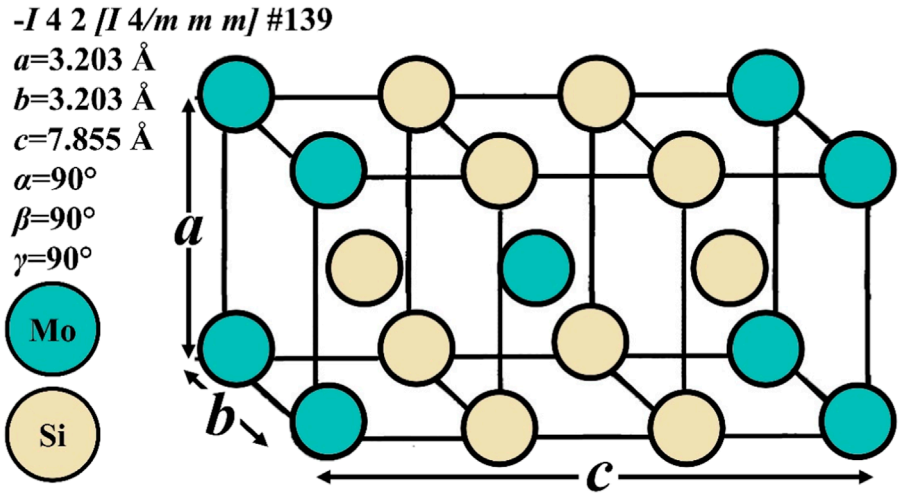

FIGURE 1
Tetragonal crystal structure and lattice parameters of $MoSi_2$ (Petrovic, 1992; Yao et al., 1999).

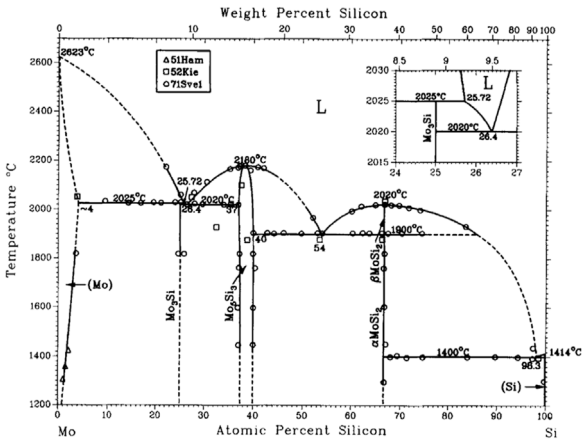

FIGURE 2
Mo-Si phase diagram (Gokhale and Abbaschian, 1991).

forms a silica layer that seals the composite's surface, thus preventing ZrB_2 degradation (Sciti et al., 2005; Sciti et al., 2006b).

As illustrated in the micrograph of Figure 3A, the typical microstructure of a $MoSi_2$ monolith features a grey color phase corresponding to the $MoSi_2$ matrix. The dark phases are SiO_2 and the white areas belong to the Mo_5Si_3 phase due to a silicon deficiency or a reaction with oxygen (Shan et al., 2002). EDS analysis (Figure 3B) shows the elements present in the microstructure phases. The diffractogram made by XRD analysis (Figure 3C) shows the phases present, with the $MoSi_2$ phase with the highest intensity peaks, compared to the Mo_5Si_3 and SiO_2 phases with the low-intensity peaks. The DSC-TG diagram (Figure 3D) shows that $MoSi_2$ is stable at temperatures of 1200°C and with weight losses down to 0.14%.

Mitra et al. (2003) studied the behavior of $MoSi_2$ at elevated temperatures (1000°C–1350°C). One of the samples fabricated for the study was made by the reaction-hot-pressed (RHP) process. For this purpose, Mo (3 μm) and Si (20 μm) powders were mixed and degassed under vacuum at 800°C for 4 h. Subsequently, the samples were hot pressed (1500°C) under vacuum conditions (1024 Pa), using 26 MPa pressure. Figure 4A shows the microstructure of the RHP $MoSi_2$ sample, and according to the authors, the average grain size obtained using the linear intercept method was 5 μm. The HRTEM image (Figure 4B) shows a grain boundary of RHP $MoSi_2$, which deformed at 1,200°C, with a strain rate of 10^{-3} s^{-1}.

Intermetallic $MoSi_2$ has been used as a reinforcing material and in the matrix phase in composites. In aluminum matrix composites (AMC), as a reinforcement, $MoSi_2$ has improved the material's wear and corrosion resistance compared to a monolithic matrix material (Gousia et al., 2016). Recently, $MoSi_2$ has been considered a strong candidate as a contact material in microelectronic components or circuits where it is necessary to increase the conductivity and signal speed (Wetzig and Schneider, 2006). Also, due to its thermal properties, it is an ideal material for applications in components subjected to high temperatures, such as heating elements in electric furnaces or combustion chamber components.

Since the early 1990s, studies have been conducted on developing and processing (Maloney and Hecht, 1992) and determining the physical, mechanical, and thermal properties of $MoSi_2$-containing composites (Xiao et al., 1991; Xiao and Abbaschian, 1992; Pill Lee et al., 2012). More recently, intense research has been done exploring different applications, such as vehicle components for satellite launches, of $MoSi_2$ and $MoSi_2$-based composites rather than their thermal uses (Khanra et al., 2012; Zhang et al., 2019; Zhu et al., 2022a). Remarkably, in 2022, researchers from Bangladesh (Liton et al., 2022) reported a breakthrough in the optoelectronic properties of α-$MoSi_2$ and concluded with an analysis of optical parameters (parts of dielectric constants, absorption coefficient, reflectivity, etc.) that α-$MoSi_2$ may be suitable for applications in QLED (Quantum Light Emitting Diode) and OLED (Organic Light Emitting Diode) devices, as well as potential absorbers to hinder solar heating by UV radiation.

TABLE 1 Physical, mechanical, and thermal properties of $MoSi_2$.

Physical properties		Ref
Molar Mass (M_a)	152.11 g/mol	Villars and Cenzual (2012)
Density (ρ)	6.23 g/cm^3	Lide, 2004; Cardarelli (2008)
Mechanical Properties		
Vickers Hardness (HV)	900–1,200	Gokhale and Abbaschian (1991), Cardarelli (2008)
Ultimate Tensile Strength (UTS)	276 MPa (at 25°C)	Cardarelli (2008)
Modulus of Elasticity (E)	440 GPa (at 25°C)	Abdollah-Pour et al. (2014)
Flexural Yield Strength (MOR)	220 MPa	He et al. (2018)
Compressive Yield Strength (σ_{mc})	600 MPa (at 1200°C)	Sharif et al. (2005)
Fracture Toughness (K_{IC})	3 MPa m$^{1/2}$ (at 25°C)	Petrovic (1995)
Thermal Properties		
Coefficient of Thermal Expansion (CTE, α_l)	7×10^{-6} K^{-1} (at 25°C)	Kulczyk-Malecka et al. (2018)
Thermal Conductivity (κ)	66.2 W/m·K (at 25°C)	Kulczyk-Malecka et al. (2016)
Melting Point (T)	2030°C (2300 K)	Gokhale and Abbaschian (1991), Christensen (1993)

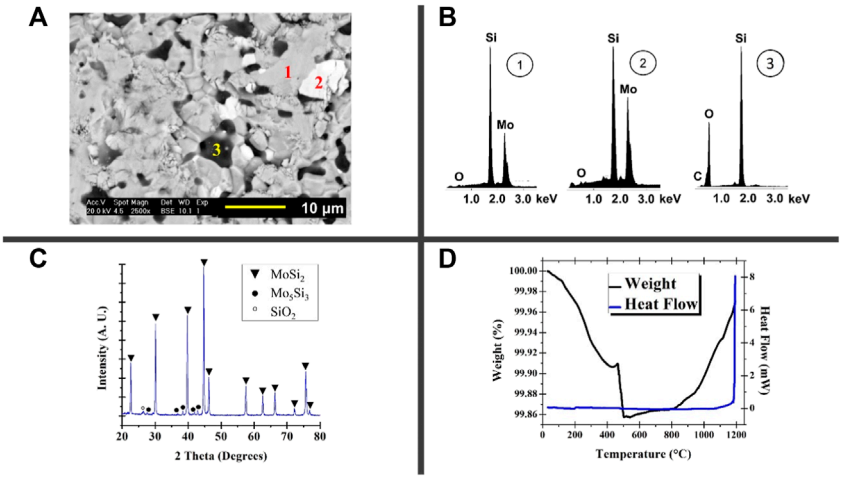

FIGURE 3
(A) SEM image by backscattered electrons, **(B)** diffractograms of point EDS analysis of the phases present, **(C)** XRD diffractogram, and **(D)** DSC-TG analysis diagram of a recycled $MoSi_2$ heating element section (Kanthal® Super 1800).

2.1 Oxidation of $MoSi_2$

Because intermetallic $MoSi_2$ has excellent resistance to oxidation in dry or humid air, and in oxygen or strongly oxidizing environments when reaching temperatures higher than 600°C (Ito et al., 1996), its oxidation resistance becomes of particular interest in studies of $MoSi_2$-based composites. However, heavy oxidation of $MoSi_2$ has been observed because of the slow growth of the protective layer of amorphous SiO_2, in addition to the fracture of an initial protective layer of Mo_9O_{26}-SiO_2 (Meschter, 1990). Due to this oxidation resistance, $MoSi_2$ is an ideal candidate as a protective coating on materials that do not support highly oxidizing environments. The presence of oxygen impurities that exceed 2 at % in the $MoSi_2$-silicon interface causes the formation of metal-rich silicide phases; the coexistence of these phases is predictable using a Mo-Si-O ternary phase diagram (Wakita et al., 1984). It is worth noting that at 1300°C, SiO_2 undergoes a phase transformation from tridymite to cristobalite. This modification in the oxide causes a greater formation of SiO_2 and evaporation of MoO_3 (Melsheimer et al., 1997).

SiO_2 is formed when $MoSi_2$ encounters oxygen (Reaction 1) (Wakita et al., 1984; Meschter, 1990; Melsheimer et al., 1997). There are two possible oxidation reactions (Reactions 2 and 3) and both are thermodynamically feasible (Gamutan and Miki, 2022).

However, the reaction where MoO_3 is present is favored since it results from oxygen saturation between 400°C and 600°C (Yao et al.,

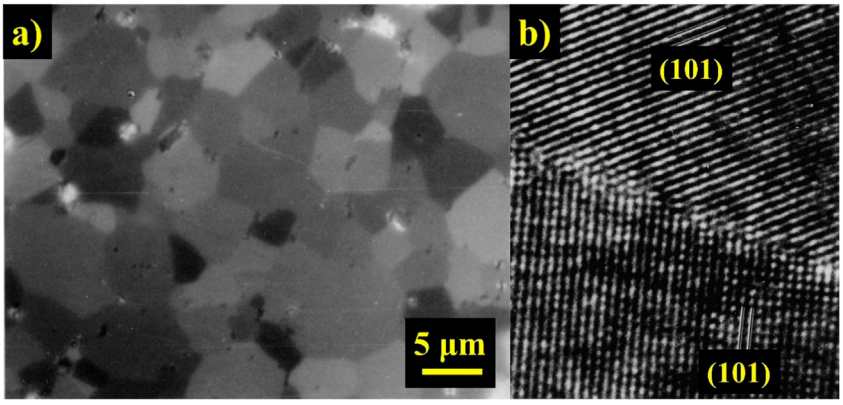

FIGURE 4
(A) Polarized-light optical micrographs of reaction-hot-pressed (RHP) $MoSi_2$ and **(B)** HRTEM image of a typical grain boundary in RHP $MoSi_2$ after testing at 1200°C with a strain rate of 10^{-3} s^{-1} (Mitra et al., 2003).

$2MoSi_{2(s)} + 7O_{2(g)} \rightarrow 2MoO_{3(s)} + 4SiO_{2(s)}$	$\Delta G^o[kJ/mol] = -547.918 + 0.1146T$	**Reaction 1**
$5MoSi_{2(s)} + 7O_{2(g)} \rightarrow Mo_5Si_{3(s)} + 7SiO_{2(s)}$	$\Delta G^o[kJ/mol] = -808.260 + 0.1672T$	**Reaction 2**
$3MoSi_{2(s)} + 5O_{2(g)} \rightarrow Mo_3Si_{(s)} + 5SiO_{2(s)}$	$\Delta G^o[kJ/mol] = -800.038 + 0.1729T$	**Reaction 3**

1999). At high temperatures (1,050°C–1,470°C), SiO_2 undergoes a crystal structure transformation from tridymite (orthorhombic) to cristobalite (tetragonal) and these silica polymorphs have lower densities (Nakae et al., 1998). The formation of the SiO_2 layer is crucial since it is the reason for oxidation resistance in corrosive environments, and the faster its formation, the greater the oxidation resistance, which is an advantage for those materials (metals, ceramics, MMC or CMC) protected with $MoSi_2$ layers or $MoSi_2$ composite layers (Pech-Canul et al., 2000a; Mortensen, 2000; Zumdick et al., 2001; Rodríguez-Reyes et al., 2003). However, in the manufacture of composites by liquid state routes, forming this silica layer can make it challenging to incorporate the liquid metal into the porous body to be infiltrated since ceramics have poor wettability on their surface (Nakae et al., 1998; Pech-Canul et al., 2000a; Mortensen, 2000; Rodríguez-Reyes et al., 2003; Tapia-López, 2018).

In investigations of the last two decades, $MoSi_2$ and $MoSi_2$ composites have been used as protective layers to protect ceramic matrix composite (CMC) materials that will be exposed to high temperatures (Ibano et al., 2007; Zhu et al., 2022b; Ren et al., 2022) and corrosive environments, especially in highly oxidizing environments that can embrittle the material (Xu et al., 1999; Zhao et al., 2006; Fu et al., 2009; Sooby Wood et al., 2016; Bezzi et al., 2019; Ren et al., 2020). Most researchers seek for the materials to be protected to not only not rust easily but also have high thermal shock resistance and that a dense protective SiO_2 layer forms at high temperatures to prevent oxidation for as long as possible (Yan et al., 2009; Zhao, 2010; Wang et al., 2017; Yang et al., 2021; Wei et al., 2022).

3 $MoSi_2$-based composites

$MoSi_2$-based composites have used different types of reinforcement, ranging from fine particles (Khanra et al., 2012), continuous fibers (Bhatti and Shatwell, 1997), and whiskers (Gac and Petrovic, 1985; Sun and Pan, 2001). Continuous fiber reinforcements have been used, such as ceramic fibers (SiC, Al_2O_3) and high-resistance ductile fibers (tungsten and molybdenum alloys), achieving composite creep strength values of 70–100 MPa at 1100°C, high-temperature resistance (T > 1400 C), and a fracture toughness value of 37.5 kJ/m^2 (Maloney and Hecht, 1992). However, SiC whiskers used as reinforcement have shown better properties at high temperatures and oxidation resistance due to the formation of a silica layer (Carter et al., 1989).

In 1978, $MoSi_2$ became a matrix material for composites and ceramics or refractories as reinforcement material (Schlichting, 1978). Since the mid-1980s, other types of materials have been used to reinforce $MoSi_2$ composites, improving their properties even at room temperature (Yao et al., 1999). Such is the case of the niobium wire reinforcements with which the mechanical properties were improved at room temperature (Fitzer and Remmele, 1985). Niobium is a ductile material, making it suitable for $MoSi_2$ matrix composites due to its high melting temperature and a CTE close to that of the matrix. This makes fewer cracks in the matrix under a thermal cycle (Alman et al., 1992; Gibala et al., 1992; Xiao and Abbaschian, 1992; Shaw and Abbaschian, 1993). Silicon carbide whiskers (SiC_w) improved fracture toughness and strength at room temperature (Gac and Petrovic, 1985). Using 0.1 μm diameter SiC_w as a reinforcing material within the composite showed that the levels

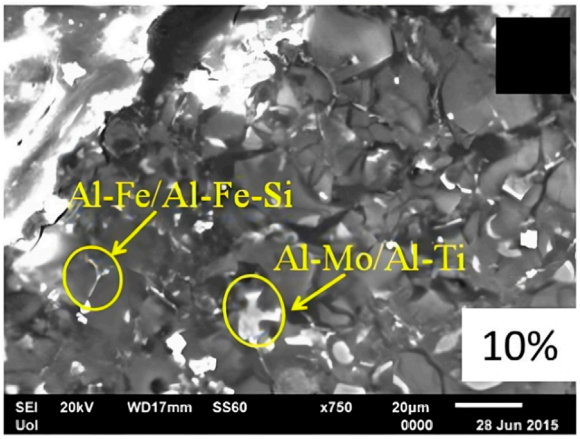

FIGURE 5
SEM micrograph of the produced composite of Al/10%$MoSi_2$ (Gousia et al., 2016).

of mechanical properties were within the range of high-temperature engineering applications (Carter et al., 1988). The carbon present in the SiC helps to reduce the appearance of oxides in the $MoSi_2$, in addition to avoiding the formation of the siliceous phase at the grain boundaries, especially in samples processed by hot pressing (Yao et al., 1999). Furthermore, according to Carter et al. (1989) the $MoSi_2$ matrix should not react with SiC from the thermodynamics viewpoint. However, as it has a ceramic reinforcement, such as SiC, the composite can show fragile behavior at room temperature due to the lack of independent, active sliding systems.

In some cases, $MoSi_2$ is used as a reinforcing material in metal matrix composites (MMC). Such is the case of aluminum matrix composites (AMC), which are materials with good mechanical properties and that have structural, functional, or high-tech applications, as is in the aerospace, automotive, defense, and electronic packaging industries, to name a few (Gousia et al., 2016). The most common reinforcements in an AMC are those with ceramic phases such as Al_2O_3, SiC, and AlN. However, intermetallic reinforcements have become relevant and offer properties with functional applications (Surappa and Rohatgi, 1981; Mortensen and Jin, 1992; Surappa, 2003; Kainer, 2006a).

In the work by Gousia et al. (2016), an AMC was prepared with an Al 1050 matrix and the addition of $MoSi_2$ powder with a particle size of 100–300 nm at approximately 1–10 µm. The reaction between the Al 1050 alloy and 10% of the intermetallic $MoSi_2$ led to intermetallic phases such as $Al_{12}Mo$, Al_5Mo, Al_4Mo, and Al_3Ti. It was also observed in the micrographs (Figure 5), the Al-Fe-Si phase, which increased the hardness of the material, and the Al-Mo-Ti intermetallic phase, showing a benefit in sliding wear and erosion behavior.

Some challenges faced during the heat treatment of $MoSi_2$-containing composites are the formation of undesirable phases or excessive oxidation of $MoSi_2$ that may deteriorate the composites' integrity and properties. Accordingly, some authors have focused on process temperature, residence time, and atmosphere as critical heat treatment parameters (Zeng et al., 2002; Zamani et al., 2012; Niu et al., 2013; Abdollah-Pour et al., 2014).

3.1 Processing routes for $MoSi_2$-based composites

The production of metal matrix composites (MMC) can be done using different processing techniques (Figure 6), ranging from the solid-state technique, the liquid-state technique, the metallurgical powder (PD) technique, the physical vapor processing (PVD) technique, and direct processing/spray deposition technique, (Adeosun et al., 2014; Contreras-Cuevas et al., 2018). Instead, in recent years, only the simplest and most specialized techniques, such as mechanical alloying and the infiltration process, have been used because they are viable, low-cost processes and have the possibility of making irregular shapes. These pieces, in the end, can have a net shape or a near-net shape (Zweben, 1992; Contreras-Cuevas et al., 2018).

According to work by Lee et al. (2019), the conventional manufacturing processes used to prepare aluminum matrix composites (AMCs) can become complex and highly specialized processes that require specific skills and unique equipment, becoming expensive and limited in their application. Therefore, the authors outline a more straightforward route to AMC manufacturing, which involves mixing Al powder and ceramic reinforcement.

However, the most straightforward methodologies to produce MMC and AMC have had better results in terms of mechanical and thermal properties, especially for hybrid composites that require these properties for their final application (Adams and Miller, 1975; Premkumar et al., 1992; Surappa, 2003; Adeosun et al., 2014). In this review, the authors discuss the methodologies that have presented favorable results in producing composites with $MoSi_2$, either as a reinforcement material or as a matrix phase.

3.1.1 Solid state processing methods

The solid-state process is suitable for reactive systems, in addition to having some advantages over liquid-state processes such as minimal segregation and reaction between the reinforcement and the matrix (Contreras-Cuevas et al., 2018). The goal of using these solid-state processes is to achieve higher mechanical properties in MMCs, but this makes them relatively expensive processes.

Krakhmalev et al. (2004) developed a composite consisting of $MoSi_2$ as the matrix and an aluminum alloy, using three different reinforcements: Al_2O_3, SiC, and ZrO_2. The composites were prepared using the spark plasma sintering method (SPS) of powders produced by mechanical alloying (MA), annealing the specimens at 1600°C for 5 h. It was explained that MA allows for achieving a fine-grained intermetallic alloyed powder and that, together with SPS, the $MoSi_2$-based composites can be effectively sintered with fine microstructure. The SPS of MA powders method proved effective for forming an ultra-fine structure of microns or submicrons of $Mo(Si,Al)_2$, containing retained phases of Mo and/or Mo_5Si_3.

The powders for the $MoSi_2$ composite were prepared by mixing elemental powders with a ceramic phase in a planetary ball mill in an argon atmosphere for 12 h. The elemental powders (99.99% purity) were mixed with a second phase (average particle size ≤1 µm) in a high-energy ball mill. For the SPS of the MA powders, the samples were compacted and then sintered at 1350°C for 3 min.

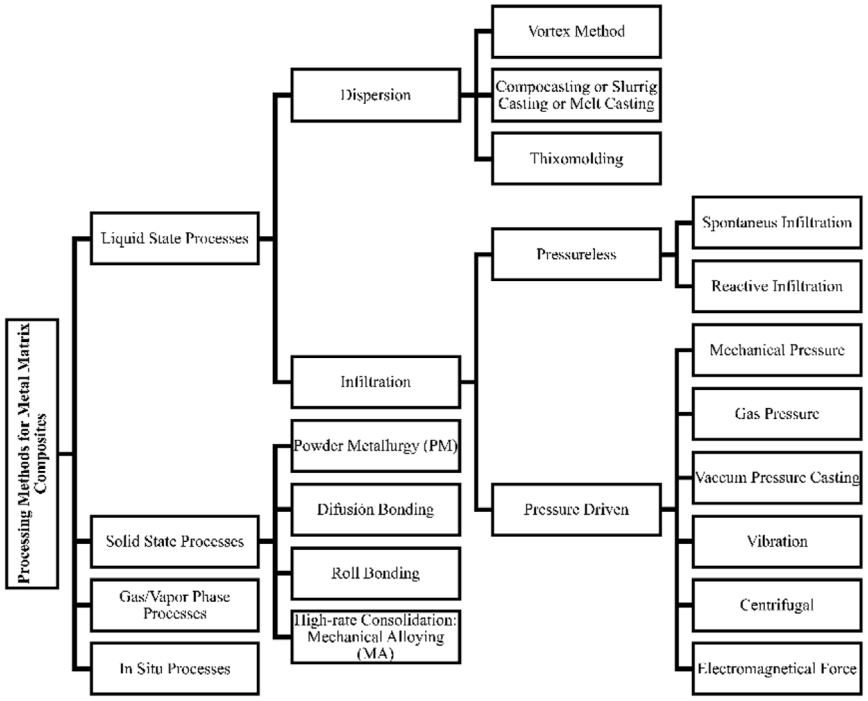

FIGURE 6
Solid-state and liquid-state fabrication processes for metal-matrix composites.

Subsequently, the samples were isothermally treated in an argon atmosphere at 1600 C for 5 h.

The results showed that in the sample of $Mo_{0.63}Si_{0.27}Al_{0.10}$ (wt%)/20 vol% SiC, an ultrafine structure of $Mo(Si,Al)_2$ and retained phases of Mo and/or Mo_5Si_3 were observed after sintering of the composite. In the sample heat-treated by annealing (T = 1600 C for 5 h), a coarsening of the C40 $Mo(Si_{0.75}Al_{0.25})_2$ matrix and the ceramic phase (SiC) was observed as well as the transformation of the solid solution of retained Mo (Figure 7).

Krakhmalev et al. (2004) conclude that the composites show higher thermal stability with certain annealing conditions and that the fracture toughness depends on the quantity and nature of the second phase. Furthermore, it was found that microstructure refinement combined with a composite structure improves fracture toughness by 20%–30%. However, this would be an insufficient improvement for industrial applications with C40 $Mo(Si_{0.75}Al_{0.25})_2$ matrix, as the negative effect of matrix coarsening on hardness and fracture toughness due to the annealing treatment of the samples has been demonstrated.

In the work by X. Yi, W. Xiong, and J. Li (Yi et al., 2007), a Cu-$MoSi_2$ composite was manufactured using the powder metallurgy process to develop an advanced thermal material with high thermal conductivity and a low coefficient of thermal expansion. According to the authors, this material can be applied in heat sinks, electrical packaging of semiconductors, or high-power electronic devices. The choice of intermetallic $MoSi_2$ over other more common reinforcing materials, such as SiC or Al_2O_3, is because $MoSi_2$ has excellent oxidation resistance and good thermal and electrical conductivity, which makes it an ideal candidate to be used as a thermal or functional phase in the composite.

Electrolytically precipitated pure Cu powders (>99.5%, 40 μm) and high purity $MoSi_2$ ultrafine powders (1.8–2.3 μm) were used as raw materials. Each composite material was processed through a high-energy ball mill for 10 h and a rotation speed of 300 rpm. The $MoSi_2$ and Cu powders were placed into a marble container and mixed in a planetary ball mill. Subsequently, the bar shape samples (40 mm × 80 mm × 10 mm) were cold pressed at a compaction pressure of 200 MPa. To increase the green strength, they were compacted at room temperature under cold isostatic pressure (CIP) at 60 MPa. Once the compaction process was completed, the samples were sintered at 950°C in an atmosphere of H_2. Finally, each sample was subjected to three passes of hot rolling, having a maximum deformation of 55%, and then annealed in an atmosphere of H_2 gas at 950°C for 2 h.

Scanning electron microscopy results showed the uniform distribution of $MoSi_2$ particles. In the micrographs of the fracture surface (Figure 8), the voids present at the interfaces during plastic deformation are observed, where the bond strength between $MoSi_2$ and the matrix is weak. As the deformation continues, the voids agglomerate into larger holes, resulting in the final fracture. These agglomerated voids dimple the surface of the fracture.

Regarding the composite's thermal properties, the results showed that the samples with the highest thermal conductivity were the pure copper control sample and the Cu-2% $MoSi_2$ composite. As the $MoSi_2$ content increased, the thermal conductivity decreased considerably, attributed to the constant increase in the electron scattering in the crystal structure. According to Yi et al. (2007), the CTE value of pure copper at 100°C is 9.7×10^{-6}/°C. However, in Cu/$MoSi_2$ composites, the composites tested at 100°C showed

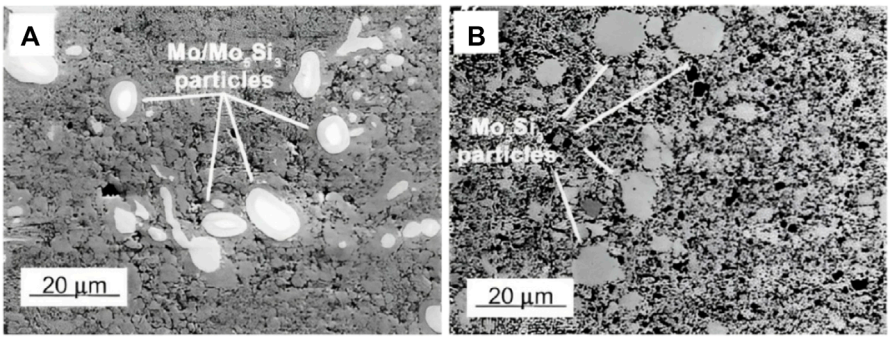

FIGURE 7
The microstructures (backscattering electrons images) of the $Mo(Si_{0.75}Al_{0.25})_2$/20 vol% SiC matrix composites in the as-sintered **(A)** and annealed **(B)** states. The white phases in bright inclusions in **(A)** are Mo solid solution cores and the bright inclusions in **(B)** are Mo_5Si_3 (Krakhmalev et al., 2004) (With permission).

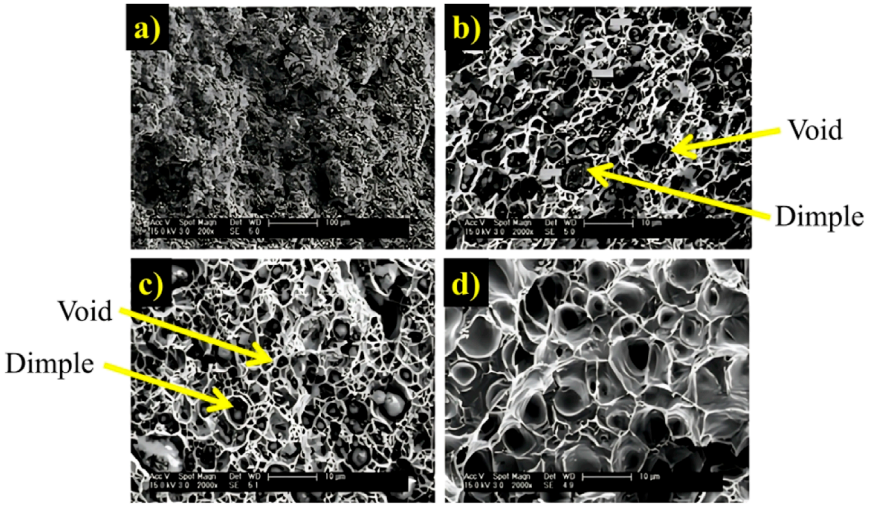

FIGURE 8
SEM images of the tensile fracture surface of the composites: **(A)** Overall morphology (Cu-2%$MoSi_2$); **(B)**, **(C)** dimples and voids in Cu-2% and −8%$MoSi_2$ samples respectively; **(D)** high magnification of the pure Cu sample (control sample) (Yi et al., 2007) (With permission).

good thermal conductivity, lower thermal expansion coefficient, and better tensile stress, ascribed to the Orowan reinforcement mechanism. The sample with the lowest CTE value was Cu-2% $MoSi_2$ (3.4×10^{-6}/°C), while the Cu-6% $MoSi_2$ and pure Cu samples had the highest CTE values with 9×10^{-6}/°C and 9.7×10^{-6}/°C, respectively. This result is ascribed to an uneven distribution of secondary phases and 10% porosity on average in the samples. On a macroscopic scale, the existence of pores prevents the composites from expanding. It was concluded that the Cu-$MoSi_2$ composites show good thermal conductivity and a low CTE (Yi et al., 2007) and it was proposed to improve the purity and density of the materials used so that they can be applied as heat sinks in electronic packaging systems. Although Cu/$MoSi_2$ composites are promising prospects for the aerospace, electrical, and electronic industries, research works are very limited or scarce, particularly regarding microstructure characterization, such as identifying secondary phases formed during synthesis (Yi et al., 2007; Hu et al., 2008).

3.1.2 Liquid state processing methods

The liquid-state processes are one of the most used fabrication routes to produce composites at an industrial level because they are low-cost and allow cost-effective final products. These products have good properties compared to those fabricated in solid-state, so they are strong candidates to be applied in different industrial sectors (Bezzi et al., 2019). The advantage of using the liquid-state processes is that there is a strong bond between the matrix and the reinforcement. However, the molten state route may produce undesirable reaction products that embrittle the metal/reinforcement interface and lower the material´s strength. Various pieces of research have been conducted to fabricate Metal/$MoSi_2$ composites.

In the work by Ramasesha and Shobu (1998), aluminum was infiltrated into a $MoSi_2$ preform using the reactive melt infiltrating process. $MoSi_2$ powder with an average particle size of 2.93 μm and Al-12.24%Si alloy powder with an average particle size of 17.33 μm

were used. For the preforms, compacts of 12 mm diameter pellets with different densities were fabricated: compacts with 40% density were made by cold pressing, and compacts with 85% density were prepared by hot pressing at 1300°C using a graphite die. Each compact was brought to a pressure of 20 MPa. The aluminum alloy powders were also compacted into pellets. The pellets were placed in a graphite crucible, first accommodating the aluminum pellets and the $MoSi_2$ pellets on top of them. The test was performed under a vacuum or with an argon atmosphere for infiltration. The temperature range ranged from 1050°C to 1500 C and the permanence times ranged from 5 to 30 min. The obtained composite was cut in half and prepared metallographically to be analyzed in a scanning electron microscope. The results showed that the $MoSi_2$ compacts with 60% porosity and using a temperature of 1050°C for 15 min, the infiltration was superficial, that is, only around the circumference of the pellet, so it did not penetrate the preform. A similar case occurred with the compact subjected to a temperature of 1100°C and 20 min of permanence, but the infiltrated layer was thick (approximately 6 mm thick). In the sample treated at 1150°C, the infiltration was completed in 20 min, and in another sample subjected to a temperature of 1200°C, it took only 5 min to reach complete infiltration.

For the cold-pressed samples with excess molybdenum (8 at%), the infiltration was carried out both under vacuum conditions and under an inert atmosphere of argon. A temperature of 1200°C was reached for 20 min and subsequently annealed at 1500°C for 10 min. The images taken by SEM (Figure 9) show that if an inert atmosphere is used, this does not affect the degree of infiltration compared to the sample under vacuum conditions and whose infiltration is completed in 5 min. Regarding the microstructure, it was observed that the $Mo(Al,Si)_2$ phases have a hexagonal morphology in any of the conditions for infiltration. As for the hot-pressed samples, as they have a high density (85%), the infiltration is completed in 20 min. The SEM images show a remarkable similarity when compared to the cold-pressed samples. If the sample was cooled from 1200°C, the morphology of the phases was random, but upon annealing at 1500°C, the morphology changed to a hexagonal-shaped grain. It was concluded that it is possible to infiltrate aluminum into $MoSi_2$ preforms satisfactorily, regardless of the density percentage of the compacts. The factor determining the wettability of $MoSi_2$ by liquid aluminum is the reaction at 1050°C between the SiO_2 film on the raw $MoSi_2$ powder and aluminum, causing the disintegration of the thin silica film. However, the degree of infiltration is low. Only at 1100°C does the degree of infiltration increase drastically. Also, the hexagonal morphology of $Mo(Al,Si)_2$ is achieved by performing a heat treatment at 1500°C for 10 min (Ramasesha and Shobu, 1998).

Gao et al. (2019) prepared $MoSi_2(Cr_5Si_3)$–RSiC composites using a combination of precursor impregnation pyrolysis (PIP) and an active melt infiltration process (AAIM) of $MoSi_2$–Si–Cr alloy. Before the infiltration process, a mixture of $MoSi_2$ (15, 20, 25, 30, and 35 wt%), Si (55, 60, 65, and 70 wt%), and Cr (15, 10 wt%) powders with different concentrations of each constituent was made. For the PIP process, RSiC (re-crystallized silicon carbide) preforms were placed in an autoclave and heated to 30°C. Still inside, later, the sample was placed under vacuum conditions (0.01 MPa), and then the PF (phenol-formaldehyde) solution diluted in ethanol at 30°C was injected. This operation was carried out until the preform was immersed in the solution and left for 2 h. Afterward, the preform was centrifuged at 80°C and cured for 2 h. Finally, the preform was carbonized up to 1000°C for 2 h, using an argon atmosphere.

A graphite crucible was used to infiltrate the preforms at a temperature of 1900°C in a furnace under an argon atmosphere. The $MoSi_2(Cr_5Si_3)$ blocks and the RSiC preform were placed inside the crucible and left for 1 h until the alloy infiltrated by capillary action. The resulting samples were prepared and analyzed by various characterization techniques to measure physical properties (bulk density and apparent porosity) and mechanical properties, such as the modulus of rupture by three-point bending tests and fracture stress. Oxidation resistance was also analyzed, and the samples were prepared metallographically to be analyzed by SEM.

The SEM images (Figure 10) showed that after the infiltration process at 1900°C, the almost dense $MoSi_2(Cr_5Si_3)$-RSiC composites exhibited an interpenetrating three-dimensional network structure. By increasing oxidation times at 1500°C, the mechanical properties fluctuate due to the synergistic effect of the reduction of surface defects and the relaxation of thermal stress, in addition to the growth of the crystals and thickness increase of the oxidized film (up to 4 μm). The fracture toughness of the $MoSi_2(Cr_5Si_3)$ phase reaches a value of 2.24 MPa $m^{1/2}$ when reaching a temperature of 1400°C, showing an improvement of 31.70% compared to the values obtained at room temperature. The three-point flexural strength of the $MoSi_2(Cr_5Si_3)$ phase exhibited an improvement of 16.7% from 139.54 MPa at room temperature to 162.90 MPa at 1400°C. The conclusions indicate that: 1) the $MoSi_2(Cr_5Si_3)$ phase exhibited the highest mechanical properties compared to the RSiC phase, and 2) as temperature increased, both the flexural strength and the fracture toughness increased compared to the results obtained at room temperature. Table 2 compares the mechanical properties of $MoSi_2$-based composites manufactured by the liquid-state and the solid-state processing methods, with $MoSi_2$ as the matrix phase.

There are also works where the mechanical properties of $MoSi_2$-containing composites have been reported (Carter et al., 1988; Gibala et al., 1992; Petrovic, 1995; Ramasesha and Shobu, 1998; Shan et al., 2002; Zeng et al., 2002; Sciti et al., 2006b; Yi et al., 2007; Zhang et al., 2019), however, often only two or three properties are reported and no further information is provided in terms of a comparison between the materials composing the composite, so a satisfactory comparison table of the physical, mechanical, or thermal properties is difficult to make, as much of the data is scattered.

3.2 $MoSi_2$ as matrix phase

Vasudévan and Petrovic (1992) state that silicide matrix composites are an alternative to structural ceramic materials, especially composites designed with $MoSi_2$ as a matrix phase. The use of high-temperature materials can enhance cooling and minimize the weight of the component to be designed. For example, in the case of air turbines, this would lead to reduced design and production costs and efficient fuel combustion, which has a beneficial impact on the environment.

$MoSi_2$ can be combined with other silicides like those listed in Table 3, though some of them are unsuitable, such as W_5Si_3, because it has a higher density. Other ceramic materials (Table 4), such as some oxides, carbides, and nitrides are chemically compatible with $MoSi_2$, making them attractive for

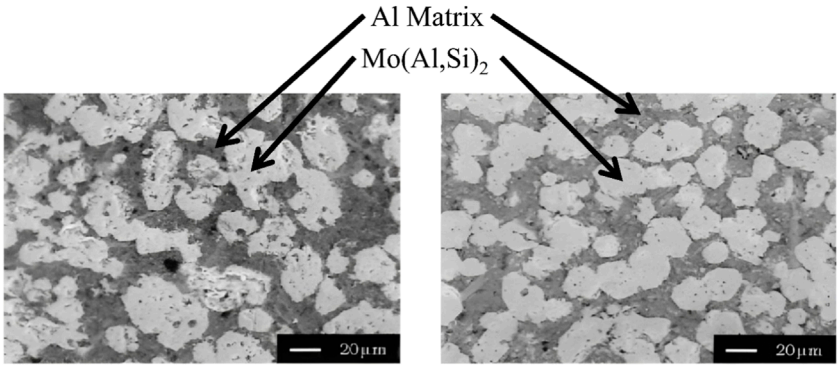

FIGURE 9
$MoSi_2$ preforms containing excess molybdenum. SEM images of cold-pressed samples infiltrated at 1200°C for 20 min and annealed at 1500°C for 10 min: **(A)** Ar atmosphere, **(B)** under vacuum (Ramasesha and Shobu, 1998). Aluminum matrix (dark grey region) and $Mo(Al,Si)_2$ phase (clear grey region) (With permission).

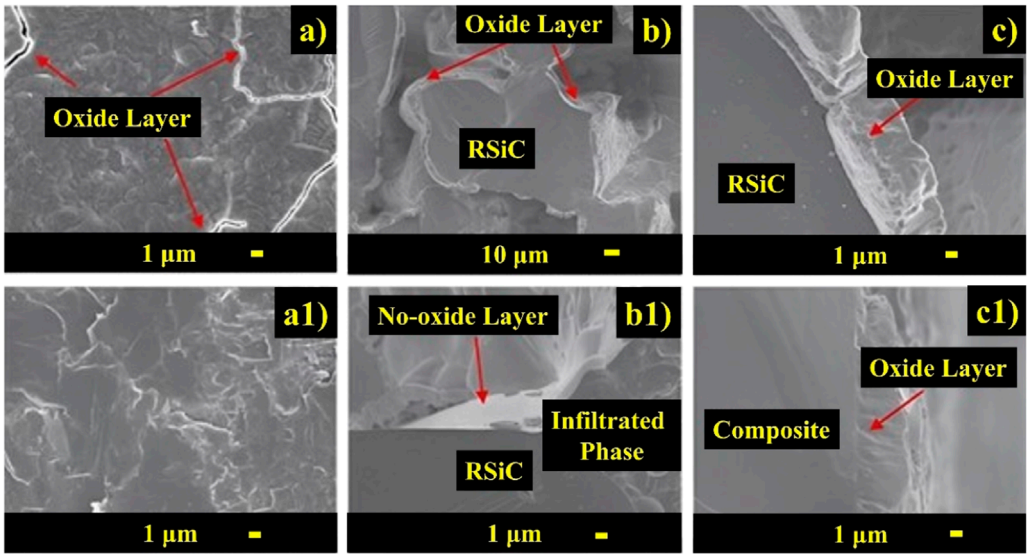

FIGURE 10
SEM images of the microstructure of surface and fracture of RSiC and $MoSi_2(Cr_5Si_3)$–RSiC composites oxidized at 1773 K for 100 h: **(A)** RSiC (surface), **(B)** RSiC (fracture interior), **(C)** RSiC (fracture exterior), **(A1)** $MoSi_2(Cr_5Si_3)$ (surface), **(B1)** $MoSi_2(Cr_5Si_3)$ (fracture interior), and **(C1)** $MoSi_2(Cr_5Si_3)$ (fracture exterior) (Gao et al., 2019) (With permission).

the design and manufacture of $MoSi_2$-containing composites in comparison with SiC, which is chemically unstable with Al_2O_3 and Y_2O_3 as reaction products may form at the interface or undesirable elements may diffuse into the matrix, making them unsuitable for use as reinforcements in a SiC matrix composite.

However, $MoSi_2$-based composites that use the intermetallic as the matrix phase make it a desirable material to be applied in aggressive and corrosive environments, in addition to withstanding high temperatures in a range of 1100°C–1800 °C compared to other silicides that have similar melting points to $MoSi_2$ (Vasudévan and Petrovic, 1992; Lee, 2011).

According to Xiao et al. (1990), significant hardening in $MoSi_2$ can be achieved by strengthening the ductile phase with niobium and W-Re. It was observed that having a 10 vol% Nb, the toughness of $MoSi_2$ is doubled up to an approximate value of 5 MPa $m^{1/2}$. In a similar case, Nekkanti et al. (1990) observed that using a Nb_5Si_3-50 vol% Nb can increase the toughness value of 6–12 MPa $m^{1/2}$. However, these ductile reinforcement phases benefit the silicides' ductility at room temperature. However, in high-temperature applications, the composites can suffer considerable degradation by creep and oxidation.

According to Petrovic (1995), $MoSi_2$ composites' creep mechanisms combine dislocation sliding/scaling processes and grain boundary sliding. As stated by Zamani et al. (2012), possible mechanisms for improving the creep resistance with nanoscale second phases (e.g., Ta, Nb, HfO_2, ZrO_2, SiC, and Mo_5Si_3) are:

TABLE 2 Typical mechanical properties of $MoSi_2$-based composites, fabricated by MA or AAMI.

Composite	Processing technique	Process parameters	Mechanical property	Ref
$Mo(Si,Al)_2$/SiC	Mechanical Alloying (MA)	• Mixing of powders by planetary ball milling for 12 h	• Hardness = 12.9–14.8 GPa	Krakhmalev et al. (2004)
		• Sintering temperature = 1350°C and dwelled for 3 min	• Fracture toughness = 1.8–3.67 MPa $m^{1/2}$ at room temperature	
		• Isothermal heat treatment = 1600°C and dwelled for 5 h in an Ar atmosphere		
$MoSi_2(Cr_5Si_3)$–RSiC	Alloy Active Melt Infiltration (AAMI)	• Mixing of powders by ball milling and heated at 1400 °C for 2 h	• Flexural strength = 139.54 MPa	Gao et al. (2019)
		• PIP Process	• Elastic modulus = 276.77 GPa	
		• Infiltration Temperature = 1900°C, dwelled for 1 h in an Ar atmosphere	• Fracture toughness = 2.24 MPa $m^{1/2}$ at room temperature	

TABLE 3 Crystal structure and properties of high-temperature silicides. Partially from (Petrovic, 1992; Vasudévan and Petrovic, 1992), substantially modified.

Crystal structure	Space group	Silicide	Melting point [°C]	Density [g/cm^3]
Hexagonal	No. 180, $P6_222$	$NbSi_2$	1930	5.66
Tetragonal	No. 139, $I\,4/m2/m2/m$	$MoSi_2$	2030	6.24
		WSi_2	2160	9.86
	No. 140, $I\,4/m2/c2/m$	Mo_5Si_3	2160	8.24
		W_5Si_3	2370	14.50
		Nb_5Si_3	2480	7.16

TABLE 4 Compatibility of reinforcements with $MoSi_2$ and SiC matrices. Partially from (Vasudévan and Petrovic, 1992), substantially modified.

Matrix		
$MoSi_2$	Reinforcement	SiC
Chemically compatible[1]	SiC	Chemically compatible[a]
	Si_3N_4	
	TiB_2	
	Al_2O_3	Chemically unstable[b]
	Y_2O_3	

[a]Chemically compatible with no interfacial reactions;
[b]Chemically unstable with the formation of interface reaction products or the possible diffusion of one or more elements into the matrix.

1) that the melting temperature of the second phase is higher than that of $MoSi_2$, 2) that the second phase has a higher creep resistance than $MoSi_2$, and 3) that the unit cell of the second phase is more complex than that of $MoSi_2$. Another possible factor is the second phase volume fraction in the composite.

Gac and Petrovic (1985) demonstrated that the addition of 20 vol% SiC whiskers (grown by the vapor-liquid-solid (VLS) technique) as a reinforcing medium improves the fracture toughness at room temperature (8.20 MPa $m^{1/2}$) when the $MoSi_2$ matrix is brittle and in addition to increasing the flexural strength at elevated temperatures (310 MPa at 1600°C) when the matrix is ductile.

Carter et al. (1989) showed that SiC whiskers (SiC_w) fabricated by the VLS and vapor-solid (VS) techniques are good structural materials for the reinforcement of $MoSi_2$-matrix composites for high-temperature applications. They prepared composites to increase the matrix phase's mechanical properties by mixing $MoSi_2$ powders with 20 vol% SiC VS and VLS whiskers. Test results at 1200°C showed that composites with SiC_w grown by the VS technique outperform the yield strength value (400 MPa) of those produced by the VLS technique (250 MPa) and those of monolithic $MoSi_2$ samples (150 MPa). Nonetheless, above 1200°C, the yield strength of the composites with the VLS whiskers decreases. The latter is ascribed to the dislocations generated during solidification by the long size SiC_w (200 μm) at a temperature equal to or below 1000°C, making the mean free path larger than the grain size of the $MoSi_2$ matrix (Carter et al., 1988).

3.3 $MoSi_2$ and other reinforcement materials

Due to its physical, mechanical, and thermal properties, intermetallic $MoSi_2$ is compatible with metallic or ceramic materials

such as aluminum, silicon carbide, and others (Minges, 1989). Composites that contain $MoSi_2$ as a reinforcing/thermal/functional or matrix phase have the advantage of withstanding high temperatures and exhibit good thermal conductivity and a low thermal expansion coefficient, making them ideal materials for electronic packaging applications (Yi et al., 2007; Khanra et al., 2012) (Table 5).

When dealing with composites where $MoSi_2$ is used as a reinforcing material, the thermal and mechanical properties become relevant, particularly the thermal conductivity and the thermal expansion coefficient. Table 6 compares the thermal properties of different metal- and ceramic-matrix composites. Due to the ceramic´s chemical composition and crystalline structure, ceramic matrix composites (CMCs) have lower thermal conductivity compared to metal matrix composites (MMCs) (Kingery and McQuarrie, 1954). When comparing the reinforcement materials in the MMCs, using $MoSi_2$ increases the thermal conductivity compared to the composites that use SiC. However, $MoSi_2$ does not improve the thermal conductivity of the Cu matrix (385 W/mK) (Davis, 2001), but it does improve the CTE (CTE_{Cu} = 1.7 × 10^{-5}/°C) (Davis, 2001). On the other hand, the coefficient of thermal expansion is lower in CMCs compared to MMCs, which can be detrimental for thermal dissipation applications in electronic components; so, amongst MMCs, Al/SiC composites have better thermal properties compared to those that are reinforced with $MoSi_2$.

Within the mechanical properties of MMC reinforced with $MoSi_2$, the most reported in scientific articles are Young's modulus, tensile strength, and modulus of rupture. Properties such as hardness, elongation, and compressive strength are also mentioned, but to a lesser extent. When making a comparison of the mechanical properties between composites (Table 7), CMCs are the ones with the highest Young's modulus (E = 479 GPa) and modulus of rupture (MOR = 332 MPa) compared to MMC (E = 235 GPa and MOR = 298 MPa), however, when comparing the reinforcement materials used in the MMC, the SiC particles have the highest values of Young's modulus (E = 235 GPa), tensile strength (UTS = 288 MPa) and modulus of rupture (MOR = 369 MPa) compared to the intermetallic $MoSi_2$ (UTS = 226 MPa and MOR = 50.5 MPa).

4 Potential applications

At the end of the 1980s, the feasibility of manufacturing metallic or ceramic matrix composites reinforced with intermetallic particles or fibers was studied (Fitzer and Remmele, 1985; Gac and Petrovic, 1985). Intermetallic $MoSi_2$ was of great interest due to its chemical, mechanical, and thermal properties, as well as being chemically stable with the matrix of the composite materials (Maxwell, 1945; Schlichting, 1978). The structural applications of $MoSi_2$ as a single-phase material have not been relevant due to its relatively low thermal stability at temperatures higher than 1400°C (Petrovic, 1992; Zumdick et al., 2001). However, most studies published in the last three decades have only focused on the manufacturing and the properties or characteristics offered by composites reinforced with $MoSi_2$, but with limited highlighting of their potential application. In comparison, metal matrix composites reinforced with ceramic particles or fibers such as Al_2O_3 (Rao and Jayaram,

TABLE 5 Thermal and mechanical properties of composite materials with reinforcement and unreinforced for electronic packaging. Reproduced and updated from (Minges, 1989).

Reinforcement	Matrix	Thermal conductivity (κ) [W/m·K]	Coefficient of thermal expansion (CTE, α_l) [$\times 10^{-6}$/°C]	Modulus of elasticity (E) [GPa]	Ref
Unreinforced	Silicon	150	4.10	-	Minges (1989)
	Aluminum	120	23.0	69	
	Alumina	20	6.70	380	
Copper	Copper	400	17.0	117	
	Tungsten	167	6.50	248	
	Molybdenum	184	7.00	282	
	Aluminum	290	6.50	131	
Discontinuous Carbon Fibres	Aluminum	185	6.00	14	
SiC Particles	Aluminum	170–220	6.2–7.3	225–265	
MoSi2 Particles	Copper	265	3.4	-	Yi et al. (2007)
	SiC particles	-	6.4	410	Khanra et al. (2012)

TABLE 6 Comparative table of thermal properties of different composites.

Composite	Thermal conductivity (κ) (W/m·K)	Coefficient of thermal expansion (CTE, α_l) ($\times 10^{-6}$/°C)	Description	Ref
Cu-2%$MoSi_2$	265	3.4 at 100°C	Cu-2%$MoSi_2$ composite manufactured by powder metallurgy	Yi et al. (2007)
Al-SiC	150	-	Al-SiC composite manufactured by powder metallurgy, sintered at 550°C	Abdul-Ameer (2013)
Al-30%SiC	140,160 (annealed)	15	Al-30%SiC composite manufactured by the kinetic spray deposition method	Eesley et al. (2003)
Al-20%SiC	61.5	15	Al-20%SiC composite manufactured by hot pressure molding	Zare et al. (2019)
ZrB_2-10%SiC-10%$MoSi_2$	53	6.9	ZrB_2-10%SiC-10%$MoSi_2$ composite sintered at 1000°C by vacuum hot pressing method	Venkateswaran et al. (2015)
TiC-Mo_2C-SiC-$MoSi_2$	-	4.64	TiC-Mo_2C-SiC-$MoSi_2$ composite manufactured from the solid-state reaction between $MoSi_2$, graphite and TiC at 1560°C	Liu and Li (2011)
Al-SiC-RHA	-	8.9	Al-SiC-RHA composite manufactured by the pressureless infiltration method	Bahrami et al. (2017)

2001; Zhang et al., 2014) or SiC (Cui, 2007; Leng et al., 2008; Yang and Xi, 2011; Patel et al., 2012; Prosviryakov, 2015) have been extensively studied and reported in different scientific publications, in addition to having a potential application for industries such as the automotive (Allison and Cole, 1993), aerospace (Kainer, 2006b), or electronic (Geffroy et al., 2008; Lu and Wong, 2009; Tong, 2011) industries. It was not until the early 2000s that some applications began to be found for $MoSi_2$-based composites (Cemal et al., 2012), specifically for high-temperature heating elements that may be in corrosive or oxidizing environments. The following paragraphs describe some research works where $MoSi_2$-reinforced composites have a potential application.

Various ceramics and some metallic materials have been used for decades to produce heating elements ranging from graphite (Taylor et al., 1991; Chung, 2002), silicon carbide (Pelissier et al., 1998), or stainless steel (Fang et al., 2020), to name a few. In recent years, $MoSi_2$ has been widely used in producing high-temperature heating elements due to its superior thermal properties compared to the materials mentioned above (Zhang et al., 2019). Wick-Joliat et al. (2021) described the production of high-temperature heating elements using ceramic injection molding (CIM). The sintered product was obtained by embedding $MoSi_2$ particles in a matrix of alumina and vitrified feldspar. The conductivity of the heating element was 1662–1985 S/m when using 17% by volume of $MoSi_2$. According to Wick-Joliat et al. (2021) the conductivity of sintered products can be adjusted by varying the content of the conductive phase, in this case $MoSi_2$.

Intermetallic $MoSi_2$ has also been used as a reinforcement material to manufacture ultra-high-temperature ceramics (UHTC) that can resist temperatures greater than 1800°C since it can increase its oxidation resistance when reaching 1000°C (Zhang et al., 2019). Silvestroni and Sciti (2013) developed a reinforced composite with 15 vol% $MoSi_2$ using different borides and carbides of Zr, Ha, and Ta as matrices. The hot-pressing process was used for manufacturing the composite. Carbide composites attained full density when reaching a temperature range of 1700°C–1900°C, in contrast to boride composites, which reached total density when reaching 1800°C–1900°C. As for the mechanical properties, there was no significant change; however, the composites exhibited excellent resistance in oxidizing environments.

Köbel et al. (2004) fabricated an Al_2O_3-$MoSi_2$ electroconductive ceramic composite by cold isostatic pressing and vacuum sintering. The composites have potential applications in gas turbine engines. Two volume fractions of $MoSi_2$ were used: 16% and 40%. It was concluded that the addition of $MoSi_2$ slightly influences the densification of the composite material, being 98 and 94% when having 16 and 40 vol% intermetallics, respectively. Composites containing ≥20 vol% of $MoSi_2$ were shown to be electroconductive due to the formation of a three-dimensional percolation network of the conductive phase of $MoSi_2$.

Khanra et al. (2012) have developed a $MoSi_2$-20vol.%SiC component with a potential application for a satellite launch vehicle. The composite was made through the mixture by mechanical milling of Mo, Si, and SiC powders and consolidated by vacuum hot pressing. Beneficial mechanical (E = 410 GPa, MOR = 338 MPa) and thermal (CTE = 6.4×10^{-6}°C^{-1}) properties were obtained, compared with unreinforced $MoSi_2$ (E = 395 GPa, MOR = 257 MPa, CTE = 7.79×10^{-6}°C^{-1}), due to the good bonding amongst the constituent

TABLE 7 Comparative table of mechanical properties of different composites.

Composite	Young's modulus (*E*) (GPa)	Tensile strength (UTS) (MPa)	Modulus of rupture (MOR) (MPa)	Description	Ref
ZrB_2-10%SiC-10%$MoSi_2$	479	-	332	ZrB_2-10%SiC-10%$MoSi_2$ composite sintered at 1000°C by vacuum hot pressing method	Venkateswaran et al. (2015)
Al-SiC	235	-	298	Al-SiC composite manufactured by the pressureless infiltration method	Pech-Canul et al. (2000b)
Al-SiC-RHA	170	-	369	Al-SiC-RHA composite manufactured by the pressureless infiltration method	Bahrami et al. (2017)
Al-5%SiC	-	288	-	Al-5%SiC composite manufactured by the accumulated roll bonding process	Meselhy and Reda (2019)
Cu-2%$MoSi_2$	-	226	-	Cu-2%$MoSi_2$ composite manufactured by powder metallurgy	Yi et al. (2007)
65%TiC-Mo_2C-SiC-$MoSi_2$	-	-	50.5	65%TiC-Mo_2C-SiC-$MoSi_2$ composite manufactured from the solid-state reaction between $MoSi_2$, graphite and TiC at 1560°C	Liu and Li (2011)

materials and the uniform distribution of the SiC particles within the matrix. As components designed for oxidation environments and high temperatures, the composites showed excellent oxidation resistance at a temperature of 1500°C.

Recently, intense efforts have been made to develop composite materials with a sustainable approach, where waste can be reused or recycled. In 2019, an attempt was made to use $MoSi_2$ scrap from heating element rods, which, after a grinding process, were used as raw material in $MoSi_2$-SiC coatings, which were applied as a protective coating on graphite substrates. The coated substrates through the spark plasma sintering (SPS) process were subjected to a heat treatment of 1400°C for 90 h. It was observed that a dense film of SiO_2 formed on the surface, which prevented oxygen diffusion into the system (Chen et al., 2019).

Developing materials that can withstand extreme environments has been a priority within aerospace technologies. As in the case of existing combustion chambers that have exceeded the maximum working temperature in most superalloys (Zhu et al., 2022a), composite materials that use refractory metals have been excellent options for designing new components. Zhu et al. (2021a); Zhu et al. (2021b) recycled $MoSi_2$-based materials to be used as raw material to manufacture a protective coating of $MoSi_2$-Ta_2O_5 and $MoSi_2$-HfO_2, with which niobium substrates would be protected using the SPS process. Results showed the formation of a Ta-Si-O and $HfSiO_4$ layer in addition to low oxygen permeability in the composite coating after heating at 1500°C for 20 h. It was also shown that the $MoSi_2$-Ta_2O_5 and $MoSi_2$-HfO_2 composite coating exhibited a good metallurgical combination with the niobium substrate, which makes it a potential candidate to be applied in aerospace components due to its resistance to oxidation and high temperatures (T = 1500°C).

$MoSi_2$-based composites have also been proposed to be applied in electronic packaging components, specifically in heat sinks that can be used in laptops, televisions, tablets, etc.; this is because of the frequent problems that these devices have derived from heat (Tapia-López et al., 2021). Therefore, an alternative to this problem is using MMC materials that are good thermal conductors, have a low coefficient of thermal expansion, and are lightweight materials. Aluminum matrix composites reinforced with $MoSi_2$ particles are ideal candidates that meet these requirements (Chung, 1995; Lee, 1995; Blackwell, 2000).

In 1989 Kathryn A. Schmidt and Carl Zweben presented a book chapter in the *Electronic Materials Handbook* (Schmidt et al., 1989) on composite materials focused on electronic packaging. The chapter focuses on the aspects of thermal management in electronic packaging. It is explained that the materials used in electronic packaging must have similar CTEs; they should coincide with those of the materials to be bonded, especially ceramic and semiconductor substrates since they are very fragile. Composite materials are the solution to this problem as in addition to being low-density materials they can be applied in portable devices, the primary application trend (Zweben, 2001a; Zweben, 2001b; Miracle et al., 2001). Composites with low CTE and high thermal conductivity include silicon carbide particle-reinforced aluminum (Al/SiC_p), carbon fiber-reinforced copper (Cu/C_f), carbon fiber-reinforced aluminum (Al/C_f), diamond particle-reinforced copper ($Cu/Diamond_p$), and beryllia particle-reinforced beryllium (Be/BeO_p) (Zweben, 1998).

Metal matrix composites (MMCs) offer better characteristics for the controlled elimination of heat, which is convenient because when joining different pieces by brazing or soldering, they can have high thermal impedances (Wang et al., 2019). Hence it is important for MMCs to be used as heat sinks incorporating thin cooling fins and/or fluid cooling ducts (Zweben, 1988; Zweben, 1998). $MoSi_2$-based composites have come to be used in components for electronic packaging, where it is required to dissipate heat quickly (Fackler and Kutz, 2002; Lu and Wong, 2009; Frear et al., 2017). As can be seen, the reinforcement of composites has a role in better highlighting the metallic matrix properties (Shen et al., 1994; Mitra and Mahajan, 1995). In the case of an Al/SiC_p composite, a volume fraction of the carbide particles in the range of 0.7 makes it feasible to achieve CTE values like that of silicon (Pech-Canul and Makhlouf, 2000). Currently, investigations on the CTE of $Al/MoSi_2$ composites are very limited. However, based on the $MoSi_2$ and Al volume fraction and their corresponding CTE values (CTE $MoSi_2 = 7 \times 10^{-6}$/K; CTE Al = 23×10^{-6}/K), the rule of mixtures allows for estimating the CTE. For a 0.7 vol% $MoSi_2$ composite, the CTE value tends to equate with that of ductile iron and cobalt-based superalloys (Callister, 2007). It is generally accepted that MMCs are low-density materials that are relatively inexpensive and easy to produce.

Petrovic and Vasudévan (1992) have proposed a promising future for developing $MoSi_2$-based composite materials specifically for applications that require high temperatures and good oxidation resistance. $MoSi_2$-based composites will require both ceramic [extrusion (Thom et al., 2002)] and metal [powder metallurgy (Corrochano et al., 2009; Corrochano et al., 2010) and mechanical alloying (Koch, 1998)] processing techniques as they require significant volume fractions of ceramic reinforcement for their development. The future of these materials also lies in improving fracture toughness at low temperatures since industrial applications require these values, as in the case of turbines (Petrovic, 2000).

According to a report by Research Reports World (2028) published on the web portal "The Express Wire", there is an optimistic perspective on the current and future scene of using $MoSi_2$ heating elements. This conclusion is drawn from an analysis of the status of intermetallic materials ($MoSi_2$, Mo_5Si_3, WSi_2) in industrialized countries and companies engaged in producing such heating components. The report details that the global market size for $MoSi_2$ heating elements was estimated to be USD 112.50 million in 2021 and is projected to reach USD 160.40 million by 2028, with a compound annual growth rate (CAGR) of 5.21% in the period covered. The leading distributors of heating elements are concentrated in China, South Korea, and the United States. So, it is expected that in addition to increasing the production of $MoSi_2$ heating elements, there will also be an augmentation of the disposal of such components, which creates an excellent opportunity for the development of composite materials using recycled $MoSi_2$ (Chen et al., 2019).

The present review shows that, albeit with some significant advances that have been made in the processing and characterization, the development of $MoSi_2$-containing composites is an open-ended area with a broad scope for research. The first and foremost investigations were focused on their thermal and oxidation-resistance-related applications. In addition to the former, more recently, the investigations have turned the spotlight on their functional properties, oriented to semiconductor, micro-, and optoelectronic applications. Future systematic studies can be categorized into.

1. The role of $MoSi_2$ as a matrix or reinforcement/thermal/functional phase,
2. Matrix type (metal-, ceramic-, intermetallic-matrix composites),
3. Scale or size of the involved phases (micro-, nano-composites), emphasizing the size role of $MoSi_2$,
4. Processing or fabrication routes (solid, liquid, and vapor), and
5. Uses or applications.

When designing $MoSi_2$-containing composites, one critical aspect is the chemical compatibility of $MoSi_2$ with the other phase(s) Table 4 shows the compatibility-related behavior of some reinforcement phases with $MoSi_2$. Some typical composites reported in the literature are those of ZrB_2-10%SiC-10%$MoSi_2$ (Venkateswaran et al., 2015), TiC-Mo_2C-SiC-$MoSi_2$ (Liu and Li, 2011), and Cu-2%$MoSi_2$ (Yi et al., 2007) composites. Because they are promising, during about the last 15–20 years, special attention has been paid to UHTC (ultra-high temperature ceramic) matrix composites, particularly to ZrB_2–$MoSi_2$ composites, with significant findings (Sciti et al., 2005; Sciti et al., 2006a; Sciti et al., 2006b; Silvestroni and Sciti, 2013; Silvestroni et al., 2018; Grohsmeyer et al., 2019). Furthermore, although $MoSi_2$ has some drawbacks related to its brittleness and harmful oxidation at low temperatures (limiting them for high-temperature structural applications), its global manufacture and use increment are forecast to increase for at least the next 5 years, suggesting that research efforts will steadily grow accordingly. Some detected research areas for future works include 1) mechanical property-related investigations of $MoSi_2$-based composites, particularly those on elongation characteristics. Of course, this goes hand-in-hand with the matrix nature, as in the case of ceramic- and intermetallic-matrix $MoSi_2$ composites, test type, configuration, and specimen size will play significant roles. 2) Another suggested research attention is the determination of the coefficient of thermal expansion (CTE) of some materials, as in the case of $Al/MoSi_2$ composites (Gousia et al., 2016). Inspired by an environmentally friendly materials research philosophy, one key aspect predicated in this review is the exploitation of recycled/reused $MoSi_2$ through designing and manufacturing composites containing this intermetallic. On that score, some investigation results will be reported elsewhere.

5 Conclusion and perspectives

The properties of the intermetallic compound $MoSi_2$ make it a strong candidate for applications where high temperatures and resistance in corrosive environments are paramount. Due to its chemical compatibility with aluminum, copper, SiC, and Al_2O_3, among other materials, it has been used for developing composite materials either as a matrix or reinforcing/thermal/functional phase. Different processing routes can be used to manufacture metal-matrix composites with $MoSi_2$ as a constituent phase. However, the solid and liquid state methods are preferred for their

simplicity and low cost. $MoSi_2$ composites with good mechanical (E = 276.77 GPa) and thermal (κ = 265 W/mK) properties can be attained for applications in turbines, heat sinks, or as a coating for CMC, using the solid- and liquid-state processing routes. Intense research is being conducted to diminish its size to the nanometer scale and synthesize nanocomposites by introducing second phases (e.g., Ta, Nb, HfO_2, ZrO_2, SiC, and Mo_5Si_3) to overcome the low toughness and low creep resistance of conventional microcrystalline $MoSi_2$. It is predicted that in the next 5 years, there will be an increase in the production of $MoSi_2$ heating elements, in addition to an augmentation in the scope of the global market. So, it is expected that there will be an increase in the waste of $MoSi_2$ heating elements, making it suitable to develop composites or hybrid composites with recycled $MoSi_2$. Due to the continuous growth of the microelectronics and optoelectronics industries, it can be anticipated that the applications of $MoSi_2$ in microelectronic components or circuits as a contact material will spread rapidly in the forthcoming years.

Author contributions

All authors listed have made a substantial, direct, and intellectual contribution to the work and approved it for publication.

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Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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