

Combustion Synthesis of Molybdenum Disilicide (MoSi₂) Fine Powders

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The combustion characteristics of a MoO_3+SiO_2+Mg system diluted with sodium chloride have been studied in order to synthesize molybdenum disilicide (MoSi₂) fine powders. The effect of processing variables, including the sodium chloride content and the reactants proportion on the phase composition and microstructure of the final products, was obtained. Both the combustion temperature and the wave velocity decreased with an increase in the amount of sodium chloride. With the appropriate processing parameters, the reacted product consisted of solid crystalline $MoSi_2$, and an MgO phase was prepared in the molten sodium halide matrix at $1100^\circ-1350^\circ$ C. The final product purification resulted in $MoSi_2$ powder with a particle size of 100-500 nm. The mechanism of the $MoSi_2$ formation in the sodium chloride matrix is discussed by analyzing the phase evaluation in the extinguished combustion wave.

I. Introduction

THE properties of molybdenum disilicide (MoSi₂) have been established, including a high melting point (2030°C), excellent high-temperature oxidation resistance, and high electrical and thermal conductivity. Because of its superior property combination, MoSi₂ has received considerable research attention in recent years as a potential candidate material for high-temperature structural applications.¹ A number of studies have been attempted to prepare MoSi₂ powder, using many processing routes including solid-state reaction,² spray forming,³ mechanical alloying,⁴ and self-propagating high-temperature synthesis.^{5,6} However, like other intermetallics, conventional coarsegrained MoSi₂ is brittle at normal environmental temperatures.¹ Refinement of the grain sizes to nanometer dimensions is predicted^{7,8} to improve the ductility, the fracture toughness, and the strength of intermetallics by inducing fundamental changes in the strengthening and deformation mechanisms.9 Before the present work, nanocrystalline MoSi2 was prepared using the sonochemical synthesis method.¹⁰ In the sonochemical method co-reduction of MoCl5 and SiCl4 with NaK alloys in hexane was carried out using 600 W, 20 kHz irradiation. The precipitate was annealed at 900°C, and nanocrystalline MoSi₂ with a particle size 16-31 nm was obtained. Generally, the sonochemical method is limited in scale up, and only small amounts (a few grams) are typically produced by this method. One of the routes to prepare nanocrystalline $MoSi_2$ powder is the mechanical alloying of silicon and molybdenum.¹¹ In this method α -MoSi₂ powder was obtained after 50 h of milling time. When the milling time was further increased to 96 h, a complete transformation of α -MoSi₂ to β -MoSi₂ was found. The authors concluded that the phase-transformation phenomenon is determined by the crystallite size of α -MoSi₂. When the size of previously formed α -MoSi₂ decreases to a certain critical particle dimension (approximately 8 nm), it transforms to the β -MoSi₂.

The current work studies the effects of the sodium chloride addition on the combustion synthesis characteristics and $MoSi_2$ formation from the MoO_3+SiO_2+Mg system.

II. Experimental Procedure

MoO₃, SiO₂, Mg, and NaCl reactant powders were used, and their specifications are shown in Table I. These powders were first weighed and then thoroughly mixed by ball-milling for at least 12 h. The mixed powder (150–200 g) was hand pressed into a metallic cup 5 cm in diameter and 10 cm in height. The experimental density of the pellets was $1-1.5 \text{ g/cm}^3$. The Ti+C powder, about 3 g, was placed at the top of the raw material mixture as an ignition agent.

The combustion synthesis reactions are:



Here, α is the mole number of NaCl, and its content in all the samples of the current study was 5–14 mol. β shows the mole fraction of SiO₂, and it was varied in the $2 \le \beta \le 3$ interval. The change in β affects the Mg content, because SiO₂ and Mg are balanced with a 1:2 ratio. $\beta = 2$ represents the stoichiometric composition for MoSi₂ synthesis. However, along with the MoSi₂ phase, a sufficient amount of Mo₅Si₃ (10-20 wt%) resulted also by the combustion reaction at $\beta = 2$. By including an additional portion of SiO₂+2Mg mixture in the initial system, $(\beta = 3)$ both the silicon concentration and the reactivity of the process increased, which led to the elimination of Mo₅Si₃ from the reaction product. Also, when $\beta = 3$, a defined amount of unreacted silicon was detected in the final products. After the experiments, the reaction products were subjected to a hydrochloric acid treatment and were washed by distillated water to eliminate MgO and NaCl. Free silicon containing the reaction products were additionally treated by a 10% solution of NaOH.

The details of the experimental setup are shown in Fig. 1. Briefly, the setup included a combustion chamber and an automated ignition and data acquisition system. The metallic cup with a green mixture was loaded into the combustion chamber by placing it directly on top of a nickel-chromium coil. Ignition was achieved by using power input into the nickel-chromium coil in a high-purity argon atmosphere of 2.0 MPa. The data acquisition system (DASTC, Keithley) continuously recorded the thermocouples' time histories. Two tungsten-rhenium thermocouples (W/Re-5 vs W/Re-20, 50 and 100 µm in diameter), coated by a thin layer of Al₂O₃, were used for temperature measurements. The combustion parameters examined were the combustion temperature (T_c) and the combustion wave propagation velocity (U_c) . The combustion velocity was determined from the temperature profiles and the known spacing between the thermocouples.

The final products were characterized by using a Cu $K\alpha$, Siemens D5000 X-ray diffractometer (Siemens, Karlsruhe, Germany). Scanning electron microscopy (SEM; JSM 5410, JEOL, Tokyo, Japan) was used to examine the morphology, shape, and

B. Derby-contributing editor

Manuscript No. 24122. Received December 19, 2007; approved May 14, 2008. [†]Author to whom correspondence should be addressed. e-mail: haykrasom@ hotmail.com

Table I. Reactant Properties

| Reactant | Manufacturer | Particle size (µm) | Purity (wt%) |
|---------------------|---|-----------------------|--------------|
| Silicon | Junsei Chemical Co. | <10.0 | >99.0 |
| Magnesium | Daejung Chem. and Metals Korea | 50-200 | 98.5 |
| Molybdenum oxide | Yakuri Pure Chemical | <1.0 | >99.0 |
| Sodium chloride | Co. Ltd, Japan Samchun Pure Chemical Co. Ltd, | <100 | 99.5 |
| | Korea | | |

size of the synthesized MoSi₂ powder. The specific surface area and the oxygen concentration measurements were performed using "Coulter[™] SA3100 Surface Area Instrument" (Beckman Coulter Inc., Fullerton, CA) and the oxygen analysis technique.

III. Results and Discussion

(1) The Combustion of the $MoO_3+2SiO_2+7Mg+\alpha NaCl$ System

The behavior of the NaCl-assisted combustion reactions is different, essentially, from those of the traditional combustion process. In our recent publications, the preparation of the nanocrystalline powders of transition metals (Ti, W, Ta) and appropriate carbides (TiC, WC) were reported using a sodium halide-assisted combustion reaction.^{12–14} The effect of sodium chloride on the combustion characteristics of the MoO₃+ $2SiO_2+7Mg+\alpha NaCl$ system was investigated for the first time.

Before the experiments, the MoO₃+2SiO₂+7Mg+ α NaCl system was analyzed (in a preliminary fashion) by a commercially available thermodynamic program called "THERMO" that is specially designed for combustion processes.¹⁵ The program calculates the equilibrium composition, the phase, and the adiabatic combustion temperature for multi-component systems at a set of defined temperature and/or pressure conditions. Figure 2 shows the equilibrium phases and the corresponding adiabatic combustion temperatures (T_{ad}). The initial temperatures of the reagents and the reaction pressure were set to be 25°C and 2.0 MPa, respectively. As shown in Fig. 2, the adiabatic combustion temperature of the MoO₃+2SiO₂+7Mg ternary system ($\alpha = 0$) is predicted to be 2617°C. After adding NaCl, the heat released per unit volume decreased. The reaction heat is partially consumed



Fig. 1. Schematic representation of the experimental apparatus.



Fig. 2. Thermodynamic analysis of the $MoO_3+2SiO_2+7Mg+\alpha NaCl$ system (P = 2.0 MPa).

by the heating, melting, and evaporation of NaCl, therefore, the adiabatic combustion temperature drops from α . Also, Fig. 2 shows that at $\alpha < 4$ the combustion temperatures are not only higher than the melting points of most raw materials, but also higher than the melting point of MoSi₂ ($T_m = 2030^{\circ}$ C). At that multiphase reaction, products consisting of Mo₅Si₃, Mg₂SiO₄, SiO, Mg, MgO, and NaCl phases have been predicted at $\alpha < 4$. When more than 6 mol NaCl is added, the formation of MoSi₂ phase with MgO and NaCl become thermodynamically favored.

A series of the combustion experiments was conducted in the $MoO_3+2SiO_2+7Mg+\alpha NaCl$ system to reveal the effect of adding NaCl on the phase purity and morphology of MoSi₂. X-ray diffraction (XRD) patterns of purified combustion products prepared from 5, 7, and 10 mol NaCl containing green mixtures and the corresponding values of experimentally measured combustion temperatures are shown in Fig. 3. All of the major XRD peaks were found to match with standard peaks of MoSi₂. However, the XRD patterns of the final products show a visible amount of Mo₅Si₃, also, regardless of the salt concentration and the combustion temperature.

The SEM micrographs of combustion products derived with 5, 7, and 10 mol NaCl and leached by acid are shown in Fig. 4. As seen by comparing Figs 4(a), (b), and (c), the product morphology is influenced significantly by the concentration of salt. As a result, the MoSi₂ powder obtained from 10 mol NaCl (containing mixture) has smaller characteristic size dimensions compared with the MoSi₂ powders obtained from 5 and 7 mol



Fig. 3. X-ray diffraction patterns of the MoSi₂ powders produced from the MoO₃+2SiO₂+7Mg+ α NaCl mixture: (a) $\alpha = 5$; (b) $\alpha = 7$; (c) $\alpha = 10$.



Fig. 4. Scanning electron microscopy images of the MoSi₂ powders produced from the MoO₃+2SiO₂+7Mg+ α NaCl mixture: (a) α = 5; (b) α = 7; (c) α = 10.

NaCl (containing mixtures). The fine structure of $MoSi_2$ shown in Fig. 4(c) might be attributed more to the combustion temperature, which is relatively low (1300°C), than to the melting point of silicon (1410°C).

Mo₅Si₃, as a by product phase, has been reported to be present, in a small amount, in powder processed MoSi2.16-18 In our system, a minor content of Mo₅Si₃ phase in the final products can be attributed to the initial system heterogeneity. Consequently, two approaches to eliminate the residual Mo₅Si₃ from the final product were tested. First, we used small particle size SiO₂ and Mg powders in the combustion experiments and, second, we increased the content of SiO₂ and Mg in the initial mixture. Generally, no positive change in the Mo₅Si₃ content was found when small size SiO₂ (<1 μ m) and Mg (~50 μ m) powders were used in the combustion experiments. The second approach, which was tested on the $MoO_3+(3+2\beta)Mg+$ β SiO₂+10NaCl initial system, was found to be effective to reduce the Mo₅Si₃ content in the final product. First, we examined the effect of β on the combustion parameters (T_c and U_c). The heat released per volume increased from β , therefore, both the combustion temperature and the wave velocity increased. These features are shown in Fig. 5. Also, Fig. 5 shows that the combustion temperatures are lower than the melting points of Mo, Si, Mo₅Si₃, and MoSi₂, but higher than the melting point of NaCl. The formation of a molten matrix of NaCl with solid



Fig. 5. The combustion parameters of the MoO₃+(3+2 β)Mg+ β SiO₂+10NaCl system versus β (P_{Ar} = 2.0 MPa).

particles of molybdenum silicides and MgO ($\beta = 3$) is clearly seen in the micrograph as shown in Fig. 6(a). Figure 6(b) shows the morphology of the final product particles after purification. It is noted that large size and corrugated form agglomerates had apparently formed due to chemical conversions, which occurred on the surface of the melted Mg particles. These agglomerates show porosity morphology and consist of smaller size particles (Fig. 6(b), upper right side). These agglomerates have low mechanical strength, and after the short time ball milling, transform to fine powder (Fig. 6(c)).

Figure 7 shows the XRD patterns of purified samples synthesized at different β levels. The major diffraction peaks are attributed to the MoSi₂ phase. Some minor peaks, detected at $\beta = 2$ and $\beta = 2.5$ (Figs 7(a) and (b)), were identified as the Mo₅Si₃. However, when β increased, the intensity of the Mo₅Si₃ peaks decreased and became invisible at $\beta = 3$ (Fig. 7(c)). Simultaneously, new minor peaks were found at $\beta = 3$ and identified as the Si. The formation of silicon is attributed to an excess of SiO₂, which was used in the combustion experiment. Free Si was easily dissolved into a 10% solution of NaOH, and phase pure MoSi₂ was obtained (Fig. 7(d)).

The effect of the NaCl concentration (α) on the combustion characteristics of the MoO₃+3SiO₂+9Mg+ α NaCl system and the morphology of MoSi₂ powder were also studied (Fig. 8). As can be seen, the combustion temperature drops from 1550° to 1100°C when α changes from 8 to 13.5. The diminishing character of the combustion process observed at $\alpha = 14$ had resulted in a partially combusted sample. Above this point an attenuation of the combustion occurred. Conditionally, the investigated interval of can be divided into two zones: (1) a high-temperature zone, in which the combustion temperature is higher than the silicon melting point (1410°C), and (2) a low temperature zone, in which the combustion temperature is lower than the silicon melting point. A passing from one temperature zone to the other took place at $\alpha = 9.5$, and the combustion wave velocity showed an abrupt increase during this transition (Fig. 8).

The average size and the specific surface area of MoSi₂ particles prepared by the NaCl-assisted combustion reaction ($\beta = 3$, $\alpha = 10-13$) were in the 100–500 nm and 25–30 m²/g ranges, respectively. The average concentration of oxygen in the MoSi₂ powder was about 1.0–1.5 wt%.





Fig. 6. The morphology of the final products ($\beta = 3$): (a) after the combustion; (b) after the purification; (c) ball-milled powder.

(2) Temperature Distributions and the Formation Mechanism of MoSi₂

The average temperature distributions over the combustion zones of the MoO₃+3SiO₂+9Mg+ α NaCl system are shown in Fig. 9. These distributions are started by a thermal layer (left side of ordinate) with an exponentially growing temperature. The initial reaction temperature (T^*) corresponds to the melting point of magnesium ($T^* = \sim 650$ °C). The extent *l* of the thermal layer is determined by the distance in which the temperature (starting with T^*) drops e times ($l = T^*/e$), and the extent *L* of the reaction zone is the distance between T^* and T_c . Generally, the reaction zones (*L*) are very wide in comparison with the heating zones (*l*) ($\Delta = L/l > l$), which means the domains governing the propagation velocity are near T^* ¹⁹ and have a size on the order of *l* (1.0–3.0 mm). The remaining part of the reaction zone (about 10–15 mm) is called the afterburning zone, where the postcombustion processes occur.

The character of temperature growth in the reaction zone (right side of ordinate) is attributed to the NaCl content. When 8 mol of NaCl is added (Fig. 9, curve 1), a smooth and sharp in-



Fig. 7. X-ray diffraction patterns of the $MoSi_2$ powder versus β : (a) 2.0; (b) 2.5; (c, d) 3.0.

crease in the temperature in the reaction zone is observed. At a higher concentration of NaCl (10-14 mol) temperature growth is retarded, starting with 700°C. This feature of the temperature is attributed to the consumption of reaction heat for the melting of Mg (650°C), MoO₃ (795°C), and NaCl (810°C). Note that the propagation of the combustion wave at $\alpha = 14$ was unstable (Fig. 9, curve 4) and, after passing about 4-5 cm distance, stopped at the middle part of the sample. Several zones were visually observed on the partially combusted sample. The average size of the individual zones was 2-4 mm, and we could separate them easily from each other and investigate the phase structures by X-ray analysis. A schematic of a longitudinal section of the partially combusted sample, with X-ray analysis data, is shown in Fig. 10. Using these data, the sequence of chemical reactions leading to the MoSi2 formation can be presented as follows:

Zone I (\sim 700°C): reduction of MoO₃ to MoO₂:

$$MoO_3 + Mg = MgO + MoO_2$$
(2)

Zone II (\sim 700°–850°C): reduction of MoO₂ to metallic Mo:

$$MoO_2 + 2Mg = 2MgO + Mo$$
(3)



Fig.8. The combustion parameters of the MoO₃+3Mg+9SiO₂+ α NaCl system versus α (P_{Ar} = 2.0 MPa).



Fig.9. The average temperature distributions over the combustion zones of the MoO₃+3SiO₂+9Mg+ α NaCl system: (1) $\alpha = 8$; (2) $\alpha = 10$; (3) $\alpha = 12$; (4) $\alpha = 14$.

Zone III ($\sim 900^{\circ}-950^{\circ}$ C): reduction of SiO₂ to Si and molybdenum silicides formation:

 $SiO_2 + 2Mg = Si + 2MgO$ (4)

$$Mo + 2Si = MoSi_2$$
⁽⁵⁾

$$5\mathrm{Mo} + 3\mathrm{Si} = \mathrm{Mo}_5\mathrm{Si}_3 \tag{6}$$

According to XRD analysis, the volume fraction of MoSi₂ in zone III is predominant, and then Mo, Si, and Mo₅Si₃ follow. This probably indicates that the main part of MoSi₂ forms by reaction (5).

Zone IV ($\sim 1000^{\circ}$ C): reaction (5) had been continued by converting the remaining part of Mo and Si to MoSi₂. The Mo₅Si₃ phase was also converted to MoSi₂ by reacting with Si:

$$Mo_5 + 7Si = 5MoSi_2 \tag{7}$$

The mechanism of MoSi₂ formation from Mo and Si by combustion reaction has been investigated by many researchers.²⁰⁻²² Deevi²⁰ reported that in the combustion process Mo₅Si₃



Fig. 10. Longitudinal section of a partially combusted sample with X-ray diffraction analysis results.

is formed first, which is then enriched with liquid silicon by the diffusional reaction of liquid silicon through the product layer of Mo₅Si₃, thus forming MoSi₂. Gao et al.,²¹ using a combustion front quenching technique, reported that the formation mechanism of MoSi₂ during the combustion reaction of the Mo+2Si mixture was neither the reactive diffusion mechanism exclusively nor the dissolution-precipitation mechanism exclusively. Cabouro et al.,22 using time-resolved XRD coupled to IR thermography, have observed a secondary formation of Mo₅Si₃ at the surface of the molybdenum, due to an exothermic diffusional reaction between solid silicon and solid molybdenum after 500°C. Then Mo₅Si₃, intermediate phase, and Mo were dissolved into Si melt, forming MoSi₂ particles.

The distinguishing feature of the system investigated here is the formation of MoSi₂ below the Si melting point. As reported in the literature, the reactive diffusion and dissolution-precipitation mechanisms of the MoSi2 formation are based on liquid Si. Consequently, a reasonable assumption that could explain the MoSi₂ formation below the Si melting point might be attributed to molten NaCl. The NaCl melt makes it easier for Mo and Si particles to slide and rotate, provides chances of particleparticle contacts, and accelerates Si(solid)-Mo(solid) and Si(solid)-Mo₅Si₃(solid) diffusion reactions to form MoSi₂.

Therefore, a detailed mechanism for the salt-activated combustion synthesis of MoSi₂, under the Si melting point, is the material for future studies.

IV. Conclusion

The effects of adding NaCl to the combustion characteristics and the MoSi₂ formation of the MoO₃+SiO₂+Mg system were studied using the combustion synthesis technique. The SiO₂ acts thermodynamically as a diluent to the combustion synthesis reaction, thereby reducing both the combustion temperature and the wave velocity. With appropriate processing parameters, the reacted product, consisting of solid crystalline MoSi₂, MgO phases and the molten sodium halide matrix, was prepared at 1000°–1350°C. The average size and the specific surface area of MoSi2 particles prepared by the NaCl-assisted combustion reaction ($\beta = 3$, $\alpha = 10-13$) were in the 100–500 nm and 25–30 m²/g ranges, respectively. The average concentration of oxygen in the MoSi₂ powder was about 1.0–1.5 wt%.

Acknowledgement

The authors acknowledge Prof. Joe K. Ballenger from Stephen F. Austin State University, Nacogdoches, TX, USA for his valuable advice and assistance with English grammar.

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