Chemical Engineering Journal 143 (2008) 331–336

Contents lists available at ScienceDirect

Chemical Engineering Journal

journal homepage: www.elsevier.com/locate/cej

Molten salt-assisted combustion synthesis and characterization of $MoSi_2$ and $MoSi_2$-Si_3N_4 composite powders

Kh.V. Manukyan[a,b,*], S.V. Aydinyan[a], Kh.G. Kirakosyan[a], S.L. Kharatyan[a,b], G. Blugan[c], U. Müller[d], J. Kuebler[c]

[a] *Department of Inorganic Chemistry, Yerevan State University, A. Manukyan 1, Yerevan AM-0025, Armenia*
[b] *Laboratory of Kinetics of SHS Processes, A.B. Nalbandyan Institute of Chemical Physics NAS of Armenia, P. Sevak 5/2, Yerevan AM-0014, Armenia*
[c] *Laboratory of High Performance Ceramics, Empa, Swiss Federal Laboratories of Materials Testing and Research, Ueberlandstrasse 129, Duebendorf CH-8600, Switzerland*
[d] *Laboratory of Nanoscale Materials Science, Empa, Swiss Federal Laboratories of Materials Testing and Research, Ueberlandstrasse 129, Duebendorf CH-8600, Switzerland*

ARTICLE INFO

Article history:
Received 31 January 2008
Received in revised form 30 May 2008
Accepted 16 June 2008

Keywords:
Molybdenum disilicide
$MoSi_2$–Si_3N_4 composite
Self-propagating high-temperature synthesis (SHS)
Hot pressing

ABSTRACT

The possibilities of the synthesis of submicron $MoSi_2$ and $MoSi_2$–Si_3N_4 composite powders were investigated using inorganic salt-assisted combustion synthesis method. Combustion laws in the MoO_3–3Mg–2Si–NaCl and Mo–5Si–NaCl–Si_3N_4–N_2 systems were studied. The main factors influencing the combustion parameters, phase composition and microstructure of products for both these systems were determined experimentally. Optimum synthesis conditions of submicron $MoSi_2$ and $MoSi_2$–Si_3N_4 composite powders containing 30–40 wt.% of molybdenum disilicide were determined. Compacting conditions for 30 wt.% $MoSi_2$–70 wt.% Si_3N_4 composite using the hot pressing technique were found. Compact samples 20 mm in diameter were obtained. Microstructure, phase and chemical compositions of the dense samples were studied.

1. Introduction

Among the transition-metals' silicides, molybdenum disilicide ($MoSi_2$) is an attractive candidate for electronic, heating, and high-temperature structural applications [1–3]. Some of its characteristics include a high melting point of 2020 °C, stable resistance over long periods of time, self-healing ability due to the formation of a highly protective amorphous silica layer, high electrical conductivity ($2 \times 10^5\ \Omega^{-1}\ cm^{-1}$ at room temperature) and excellent oxidation resistance.

However, low fracture toughness at ambient temperature, steep decrease in strength and creep resistance at temperatures greater than 1000 °C, and inferior oxidation resistance at intermediate temperatures are some of the drawbacks that hinder the application of $MoSi_2$ as a fully viable structural material. Different approaches were used to overcome these problems, the composites route being prominent among these [4–7]. Various composites of $MoSi_2$ were developed to improve the low-temperature ductility and the high-temperature creep resistance, without any significant reduction in the oxidation resistance of $MoSi_2$. Among them, $MoSi_2$–Si_3N_4 composites are of special interest. These materials possess numerous desirable physical and mechanical properties [8–14]. First, at low temperatures, these composites have higher fracture toughness than either constituent phase alone [8,9]. Second, at elevated temperatures (e.g. >1000 °C), $MoSi_2$ is ductile, and as such, the addition of $MoSi_2$ can improve the fracture toughness of the Si_3N_4 through ductile phase toughening mechanisms. Conversely, at these temperatures the addition of Si_3N_4 may impart strength to the $MoSi_2$. Third, both constituent phases are thermodynamically compatible and possess similar oxidation mechanisms.

The technological importance of $MoSi_2$ for various applications has generated renewed interest in the manufacturing and processing of pure $MoSi_2$, and its composites. Conventionally, molybdenum disilicide is produced by using arc melting, mechanical alloying and shock synthesis methods [15–18]. In the last two decades the method known as self-propagating high-temperature synthesis (SHS), or combustion synthesis [19–25] was recognized as a promising one to produce $MoSi_2$ powder. Combustion synthesis, when compared to traditional processing techniques has the advantages of being extremely time- and energy-efficient. These advantages come from a highly exothermic chemical reaction becoming self-sustaining after only a short energy pulse initiates the reaction. This technique was also successfully applied for one-step synthesizing $MoSi_2$–Si_3N_4 composite powders [26,27]. In

---

[*] Corresponding author at: Department of Inorganic Chemistry, Yerevan State University, A. Manukyan 1, Yerevan AM-0025, Armenia. Tel.: +374 10 281610; fax: +374 10 281634.
E-mail address: khachat@ichph.sci.am (Kh.V. Manukyan).

1385-8947/$ – see front matter © 2008 Elsevier B.V. All rights reserved.
doi:10.1016/j.cej.2008.06.013

particular, studies on combustion process in the Mo–Si–N_2 system have shown that phase composition of the final product depends on Si/Mo ratio and nitrogen pressure. The approach developed allowed obtaining composite powders with high content of molybdenum disilicide reaching 60–90 wt.% [26].

However, in all the cases discussed above the products obtained by combustion reaction had relatively large particle sizes (10–20 μm) caused by the high reaction temperatures. To produce nanostructured molybdenum disilicide powders a new synthesis route coupling high-energy ball milling with self-sustaining reactions has been proposed [28–30].

Over the past few years molten salt-assisted SHS method of synthesizing nanostructured ceramic powders was extensively employed [31–33]. It is based on the use of various inorganic salt additives (e.g. NaCl, KCl) as inert diluents to control the combustion temperature. Alkali metal halide aids in transportation of the reactant species and prevents products' grain growth by forming a protective layer around the particles. All these facilitate synthesis at low temperature, and positively affects to the formation of fine grain products.

The present work aims to study the possibility of salt-assisted combustion synthesis of fine $MoSi_2$ and $MoSi_2$–Si_3N_4 composite powders and their characterization. Combustion laws in the MoO_3–3Mg–2Si–diluent and Mo–5Si–diluent–N_2 systems were investigated. Hot pressing technique was applied to compact the 30 wt.% $MoSi_2$–70% Si_3N_4 composite powder and some characteristics of as-derived compact samples were studied.

2. Experimental

2.1. Combustion synthesis and characterization of products

All experiments were conducted in a laboratory constant-pressure reactor (CPR-3l) using argon or nitrogen (purity 99.7%, oxygen content no more than 0.2%) atmosphere at pressure of 0.5–5 MPa. At the start of the experiment, the combustion chamber was sealed, evacuated, and purged with argon. The chamber was then filled with argon or nitrogen to the desired pressure. During the experiments samples were placed into the reactor and combustion was initiated by means of a tungsten wire located on the upper surface of the sample and was programmed to produce a voltage pulse of 12–18 V for 1.0–2 s. Power to the filament was immediately discontinued after ignition of the sample.

The raw mixtures were prepared using materials listed in Table 1. A green mixture of the reactants was homogenized in a ceramic mortar. Cylindrical samples of 20–50 mm in diameter and height of 40–50 mm were prepared from the green mixture. For MoO_3–Mg–Si–diluent mixture the samples were made by cold uni-axial pressing (30 kN). For the Mo–5Si–diluent system the mixtures were placed into the stainless steel gauze with 0.3 mm thickness.

The maximum combustion temperature (T_c) and the temperature distribution on the combustion wave were measured by tungsten–rhenium thermocouples (W/Re-5, W/Re-20, 100 μm in diameter) previously covered with a thin layer of boron nitride. Three thermocouple holes (3 mm in diameter, 10–25 mm deep) were drilled into each specimen perpendicular to the cylinder axis at a spacing of ∼1 cm. The output signals of thermocouples were transformed by a data logger at the rate of 2 kHz, and were recorded by computer. The average values of combustion velocity (U_c) were calculated as follows: $U_c = l/t$, where l is the distance between thermocouples; t is temporary distance between the signals of thermocouples. The standard error of measurement for T_c and U_c were ±10 °C and 5%, respectively. Reacted samples were washed with deionized water and/or 5% hydrochloric acid. The contents of free (non-reacted) silicon in the washed powders were determined by photocolorimetric analysis method [34]. As synthesized powders were ball milled (duration: 3 h) using zirconia grinding media for further reducing in size of the particles and/or agglomerates. The mass ration between balls (ball size: 2 mm) to powder was 10:1. After the milling procedure the wet mixtures were then dried in a rotary evaporator at 75 °C for 10 h. The washed combustion products and milled powders were studied by XRD analysis with monochromatic Cu Kα radiation (diffractometer PANalytical PW 3040/60 X'Pert PRO). SEM (BS-300), high-resolution SEM (S4800, Hitachi) and TEM (CM30, Philips) analyses were conducted to study the microstructure of the powders. The particle size distribution of the powders was measured using a Malvern Mastersizer X analyzer. Specific surface area measurements were carried out with a Coulter SA3100 analyser.

2.2. Compaction of $MoSi_2$–Si_3N_4 and characterization of dense samples

The synthesized composite powder, 30 wt.% $MoSi_2$–70 wt.% Si_3N_4, was mixed with 3.5 wt.% $Y_2O_{3,}$ (H.C. Starck, Grade C) and 1.5 wt.% $Al_2O_{3,}$ (Alcoa, CT3000SG) as sintering additives in isopropyl alcohol. Additionally, after the mixing process 1 wt.% polyvinyl butyral (PVB) (Mowital B 20H, Clariant GmbH, Germany) as a binder was added to the obtained slurry. The wet powder mixture was then dried in a rotary evaporator and passed through a 125 μm sieve. Dried and sieved powder was compacted into discs of 20 mm in diameter by uni-axial pressing (30 kN). The powder compacts were sintered in BN-coated graphite dies by hot pressing (using KCE, Model HPW150/200-2200-100-LA and Thermal Technology Inc., Model 383-40 presses) with a pressure of 9.5 kN in 0.1 MPa argon atmosphere at temperatures up to 1700 °C. Compact samples were then polished and plasma etched for the microstructural analysis using scanning electron microscopy (SEM, VEGA TS 5136 Tescan, CZ). Phase composition of sintered materials was examined by X-ray analysis with Cu Kα radiation on a diffractometer (PANalytical PW 3040/6092 X'Pert PRO). X-ray photoelectron spetroscopy (XPS) by a PHI-Quantum 2000 technique (excitation source monochromatic Al Kα ($h\nu$ = 1486.6 eV) was used to determine chemical composition of the compact samples' surfaces. To clean the samples from contaminations (adsorbed carbon dioxide, oxygen, nitrogen) argon etching at 2 kV was performed. To compensate for surface charging during measurements electron and argon ion flood gun neutralizers were used.

Table 1
The list of the reagents used for SHS processing

Initial powder		Trademark	Purity (%)	Particle size (μm)
Molybdenum oxide	MoO_3	Tech. condition of manufacturing No. 6-09-4471-77 (Pobedit Co., Russia)	99.7	≤5
Molybdenum	Mo	Technical condition of manufacturing No. 48-19-00-80 (Pobedit Co., Russia)	99.5	≤10
Silicon	Si	KR-1 (Doneck Factory of Chemical Reagents, Doneck, Ukraine)	99.0	≤10
Magnesium	Mg	MPF-3 (Russia)	99.9	150–300
Sodium chloride	NaCl	High Grade, (Michailosk Factory of Chemical Reagents, Russia)	99.9	≤20
Silicon nitride	Si_3N_4	In-house SHS-derived (containing 20 wt.% of α-Si_3N_4)	Nitrogen –37.3 wt.%; free silicon –0.6 wt.%	≤5

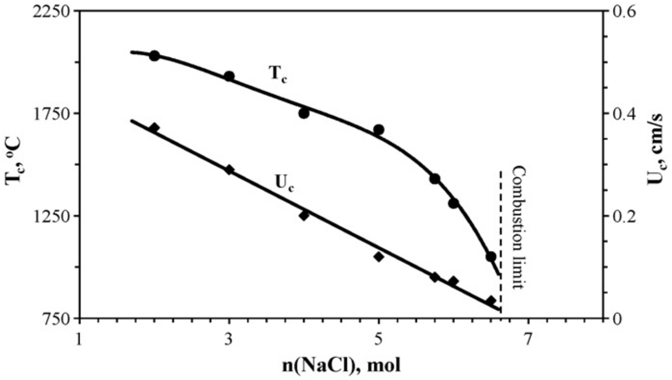

Fig. 1. Combustion temperature (T_c) and velocity (U_c) *vs.* n value for the MoO_3–3Mg–2Si–nNaCl system at P = 1 MPa.

3. Results and discussion

3.1. Combustion features in the MoO_3–3Mg–2Si–NaCl system and synthesis of $MoSi_2$ powder

Combustion in the MoO_3–3Mg–2Si system at P(Ar) = 5 MPa take place very violently, caused by realizing great amount of heat during the process. In addition, the product obtained tends to be MgO, $MoSi_2$, Mo_5Si_3, non-reacted Si and small amounts of $MgSiO_3$. To reduce the combustion temperature the MoO_3–3Mg–2Si mixture was diluted by sodium chloride. The results obtained have shown (Fig. 1) that as the amount of salt (n, moles) is increased in the reacting mixture, the combustion temperature and the velocity decrease. As the combustion temperature decreases (for n values averaging from 2 to 6.5), the reacting system forms $MoSi_2$ and magnesia. The products formed in the salt environment were washed with deionized water and 5% hydrochloric acid examined by SEM, XRD and chemical analyses. The results of SEM analysis show that product particles decrease in size significantly with increasing salt amount. In particular, the size of particles for product formed at n = 3 averages between 5 and 15 μm (Fig. 2a). One important point to note is that product particles formed at high contents of the diluent (more than 5 moles) are smaller than 1 μm (Fig. 2b).

XRD patterns of products obtained at medium contents of the diluent (from 2 to 5 moles) exhibit only diffraction lines for $MoSi_2$ (Fig. 3). At the same time chemical analysis shows low content (1–1.5 wt.%) of non-reacted silicon. In the case of $n > 5$ XRD patterns of products display diffraction lines of the $MoSi_2$ (Fig. 4a) and traces of the Mo_5Si_3 phase. The amount of non-reacted silicon in these products was approximately 5–6 wt.%. To increase the completeness of the combustion reaction, and thus to reduce the amount of non-reacted silicon the diameter of reacting samples was increase from 20 to 50 mm. It is caused by the fact that the diameter of initial sample plays dominant role in regulating the temperature regime of interaction in post-combustion stage. In our experiments increasing the diameter allowed to reduce amount of non-reacted silicon down to 2–2.5 wt.%.

$MoSi_2$ powder synthesized at high contents of the diluent (n = 6) was milled for further reducing in size of the particles and/or agglomerates. XRD analysis of milled powder shows (Fig. 4b) that milling leads to a significant broadening of the diffraction peaks. At the same time the intensities of the peaks decrease by four to five times. This feature probably was caused by the reduction of particle sizes and/or the introducing of micro-strains in the crystallites.

The specific surface area of powders were calculated from BET isotherm model. Note, that specific surface area measured before milling of $MoSi_2$ powder was 1.04 m^2/g. After the milling the spe-

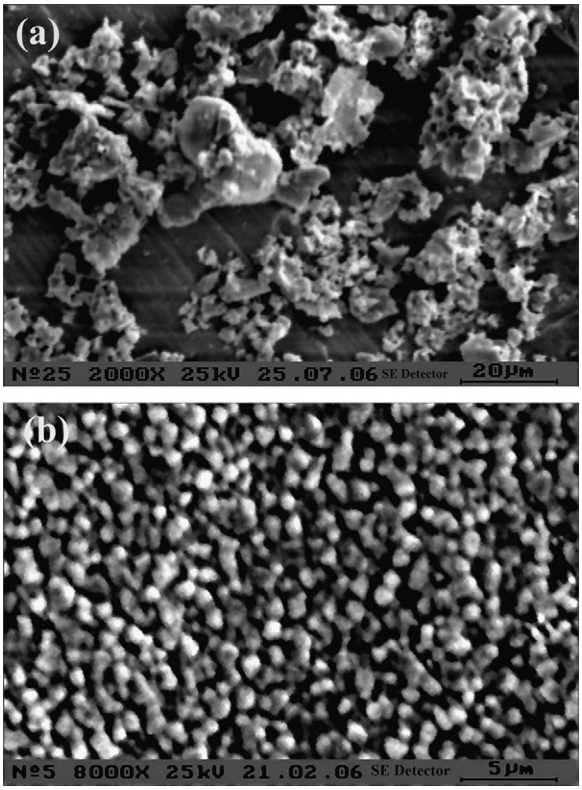

Fig. 2. Microstructure of washed products formed at the combustion of MoO_3–3Mg–2Si mixture at different NaCl amounts: (a) n = 3 and (b) n = 6.

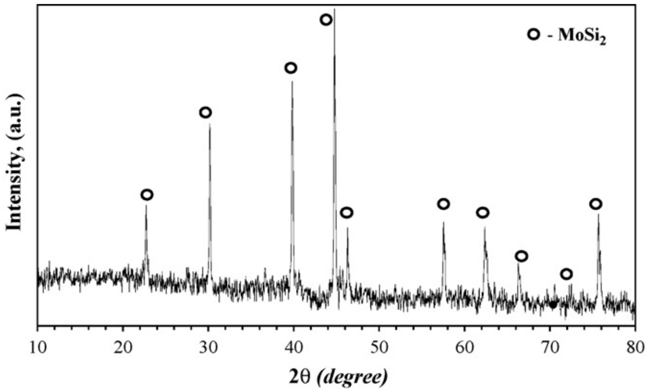

Fig. 3. XRD pattern of the washed product for the MoO_3–3Mg–2Si–3NaCl initial mixture.

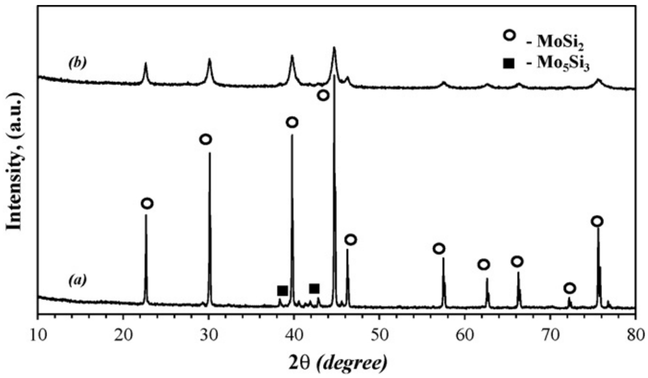

Fig. 4. XRD pattern of the washed product for the MoO_3–3Mg–2Si–6NaCl initial mixture befor (a) and after milling (b).

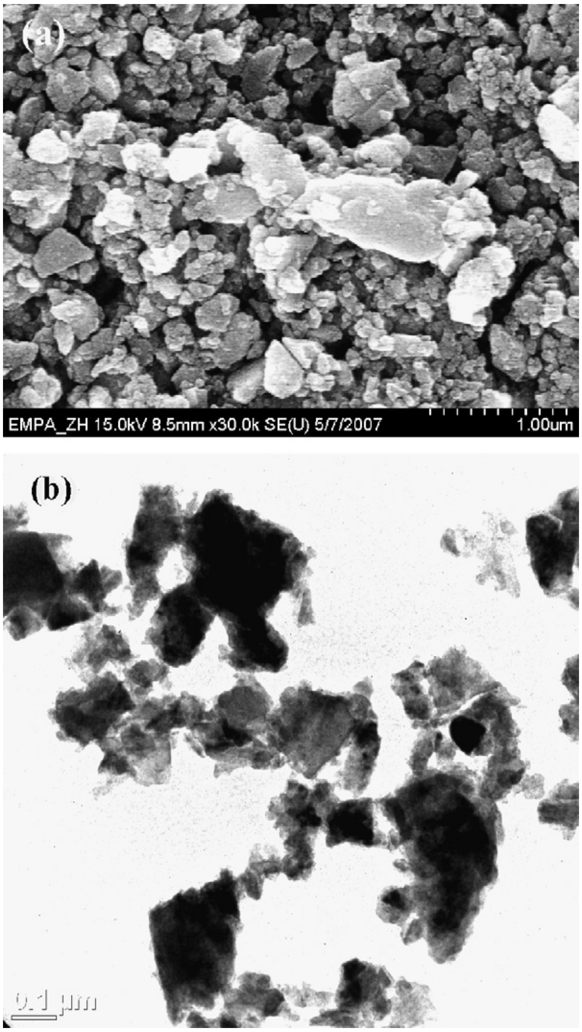

Fig. 5. Microstructure of $MoSi_2$ powder after milling.

cific surface area increased significantly (24.5 m^2/g). In order to reveal ball milling effect on microstructure of $MoSi_2$ powder high-resolution SEM and TEM analyses were also carried out. It was found that short time ball milling leads to significant reduction of products' particle size. Particularly, the results of these investigations (Fig. 5) show that milled (duration: 3 h) powder has a particulate structure with irregular shapes and sizes below 0.5 μm.

3.2. Combustion in the Mo–5Si–NaCl–N_2 system and synthesis of $MoSi_2$–Si_3N_4 composite powders

Experiments indicate that the self-sustaining regime of interaction in the Mo–5Si–N_2 system is possible for samples with a relatively low density (down to 0.45) and nitrogen pressures more than 1.5 MPa. In our previous investigation [27] it was shown that at combustion of Mo + 5Si mixture in nitrogen the reaction temperature changed from 1800 to 2000 °C, yielding the products containing $MoSi_2$, Si_3N_4 and significant amount of non-reacted silicon. In addition the product contains coarse particles (up to1 cm) with metallic glance. XRD analysis was testified that these particles represent pure silicon, probably formed due to the coalescence of melted silicon drops in the interaction zone. It is expected that the presence of molten sodium chloride will prevent coalescence silicon drops and by this way promotes the fast nitriding of silicon. For this purpose combustion of the initial Mo–5Si–*n*NaCl mixture under the nitrogen atmosphere ($P(N_2)$ = 5 MPa) was studied. According to results obtained (Fig. 6) for the Mo–5Si–*n*NaCl–N_2 system both the combustion parameters show complex character (curves with a maximum) from the *n* value and a combustion limit was registered at $n = 1.8$. The results of chemical analyses show that NaCl addition leads to significant change in completeness of silicon nitriding. In particular, a steep decrease in free silicon amounts (from 10 to 0.7 wt.%) was observed when NaCl (more then 0.5 moles) introduced into the green mixture. Simultaneously significant amount of Mo_5Si_3 was detected by XRD analysis of the products. A possible explanation for molybdenum-rich Mo_5Si_3 phase formation is that sodium chloride provides better conditions for silicon nitriding thus decreasing amount of silicon for $MoSi_2$ to be produced. Nevertheless, it is not excluded the partial nitriding of the formed $MoSi_2$ by the reaction:

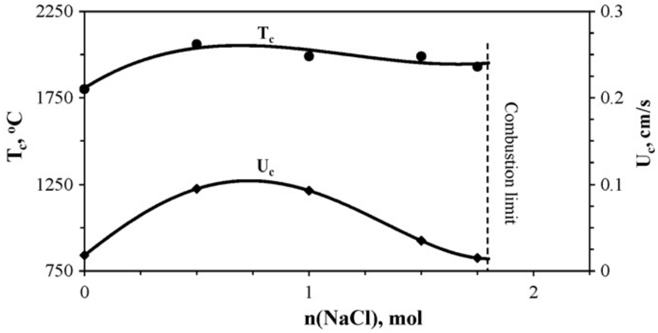

Fig. 6. Combustion temperature and velocity *vs*. *n* value for the Mo–5Si–*n*NaCl––N_2 system at $P = 5$ MPa.

$$15MoSi_2 + 14N_2 \rightarrow 3Mo_5Si_3 + 7Si_3N_4 \qquad (1)$$

Therefore, for further optimization of the process it is necessary to prevent formation of Mo_5Si_3. For this purpose silicon nitride was introduced into the Mo–5Si–0.5NaCl mixture as the second diluting compound.

Experiments for the Mo–5Si–0.5NaCl–$mSi_3N_4 + N_2$ system (*m* is moles of Si_3N_4) in nitrogen ($P = 5$ MPa) show that addition of silicon nitride leads to decrease in combustion temperature from 2050 to 1750 °C. Accordingly, the combustion front velocity was reduced by about 2.5 times (from 0.1 down to 0.04 cm/s). Moreover, addition of 1.7 moles of silicon nitride resulted in a combustion limit.

Hence, decreasing the combustion parameters by this way makes it possible to regulate the phase composition of the combustion products. Particularly, within the $0 \leq m < 0.7$ range the products contained $MoSi_2$, Si_3N_4, NaCl, as well as significant amounts of Mo_5Si_3. The product formed in the $0.7 \leq m \leq 1.65$ interval contained $MoSi_2$, Si_3N_4, NaCl, and the amount of free silicon was small enough (0.5–0.7 wt.%).

For exploring the effect of nitrogen pressures a series of experiments using samples made from Mo–5Si–0.5NaCl–Si_3N_4 initial mixtures (with a relative density of 0.37) was conducted. The results obtained have shown that this mixture does not combust at nitrogen pressure less than 1 MPa. In the vicinity of the combustion limit the registered combustion parameters (T_c and U_c) have minimal values of 1680 °C and 0.02 cm/s, respectively. Increasing the pressure from 1 to 3 MPa resulted in increasing the combustion parameters (T_c = 1880 °C and U_c = 0.06 cm/s). In this interval of pressure unstable (pulsing) propagation of combustion wave was observed. At $P \geq 3$ MPa transition of pulsing to stable combustion mode takes place without significant change in the combustion parameters. It was shown by XRD analysis that at pressures below 3 MPa combustion products contain significant amounts of non-reacted silicon. The products obtained at nitrogen pressures higher

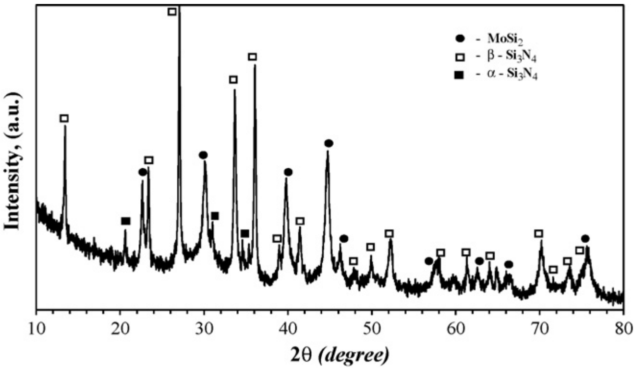

Fig. 7. XRD patterns for milled 30 wt.% $MoSi_2$–30 wt.% Si_3N_4 composite powder.

than 3 MPa exhibited diffraction lines for $MoSi_2$, β-Si_3N_4 and small amount of α-Si_3N_4. In addition, the amount of non-reacted silicon was about 0.6–0.8 wt.%.

Thus, optimization of combustion conditions by the amounts of diluents (NaCl and Si_3N_4) and the nitrogen pressure allows the control main parameters of the process and to synthesize $MoSi_2$–Si_3N_4 powders containing from 30 to 40 wt.% $MoSi_2$.

As synthesized 30 wt.% $MoSi_2$-70 wt.% Si_3N_4 composite powder was milled in ball mill for 3 h. XRD analysis of milled powders has shown (Fig. 7) diffraction lines for $MoSi_2$ and β-Si_3N_4. In addition small amount of α-Si_3N_4 was also detected by XRD analysis. The results on particle size distribution for the 30% $MoSi_2$–70% Si_3N_4 composite after the milling (Fig. 8) have shown that approximately 40 vol.% of the particles are of a submicron size. The size of 50 and 90 vol.% of particles is less than 1.5 and 3.5 μm, respectively. The specific surface area of composite powder measured before milling was 0.85 m^2/g. After the milling the specific surface becomes 22 m^2/g. High-resolution SEM and TEM analyses (Fig. 9) show that milled composite powder has two characteristic ranges for particle dimensions: from 0.5 to 1.5 μm and from 100 to 500 nm.

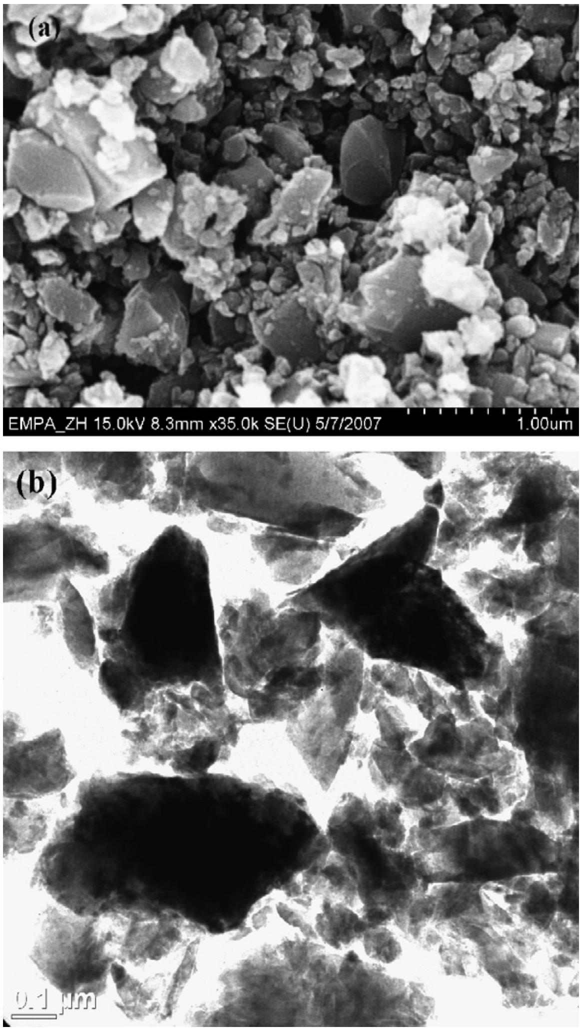

Fig. 9. Microstructure of $MoSi_2$–Si_3N_4 composite powder after milling.

3.3. Densification of 30 wt.% $MoSi_2$–70 wt.% Si_3N_4 composite powder

Combustion synthesized 30 wt.% $MoSi_2$–70 wt.% Si_3N_4 composite powder was compacted by hot pressing in a low-pressure (0.1 MPa) argon atmosphere. The hot pressed samples were produced in a disc shape with a diameter of 20 mm and a thickness of 3–5 mm. The hot pressing conditions, absolute shrinkage (s) and its differential curve are illustrated in Fig. 10. The heating cycle was developed to produce high-density composite according to literature data [13,14]. It may be seen that main shrinkage of powder takes place at temperatures from 1500 to 1700 °C. The first maximum on the ds/dt curve was registered when 3–4 kN force was applied on the sample. The second maximum on the same curve was registered at a constant pressure (9.5 kN) of pressing during the increasing temperature.

After the hot pressing, the surfaces of the sintered discs were cleaned by diamond grinding. The density of discs, measured by water displacement method, was 0.98–0.99 of the theoretical

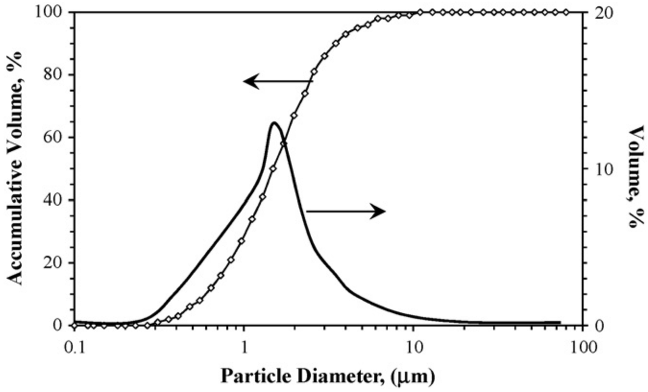

Fig. 8. Particle size distribution for the 30 wt.% $MoSi_2$–70 wt.% Si_3N_4 composite.

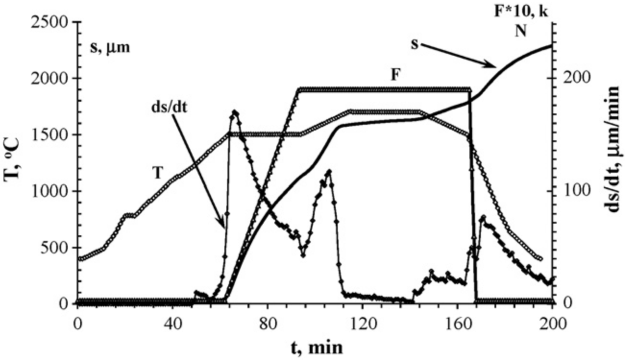

Fig. 10. Sintering curves for the 30 wt.% $MoSi_2$–70 wt.% Si_3N_4 composite powder.

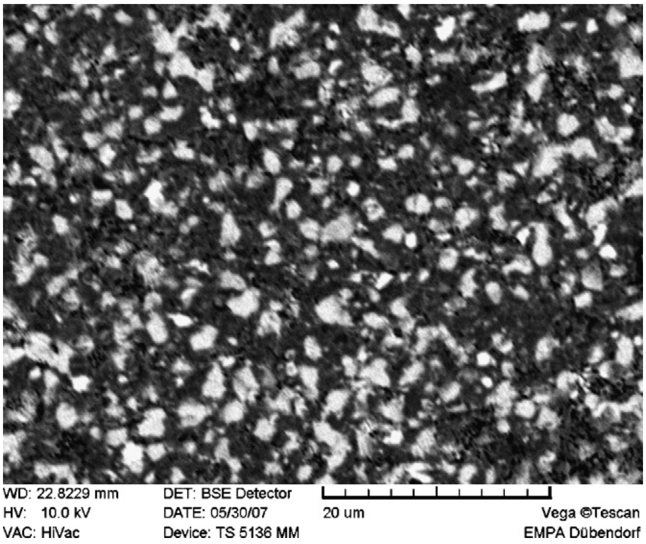

Fig. 11. SEM micrograph of compact sample obtained at hot pressing of combustion synthesized $MoSi_2$–Si_3N_4 composite powder.

density. The compacts consolidated by this way have two-phase structure consisting $MoSi_2$ and β-Si_3N_4. Note that no crystalline phases containing Y and Al were observed in the XRD patterns. SEM investigation of the dense material has shown that $MoSi_2$ grains (with particles 2–4 μm in size) have irregular shape and are homogeneously distributed in the Si_3N_4 matrix (Fig. 11).

The surface chemical composition of compact discs was analyzed by XPS method. On each sample two survey measurements were made: one on the surface of "as received" and the second one after argon ion sputtering. On the surfaces of the "as received" discs before etching considerable amounts of carbon and oxygen were detected. After the sputtering the concentrations of these elements were reduced significantly. Particularly, the carbon concentration decreased from 25–30 to 2–4 at.%. Oxygen concentrations on sputtered discs varied from 11.5 to 17.5 at.%. Argon sputtering helps also for increasing the peak intensities on XPS spectra for nitrogen, silicon and molybdenum. On the surface of the samples contaminations of iron and zirconium were registered. The results of analyses have shown that yttrium and aluminium are absent on the disc surfaces.

4. Conclusions

(1) The results of investigation were shown that the combustion parameters (T_c and U_c) in the MoO_3–3Mg–2Si system can be regulated using sodium chloride as an inert diluent with the respect to synthesis of $MoSi_2$. Addition of sodium chloride helps to regulate also size of product's particle and to synthesize $MoSi_2$ particles below 1 μm.

(2) In the Mo–5Si–N_2 systems both the reaction conditions and phase composition can be regulated with combined NaCl–Si_3N_4 diluent. Optimum conditions for synthesizing $MoSi_2$–Si_3N_4 powders containing 30–40 wt.% of molybdenum disilicide were determined.

(3) It was shown that the synthesized $MoSi_2$–Si_3N_4 composite material could be hot pressed with the standard technique for silicon nitride to 0.98–0.99 theoretical density. Dense materials produced by this way have two-phase structure consisting of $MoSi_2$ and β-Si_3N_4. Nitrogen, silicon and molybdenum were determined as main elements on the surfaces of sintered disks by means of XPS method.

Acknowledgements

The authors would like to thank the support by the Swiss National Science Foundation (Project no. PIOI2-117177). Further the authors would like to acknowledge Dr. Kranthi Akurati, Salvatore Fuso, Silke Liebert-Winter, Karine Asatryan and Armenuhi Yeghishyan for their help in particle sizing, chemical and microstructure analyzing.

References

[1] I.E. Campbell, E.M. Sherwood (Eds.), High Temperature Materials and Technology, Wiley, New York, 1967, p. 399.
[2] A.K. Vasudevan, J.J. Petrovic, Mater. Sci. Eng. A 155 (1992) 1.
[3] S.C. Deevi, N.N. Thadhani, Mater. Sci. Eng. A 192–193 (1995) 604.
[4] A.K. Bhattacharya, J.J. Petrovic, J. Am. Ceram. Soc. 74 (1991) 2700.
[5] D.E. Alman, K.G. Shaw, N.S. Stoloff, K. Rajan, Mater. Sci. Eng. A 155 (1992) 85.
[6] D.E. Alman, R.D. Grover, Scripta Mater. 34 (1996) 1287.
[7] J.J. Petrovic, Intermetallics 8 (2000) 1175.
[8] J.J. Petrovic, R.E. Honnell, J. Mater. Sci. Lett. 9 (1990) 1083.
[9] J.J. Petrovic, M.I. Pena, H.H. Kung, J. Am. Ceram. Soc. 80 (1997) 1111.
[10] M.G. Hebsur, Mater. Sci. Eng. A 261 (1998) 24.
[11] M.Y. Kao, J. Am. Ceram. Soc. 76 (1993) 2879.
[12] D.E. Alman, J.H. Tylczak, J.A. Hawk, M.G. Hebsur, Mater. Sci. Eng. A 261 (1999) 245.
[13] Zh. Guo, G. Blugan, T. Graule, M. Reece, J. Kuebler, J. Eur. Ceram. Soc. 27 (2007) 2153.
[14] Zh. Guo, M. Parlinska-Wojtan, G. Blugan, T. Graule, M. Reece, J. Kuebler, Acta Mater. 55 (2007) 2875.
[15] R.B. Schwarz, S.R. Srinivasan, J.J. Petrovic, C.J. Maggiore, Mater. Sci. Eng. A 155 (1992) 75.
[16] P.Y. Lee, T.R. Chen, J.L. Yang, T.S. Chin, Mater. Sci. Eng. A 192–193 (1995) 556.
[17] L. Liu, F. Padella, W. Guo, M. Magini, Acta Metall. Mater. 43 (1995) 3755.
[18] N.N. Thadhani, J. Appl. Phys. 76 (1994) 2129.
[19] A.R. Sarkisyan, S.K. Dolukhanyan, I.P. Borovinskaya, A.G. Merzhanov, Combust. Explos. Shock Waves 14 (1978) 310.
[20] S. Zhang, Z.A. Munir, J. Mater. Sci. 26 (1991) 3685.
[21] S.C. Deevi, Mater. Sci. Eng. A 149 (1992) 241.
[22] S.W. Jo, G.W. Lee, J.T. Moon, Y.S. Kim, Acta. Mater. 44 (1996) 4317.
[23] A. Chrysanthou, R.C. Jenkins, M.J. Whiting, P. Tsakiropoulos, J. Mater. Sci. 31 (1996) 4221.
[24] C.L. Yeh, W.H. Chen, J. Alloys Compd. 438 (2007) 165.
[25] J. Xu, H. Zhang, J. Yan, B. Zhang, W. Li, Int. J. Refract. Met. Hard Mater. 25 (2007) 318.
[26] S.L. Kharatyan, Kh. V. Manukyan, H.H. Nersisyan, Adv. Sci. Technol. 30 (2003) 433.
[27] A.V. Gabrielyan, Kh. V. Manukyan, S.L. Kharatyan, J. Alloys Compd. 454 (2008) 394.
[28] Ch. Gras, D. Vrel, E. Gaffet, F. Bernard, J. Alloys Compd. 314 (2001) 240.
[29] Ch. Gras, E. Gaffet, F. Bernard, Intermetallics 14 (2006) 521.
[30] Ch. Gras, F. Charlot, E. Gaffet, F. Bernard, J.C. Niepce, Acta Mater. 47 (1999) 2113.
[31] H.H. Nersisyan, J.H. Lee, C.W. Won, Mater. Res. Bull. 38 (2003) 1135.
[32] H.H. Nersisyan, H.I. Won, C.W. Won, Mater. Lett. 59 (2005) 3950.
[33] J.H. Lee, H.H. Nersisyan, H.H. Lee, C.W. Won, J. Mater. Sci. 39 (2004) 1397.
[34] Silicon Nitride, TU of Standard Agency of Russian Federation, No. 42-42-5-84, 25.