

# Effect of atmosphere and heating rate on mechanism of MoSi<sub>2</sub> formation during self-propagating high-temperature synthesis

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**Abstract** To study the effect of atmospheric type and heating rate on formation mechanism of MoSi<sub>2</sub>, the Mo + 2Si powder mixture was exposed to simultaneous thermal analysis (STA) in air atmosphere at different heating rates (10, 15, and 20 °C/min). To further study the changes, thermal analyses of molybdenum powders and consumed silicon were also performed separately. An amount of aluminum powder (5 wt.%) was also added to Mo + 2Si powder mixture and exposed to thermal analysis at different heating rates (10, 15, and 20 °C/min) to study the effect of the presence of active elements (like aluminum) on the trend of the performance of changes. To perform phase studies on the products of the thermal analysis at a later stage, each product was separately tested by an X-ray diffraction (XRD) method. Contrary to expectations, the XRD patterns showed that the trends of changes during thermal analysis were not in the direction of MoSi<sub>2</sub>, and the DTA–TG peaks obtained from these analyses were in fact related to other changes. Ultimately, the results showed that the peaks on the DTA curves resulted from the oxidation of molybdenum particles; and the (MoO<sub>3</sub>) melt of the product, and in continuation of the reduction of a part of this oxide, it resulted during the silicothermic and aluminothermy reactions. The results of this research also showed that with regard to the presence of intensive oxidation tendency of molybdenum particles, there is no chance for the formation of MoSi<sub>2</sub> by heating the powder mixture of Mo + 2Si in air atmosphere and at low heating rates.

**Keywords** SHS · Oxidation · Mechanism · MoSi<sub>2</sub> · MoO<sub>3</sub> · MoO<sub>2</sub>

## Introduction

The properties of molybdenum disilicide (MoSi<sub>2</sub>), such as high thermal and electrical resistances, high melting point (over 2,020 °C), and excellent resistance to corrosion at high temperatures have extended the applicability domain of this material in different industries day by day [1–5]. This material is produced by different methods. One of these methods which has been of interest to many researchers, is the self-propagating high-temperature synthesis (SHS). In the last 20 years, combustion synthesis merged as an innovative and promising powder-preparation method [6]. The self-propagating high-temperature synthesis has the advantages of low energy requirement, short processing time, simplicity of facilities, formation of high-purity products, and stabilization of metastable phases [7]. In fact, the very intense heat resulting from the reaction between molybdenum and silicon provides the chance for the production of molybdenum disilicide by this method [8]. So far, many surveys have been performed on Mo–Si system from different aspects in relation to the application of SHS process. However, one of the most important aspects of this process which has drawn the attention of many to itself in recent years, has been the issue of finding the mechanism of this process and studying the effects of different factors on it [9–11]. The use of thermal analysis test, which has been used by many researchers so far, is one of the most widely applied methods in this area. For example, in 1995, Seetarama [12] tried to study the existing diffusion reactions in MoSi<sub>2</sub> propagation synthesis and the effect of heating rate on the mechanism of the performance

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**Table 1** The specifications of raw materials

| Powder material | Purity/% | Particles size/ $\mu\text{m}$ | Company |
|-----------------|----------|-------------------------------|---------|
| Si              | >99      | 1–10                          | Merck   |
| Mo              | >99.9    | 1–40                          | Merck   |
| Al              | >99      | 100–200                       | Fluka   |

of this process by the thermal analysis test. In a study performed in 2010, the manner of the effect of the presence of aluminum element on the mechanism of the performance of this process was also studied [10].

Although many studies have been performed in this area, with regard to intense sensitivity of this process to parameters, such as heating rate and type of the governing atmosphere, it seems that more studies can present more information to achieve better control of the conditions of this process. It was also tried to study the effectiveness of aluminum on this process by relying on the results of thermal analysis, while verifying the results obtained by the previous researchers in this area.

## Materials and methods

All specifications of the raw materials, such as molybdenum powders, silicon, and aluminum, are presented in Table 1. A powder mixture with a stoichiometry ratio of Mo + 2Si and (Mo + 2Si) + 5 wt.% Al was also prepared.

To study the mechanism of MoSi<sub>2</sub> formation, the experiments were carried out in a STA device (STA503, Bahr) capable of simultaneous TG–DTA. The specifications related to these tests are presented in Table 2. In the next stage, the products obtained from the process were

studied for phase analysis by X-ray diffraction (MPD-‘X’PERT, Philips).

## Results and discussion

The DTA–TG curves for the powder mixture of Mo + 2Si at different heating rates (10, 15, and 20 °C/min) are presented in Fig. 1. The DTA curves are divided into the following regions [10, 12]:

Region I: Exothermic peak related to solid–solid reaction between molybdenum and silicon particles

Region II: Endothermic peak related to the melt of a part of silicon particles

Region III: Exothermic peak related to dissolution of molybdenum particles in melting silicon and formation of MoSi<sub>2</sub>

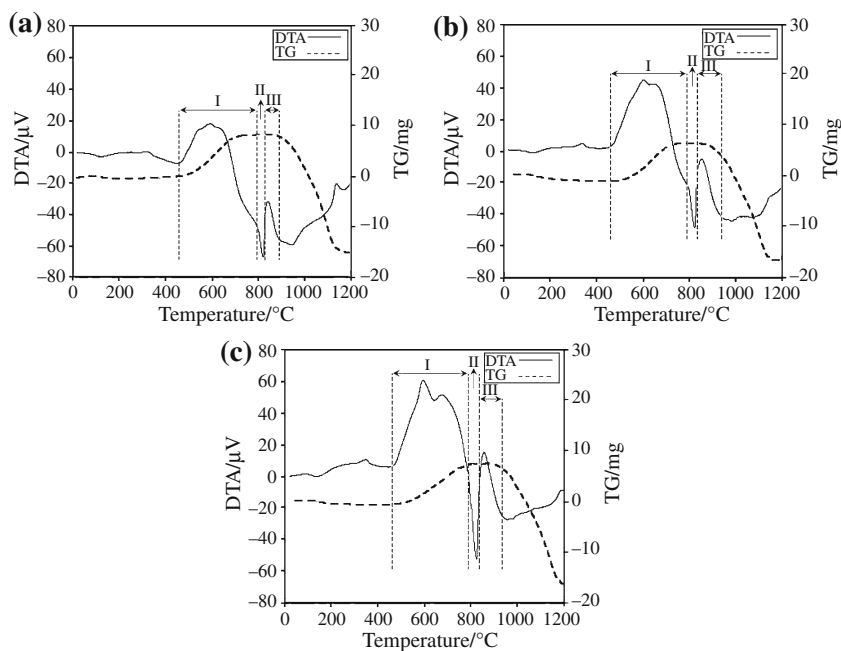
Additional studies on the changes taking place during the DTA–TG tests were performed, and the X-ray diffraction patterns of the products are presented in Fig. 2. As is observed, contrary to molybdenum, considerable silicon residual is left, and in addition, only a negligible amount of MoSi<sub>2</sub> intermetallic compound is formed. If the peaks on the DTA curves were related to the mentioned occurrences, then MoSi<sub>2</sub> phase would surely form the major part of the phases. Hence based on these results, the previous analyses performed on the DTA curves can definitely be rejected. In this regard, the peaks observed on the DTA curves should be related to other phenomena requiring further research. To investigate further, the consumed molybdenum and silicon powders were separately studied through thermal analysis. Figure 3 shows the DTA–TG curves for the consumed silicon powder. As is observed in this figure, no specific phenomenon is observed during heating. This analysis was performed in air atmosphere at 20 °C/min

**Table 2** The sample mass and the specifications of TG–DTA experiments

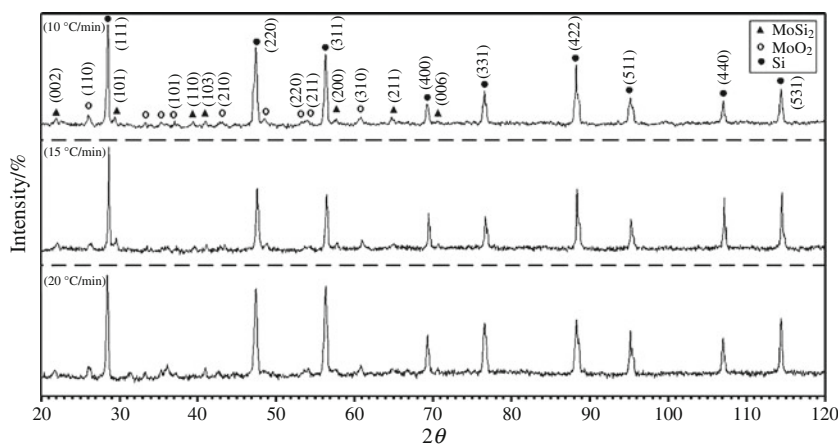
| No. | Sample                             | $W_0^a/\text{mg}$ | $T_{\text{max}}/^\circ\text{C}$ | Atm. | Heating rate/ $^\circ\text{C min}^{-1}$ |
|-----|------------------------------------|-------------------|---------------------------------|------|---|
| 1   | Mo                                 | 35.07             | 1200                            | Air  | 20                                      |
| 2   | Si                                 | 35.21             | 1200                            | Air  | 20                                      |
| 3   | Mo + 2Si                           | 35.39             | 1200                            | Air  | 10                                      |
| 4   | Mo + 2Si                           | 33.99             | 1200                            | Air  | 15                                      |
| 5   | Mo + 2Si                           | 38.80             | 1200                            | Air  | 20                                      |
| 6   | (Mo + 2Si) + 5 wt.% Al             | 35.04             | 1200                            | Air  | 10                                      |
| 7   | (Mo + 2Si) + 5 wt.% Al             | 35.17             | 1200                            | Air  | 15                                      |
| 8   | (Mo + 2Si) + 5 wt.% Al             | 35.19             | 1200                            | Air  | 20                                      |
| 9   | (Mo + 2Si) + 5 wt.% Al (Compacted) | 35.17             | 900                             | Air  | 20                                      |
| 10  | (Mo + 2Si) + 5 wt.% Al (Compacted) | 35.06             | 1200                            | Air  | 100                                     |
| 11  | Mo + 2Si                           | 35.00             | 1200                            | Ar   | 20                                      |

<sup>a</sup>  $W_0$  The initial weight of sample

**Fig. 1** TG and DTA curves for the heating processes of Mo + 2Si powders in air atmosphere up to 1,200 °C at different heating rates: **a** 10 °C/min, **b** 15 °C/min, and **c** 20 °C/min



**Fig. 2** XRD patterns of the products obtained through thermal analyses of Mo + 2Si powders at different heating rates up to 1,200 °C

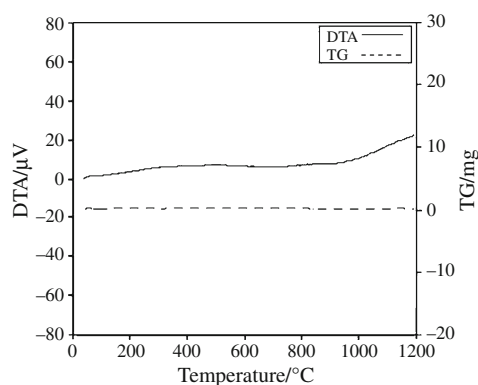


heating rate. For this purpose, the molybdenum powder was also studied separately through thermal analysis (in air atmosphere at the heating rate of 20 °C/min) and the related DTA–TG curves are shown in Fig. 4.

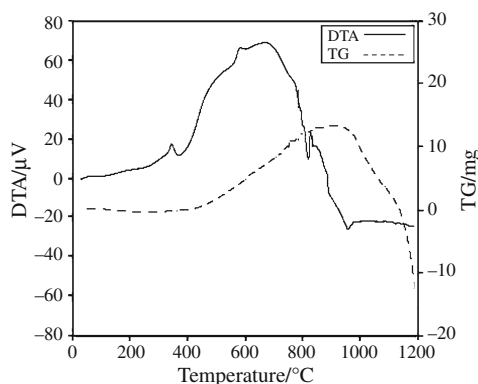
As is inferred from Figs. 3 and 4, contrary to silicon, molybdenum heating during thermal analysis is accompanied with the occurrence of some phenomena such that it bears many similarities with the trend of the changes on the DTA curves presented in Fig. 1. The presence of exothermic peaks within a rather broad thermal range (within a temperature range of 400–800 °C), and an endothermic peak at 800 °C with a subsequent exothermic peak show that there is a significant relation between the results presented in this analysis and those in Fig. 1. In addition, the TG curve related to molybdenum powder (Fig. 4) has also a good conformity with the TG curves related to the powder mixture of Mo + 2Si (Fig. 1). The phase study of

the products resulting from this analysis could provide useful information, but since no product remained in the crucible after analysis, it was not possible to perform this phase study. It is, therefore, clearly specified that the trend of the changes in this analysis has been toward the production of a product which was ultimately vaporized. It is, therefore, clearly specified that the peaks referred to as peaks related to solid–solid reactions, silicon melt, and the formation of MoSi<sub>2</sub> in the previous studies were in fact related to changes taking place only on molybdenum powders present in the mixture of the raw materials, and not related to the process of MoSi<sub>2</sub> formation. Then, what phenomena have happened in molybdenum to produce such peaks?

The DTA and TG curves in Fig. 4 clearly show that by the start of the first exothermic peak, the TG curve shows the mass increase too. With regard to the performance of



**Fig. 3** TG and DTA curves for the heating processes of silicon powder separately in air atmosphere at the heating rate of 20 °C/min up to 1,200 °C. The PDF numbers used are Si 26-1481; MoSi<sub>2</sub> 41-0612; and MoO<sub>2</sub> 02-0422



**Fig. 4** TG and DTA curves for the heating processes of molybdenum powder separately in air atmosphere at the heating rate of 20 °C/min up to 1,200 °C

this analysis in air atmosphere, this mass increase and the corresponding heat released can be related to molybdenum oxidation. The results obtained from the phase studies (Fig. 2) also prove the presence of (MoO<sub>2</sub>) oxide products resulting from this reaction among the products. This first exothermic peak is therefore related to the oxidation of molybdenum particles present in the powder mixture, which fact has been clearly proven by the earlier researchers [13] in this field.

However, there are still many points that are not clear about the results obtained from these analyses which should be clarified through further study and thorough recognition of the trend of the changes on TG and DTA curves. Therefore, the cause of the generation of endothermic peak (region II) as well as subsequent exothermic peak (region III) presented on DTA curves (Figs. 1, 4); stoppage of the ascending trend of the TG curves (Fig. 4) after passing through region I; and even their descent in

**Table 3** The enthalpy for the formation of different compounds with molybdenum and silicon elements and oxygen, along with their melting points

| Material             | $\Delta H_f/\text{kJ mol}^{-1}$ | $T_m/^\circ\text{C}$ |
|----------------------|---------------------------------|----------------------|
| MoSi <sub>2(s)</sub> | -118.826                        | 2020                 |
| MoO <sub>(g)</sub>   | 310.959                         | -                    |
| MoO <sub>2(s)</sub>  | -588.940                        | -                    |
| MoO <sub>3(s)</sub>  | -745.087                        | 802                  |
| SiO <sub>2(s)</sub>  | -910.857                        | 1723                 |

continuation; and the emptiness of the crucible after the termination of thermal analysis test performed on molybdenum powders are the indicators that can relate all these items to oxidation reaction.

Table 3 (adopted from Ref. [14]) shows the enthalpy for the formation of different compounds with molybdenum, silicon elements, and oxygen, along with their melting points.

As is observed, the formation of heat related to SiO<sub>2</sub> is more negative than the others among these compounds. However, the results of thermal analysis studies (DTA and TG curves in Fig. 3) and the phase studies (Fig. 2), show the lack of oxidation of silicon powder particles. The results obtained from the studies performed by researchers [15] showed that the cause of resistance against the oxidation of silicon powder particles is due to the formation of an amorphous oxide layer on the surface of these particles. Figures 1, 2, and 4 depict the oxidation of molybdenum particles very well. The studies done by researchers [13] showed that molybdenum affinity to oxidation is so much that if the MoSi<sub>2</sub> intermetallic compound is also exposed to the temperature range of 500–800 °C in the presence of oxygen, then it becomes decomposed and will be intensely oxidized during the following reaction. Now, if the temperature exceeds 802 °C (with regard to MoO<sub>3</sub> melting temperature), then this oxide melts and because of rather high vapor pressure, it is vaporized, leaving behind only silicon scale. Because of MoSi<sub>2</sub> decomposition, silicon remains in atomic form, and the issue of protective amorphous layer on it is immediately oxidized and cannot be set forth to allow for any discussion.

It is clearly specified that the appearance of endothermic peak of the DTA curves at about 800 °C (presented in Figs. 1 and 4) is related to the molybdenum oxide melt (MoO<sub>3</sub>) which has been formed on the surface of molybdenum particles up to this stage of heating. Studies have shown that although vaporization of MoO<sub>3</sub> takes place at 1,165 °C; with regard to high vapor pressure of this melt at 800 °C, this compound is quickly vaporized after melting [13]. The stoppage of mass increase and mass reduction observed in this region of the TG curve (Fig. 4) also shows

the vaporization of this oxide very well. As was mentioned, earlier, the intensity of this vaporization is so high that it causes the emptiness of the crucible in thermal analysis of consumable molybdenum powder.

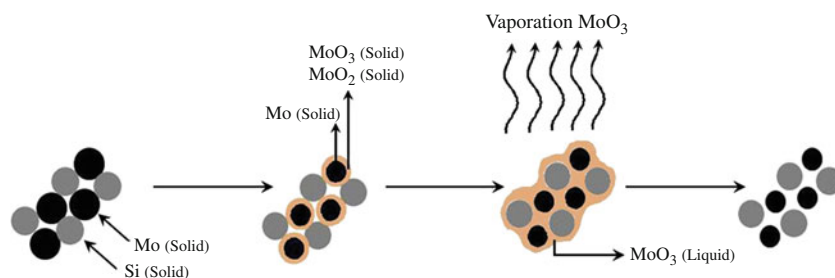
In spite of the results thus obtained, there is still a need for more studies to identify the factor producing the second exothermic peaks on the DTA curves. In previous studies, the second exothermic peak on the DTA curves was attributed to the formation of MoSi<sub>2</sub> [10, 12]. However, in phase studies, this issue is totally rejected. In addition to that, on the DTA curve related to pure molybdenum powder, this peak is also observed; therefore, this peak can result from the reactions taking place among molybdenum particles.

As was mentioned above, the molybdenum oxide formed on the surface of molybdenum particles before reaching 800 °C starts to melt when reaching this temperature (800 °C) and is vaporized after melting because of its high vapor pressure. Because of this situation, a rather great surface area of molybdenum particles is exposed to atmospheric oxygen and the possibility for intense oxidation in molybdenum particles arises. These changes are schematically shown in Fig. 5. Because of this oxidation, a rather great amount of heat is released. The released heat is shown as an exothermic peak on the DTA curves. Also, during this intense oxidation (accompanied with mass increase), the molybdenum oxide vaporization results in mass reduction of the specimen in the opposite direction, and, for this reason, no special mass changes are observed within this temperature range.

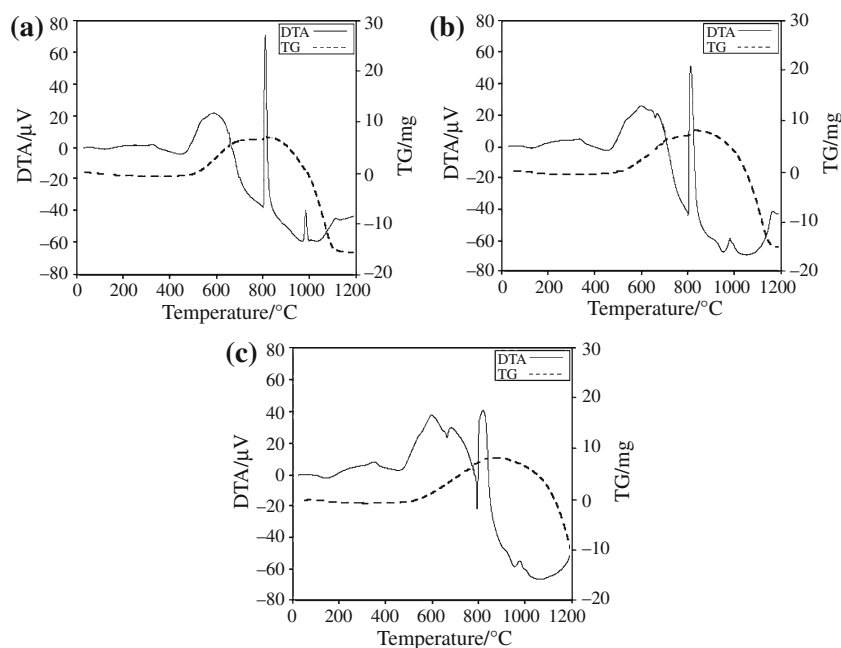
In addition, vaporization of the melted molybdenum oxide at 800 °C can provide suitable conditions for the penetration and contact of this oxide with silicon particles. Under such conditions, the possibility for the reduction of this oxide during a silicothermic reaction arises. This reaction in turn can also generate heat in this region (region III). For this reason, it is clearly observed from Fig. 1 that the existing exothermic peak in region III enjoys more intensity compared with similar peak in the DTA curve in Fig. 4. This intensity difference can result from the same reduction reaction. Under such conditions, it is expected that, if an amount of an element more active than silicon is

added to the powder mixture, then the reduction reaction becomes more intense and, consequently, more intense exothermic peak is observed in this region. For this purpose, 5 wt.% of aluminum powder was added to a part of the powder mixture of Mo + 2Si, and this powder mixture was thermally analyzed at different rates (10, 15, and 20 °C/min). The results obtained from these analyses are presented in Fig. 6. As is observed, there is no particular difference between the TG curves presented in this figure and those in Fig. 1. However, as it was expected, with the presence of aluminum, the exothermic reaction of the vaporized molybdenum oxide reduction is reinforced at this stage. From the DTA curves presented in Fig. 6, it is clearly observed that with the addition of a little amount of aluminum powder particles to the mixture of raw materials, the intensity of the second exothermic peak has greatly increased compared to the similar peaks in Fig. 1. Thus, based on these results, the reduction of a part of the vaporized molybdenum oxide by aluminothermic and silicothermic reactions is proven without doubt. In addition, up to the temperature of 1,000 °C on the DTA curves presented in Fig. 6, some exothermic peaks are observed. The studies of the earlier researchers in this area had shown that these peaks were related to the oxidation of aluminum particles present in the mixture of the raw materials. In fact, the production of a protective oxide layer prevents the oxidation of these particles up to this temperature. The studies done by the researchers [16, 17] in this area have shown that only a portion of the aluminum particles are oxidized at this temperature. The X-ray diffraction pattern of the product of the DTA–TG analysis is presented in Fig. 7 as well. Also, in these patterns, the MoSi<sub>2</sub> phase is not clearly noticeable among the phases. For this reason, the study performed on the mechanism of the formation of MoSi<sub>2</sub> in the presence of aluminum (according to the previous study [10]) is utterly rejected. The low percentage of the aluminum present in the mixture of the raw materials and the oxidation of a part of these aluminum particles during thermal analysis have made it impossible to identify this oxide phase by XRD analysis. The X-ray diffraction patterns (Fig. 7) also clearly show that a great percentage of the aluminum still remains unreactive after heating.

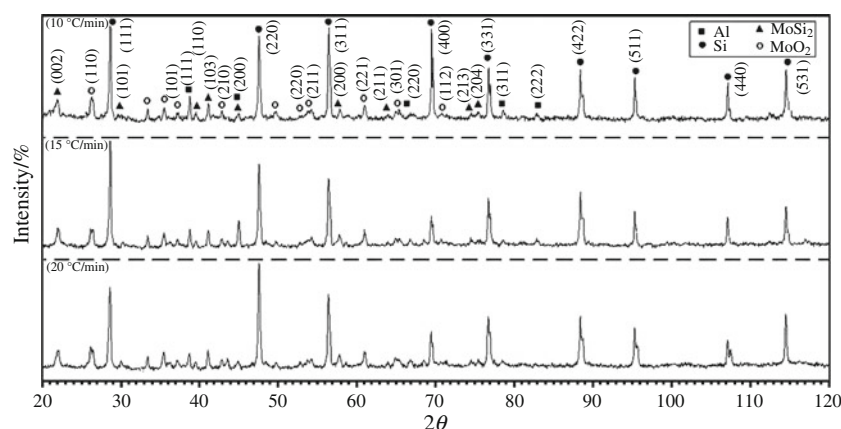
**Fig. 5** Schematic representation of oxidation process of molybdenum powder



**Fig. 6** TG and DTA curves for the heating processes of (Mo + 2Si) + 5 wt.% Al powders in air atmosphere up to 1,200 °C at different heating rates: **a** 10 °C/min, **b** 15 °C/min, and **c** 20 °C/min



**Fig. 7** XRD patterns of the products obtained through thermal analyses of (Mo + 2Si) + 5 wt.% Al powders at different heating rates up to 1,200 °C. The PDF numbers used are Si 26-1481; MoSi<sub>2</sub> 41-0612; MoO<sub>2</sub> 02-0422; and Al 04-0787



The results of the previous research studies in this area have shown that during self-propagating high-temperature synthesis (SHS), the chance for the formation of MoSi<sub>2</sub> is clearly provided. By specifying the trend of the changes taking place during the tests of DTA–TG, a question arises as to why the trend of MoSi<sub>2</sub> formation during self-propagating high-temperature synthesis (SHS) is not observed in these tests.

The response to this question should be sought in the differences existing in the conditions of SHS and thermal analysis.

Compactness of the mixture of the raw materials, high rate of the combustion front, and consequently high heating rate in different layers of the raw materials as well as generation of the initial drive force for the start of the process by igniting the surface layer of the specimen by a heat source can be mentioned as the most noticeable properties governing the self-propagating high-temperature

synthesis. However, none of these conditions was satisfied during the performed thermal analyses. Therefore, under these conditions, the thermal analysis studies cannot provide effective results for the production of MoSi<sub>2</sub> intermetallic compound. The results of this research studies showed that with regard to the performance of thermal analysis tests in air atmosphere, oxidation of molybdenum particles was the most important factor for preventing the required conditions for the formation of MoSi<sub>2</sub>. However, owing to the ignition of the specimen (usually done by oxyethylene flame) in self-propagating high-temperature synthesis conditions (even in air atmosphere), the silicon particles undergo quick melting providing the required conditions for the dissolution of the solid molybdenum particles in the molten silicon before oxidation [18]. In addition, during the progress of the combustion phase, the high speed of this phase prevents the oxidation of molybdenum particles before the formation of MoSi<sub>2</sub>. Also, the

compactness of the specimens during SHS can be helpful, too. There are several ways to prevent this oxidation before the formation of the above compound during thermal analysis tests. For example, it is possible to prevent the oxidation of molybdenum particles by controlling the existing atmosphere. However, attention should be paid to this fact that the change of atmosphere brings about changes in the trend of the process and the conditions of the final product [19], and even in certain conditions, due to the presence of oxide reactions during the process, it should inevitably be performed in oxide atmosphere [10]. Regarding this issue, it seems that by compressing the specimen, increasing the heating rate, and providing similar conditions governing the SHS, it is also possible to prevent the oxidation of molybdenum particles. Consequently we have tried to study the effects of compressing, heating rate, and atmosphere on the trend of the performed changes during thermal analysis tests.

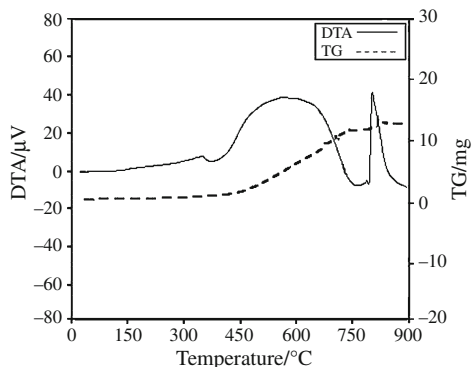
#### Effect of compressing

An amount of (Mo + 2Si) + 5 wt.% Al powdery mixture was subjected to the DTA–TG analysis after compressing (800 Mpa) at a heating rate of 20 °C/min up to 900 °C in air atmosphere. The DTA–TG curves for this study are presented in Fig. 8.

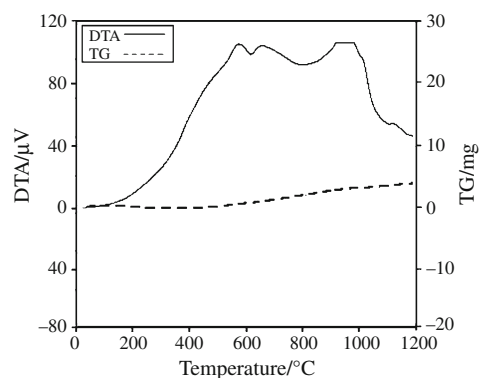
As is observed, the trend of changes reported by the curves in this figure is in fact similar to those pointed out in the previous analyses (Figs. 1, 4). Thus, it is not possible to prevent the oxidation of molybdenum particles during thermal analysis tests by compressing the specimens. To achieve this aim, there is definitely a need for other changes.

#### Effect of heating rate

In order to study the effectiveness of the heating rate on the results of these analyses, a compressed powder mixture



**Fig. 8** TG and DTA curves for the heating process of (Mo + 2Si) + 5 wt.% Al powders after compressing (800 Mpa) in air atmosphere at the heating rate of 20 °C/min up to 900 °C

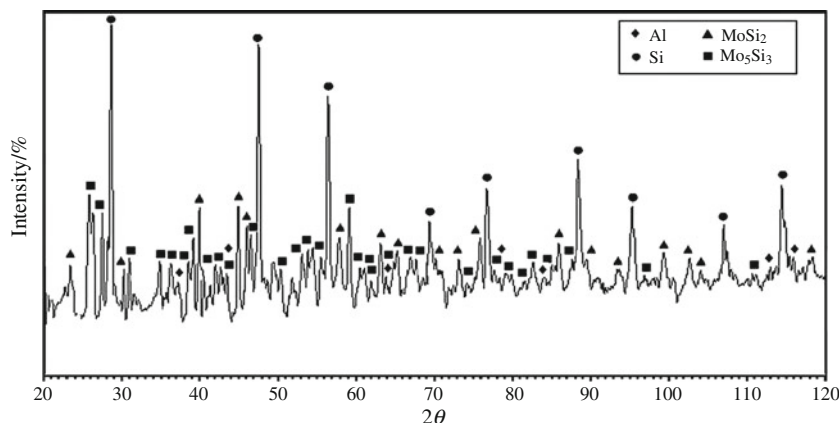


**Fig. 9** TG and DTA curves for the heating processes of (Mo + 2Si) + 5 wt.% Al powders after compressing (800 Mpa) in air atmosphere at the heating rate of 100 °C/min up to 1,200 °C

containing aluminum was exposed to the DTA–TG analysis in air atmosphere and heated up to the maximum temperature of 1,200 °C at a heating rate of 100 °C/min. The results are presented in Fig. 9. As is observed, there is no further trace of mass increase resulting from oxidation on the TG curve presented in this figure. In addition, the DTA curve related to this analysis reveals some differences compared to the previous curves (Fig. 6). These results indicate that by increasing in the heating rate up to 100 °C/min, the oxidation of the molybdenum particles is prevented. Now the question arises as to whether the trend of the changes presented in this analysis has been in the direction of the formation of MoSi<sub>2</sub> intermetallic compound or not.

For further study in this area, the products of this test were exposed to phase study. The resulting X-ray diffraction pattern is presented in Fig. 10. Although the Mo<sub>5</sub>Si<sub>3</sub> and MoSi<sub>2</sub> intermetallic compounds are observed in this X-ray diffraction pattern, it is still observed that silicon exists mostly in the remaining phase. In contrast, the remaining molybdenum is not observed among the products. This issue in a way indicates that molybdenum has been oxidized and vaporized again contrary to what the TG curve has depicted (Fig. 9). Although an increase in heating rate has prevented the oxidation of molybdenum particles within the temperature range of 400–800 °C, by observing the DTA curve (Fig. 9) it is clear that molybdenum particles are quickly oxidized and vaporized when the temperature reaches 800 °C, which in fact is because of oxidation and simultaneous evaporation of molybdenum particles, and because no mass change is observed on the TG curve. The heat released by this reaction reveals itself as an intense exothermic peak starting at 800 °C, but as is observed, the device is not able to completely register it. This is because the intensity of this peak is greater than the STA device resolution's capability to register.

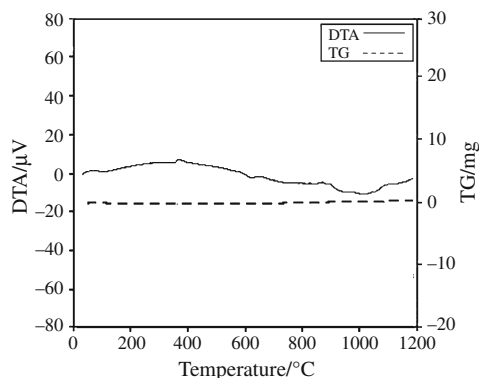
**Fig. 10** XRD patterns of the products obtained through thermal analyses of (Mo + 2Si) + 5 wt.% Al powders after compressing (800 Mpa) in air atmosphere at the heating rate of 100 °C/min up to 1,200 °C. The PDF numbers used are Si 26-1481; MoSi<sub>2</sub> 41-0612; Mo<sub>5</sub>Si<sub>3</sub> 34-0371; and Al 04-0787



Although an increase in the heating rate (up to 100 °C/min) caused the trend of the changes shift to the formation of MoSi<sub>2</sub>, it is again observed that this value of rate increase is not enough by itself and, under these conditions, the molybdenum existing in the powder mixture is oxidized and vaporized again.

#### Effect of atmosphere

To study the effect of atmosphere on the trend of the performed processes during the tests of thermal analyses, the powder mixture of Mo + 2Si was exposed to DTA–TG analysis under Ar atmosphere up to the maximum temperature of 1,200 °C at a heating rate of 20 °C/min. The results of the test are presented in Fig. 11. As is observed, there is no longer any sign of mass increase resulting from oxidation on the TG curve in this figure. In addition, the DTA curve related to this analysis shows pretty well that nothing specific has happened under these conditions, because the inert atmosphere of Ar fails to create the required conditions for the oxidation of molybdenum particles. Also, owing to the absence of the required conditions for the formation of MoSi<sub>2</sub>, it is observed that no



**Fig. 11** TG and DTA curves for the heating processes of (Mo + 2Si) + 5 wt.% Al powders after compressing (800 Mpa) in Ar atmosphere at the heating rate of 20 °C/min up to 1,200 °C

specific changes take place on the DTA curve (Fig. 11) in this regard. By revising the self-propagating high-temperature synthesis (SHS), it is clearly specified that the ignition of the specimen and the high rate of the combustion phase can play a fundamental role in the formation of the intended compound. As was pointed out earlier, the ignition of the specimen provides the chance for silicon melt, and molybdenum dissolution in this melt. A super-saturated solution of molybdenum, from which the MoSi<sub>2</sub> phase starts nucleation, is produced through the dissolution of molybdenum particles in silicon melt. In addition, a self-propagating wave starts advancing along the specimen accompanied by a high heating rate in the specimen.

High rate of the combustion phase and, consequently, the high heating rate in SHS conditions under oxide atmosphere provides no possibility for oxidation process in the specimen during the synthesis process. Providing these conditions for thermal analysis tests with the existing facilities is not feasible.

With regard to these results, it is clearly specified that the MoSi<sub>2</sub> formation process is greatly dependent on the type of the governing atmosphere and the heating rate. In order to produce a high-quality product, these two parameters should be strongly controlled during the production process of this product.

#### Conclusions

The results obtained from the DTA–TG analysis of the powder mixture of Mo–Si were studied in this research. The research findings revealed that

- The process followed in the thermal analysis tests of powder mixture of Mo–Si at low heating rates is not related to MoSi<sub>2</sub> and is in fact related to the surface oxidation of molybdenum particles.
- Formation of MoSi<sub>2</sub> is strongly affected by the type of the governing atmosphere and heating rate, and so the



failure to provide the existing conditions during the MoSi<sub>2</sub> self-propagating high-temperature synthesis in the tests of thermal analyses prevents the production of the intended product like (MoSi<sub>2</sub>) during the performance of this analysis. In other words, at high heating rates (SHS conditions), the type of atmosphere cannot have any specific effect on the trend of the formation of the intended compound. However, in low heating rates, the type of the atmosphere can play a great role in the trend of the process so that it can even result in the change of the trend of the process and formation of the not so preferred products.

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