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Investigation of the Effects of Oven-Chemical and Mechanically Activated Techniques on the In Situ Synthesis of $MoSi_2$-SiC Nanocomposites by Self-Propagating High-Temperature Synthesis

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ABSTRACT

The purpose of this study was to investigate suitable methods for the one-step synthesis of $MoSi_2$-SiC nanocomposites by self-propagating high-temperature synthesis. Therefore, oven-chemical and mechanically activated techniques were selected from the available options. First, the elemental raw materials molybdenum (Mo), silicon (Si), and graphite (C) were mixed in powder form and milled in stoichiometric ratios in a planetary mill for 0, 1, 3, and 6 h at a ball-to-powder ratio (BPR) of 10:1 and a rotational speed of 250 rpm. The resulting powder was compacted in a uniaxial press at 300 MPa. In parallel, combustion-aid samples were prepared using Mo-Si-Al raw materials. In the next step, the main samples (Mo-Si-C) and the combustion-aid samples (Mo-Si-Al) were assembled together and transferred to the combustion reactor at a temperature of 850 °C in an Ar atmosphere. After synthesis in the reactor, the samples were characterized by TEM, SEM, and XRD. Finally, a $MoSi_2$-SiC nanocomposite with crystallite sizes below 100 nm and the fewest undesirable phases was synthesized in situ from elemental raw materials by optimizing the parameters affecting the self-propagating high-temperature synthesis process.

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1. INTRODUCTION

Molybdenum disilicide has attracted great research interest due to its relatively low density (6.28 g/cm³), high melting point, high electrical conductivity, and very good oxidation resistance at high temperatures. $MoSi_2$-based materials are considered potentially useful for manufacturing high-temperature structural components. It is widely used in aerospace applications, high-temperature smelting, electronic components, high-temperature heating elements, potential structural components at elevated temperatures, and refractory materials that require high-temperature resistance, excellent oxidation resistance, and good thermal stability (Xu et al. 2010; Zhang et al. 2004; Potanin et al. 2020; Wu et al. 2017; Wang et al. 2025).

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Despite possessing unique electrical and thermal properties at high temperatures, $MoSi_2$ needs to be alloyed with metals or used as a composite with metals or ceramics in order to be applied in new engineering fields. The reason for alloying and composite fabrication is that $MoSi_2$ exhibits poor mechanical properties and weak oxidation resistance at low temperatures (Tayebifard, 1999 ; Tayebifard, 2024). SiC, as the second phase in composites, is one of the main candidates for reinforcing $MoSi_2$ (Tayebifard, 2006).

Compared with conventional methods for the synthesis of $MoSi_2$ and its composites, the Self-Propagating High-Temperature Synthesis method is a simpler process with a short reaction time, suitable efficiency, and inexpensive equipment. The purity of the product can even be higher than that of the raw materials because the samples experience very high temperatures during synthesis, causing impurities to evaporate completely. In addition, specific phases in multiphase systems can be synthesized selectively. All of these qualities have attracted the attention of many researchers to this method (Yazdani-Rad etal., 2003; Yazdani-Rad et al., 2002; Tayebifard et al., 2018). In this study, this process, Self-Propagating High-Temperature Synthesis, was used to synthesize the desired compounds.

Since SiC must be synthesized together with $MoSi_2$ in the desired composite, and initiating the reaction is, if not impossible, then extremely difficult, it is necessary to apply a technology that supports the initiation and propagation of the Self-Propagating High-Temperature Synthesis reaction throughout the material (Sedighi et al., 2024; Ganjali et al. 2014; Han et al. 2018). One of these methods is the oven-chemical technique. A novel process, referred to in the literature as oven-chemical or chemical oven Self-Propagating High-Temperature Synthesis (COSHS), was developed to prepare powders that cannot be obtained through conventional Self-Propagating High-Temperature Synthesis (Xu et al., 2006). In this approach, a mixture with a low-exothermic reaction is paired with another mixture having a highly exothermic reaction. When the highly exothermic reaction of the second mixture starts, it produces sufficient heat to force the first mixture to react (Maremaisupat et al., 1998; Jiang et al., 2018). In this work, samples with Mo-Si-Al composition are used as combustion aid.

On the other hand, nanotechnology has revealed new aspects of materials, providing new properties and applications. Similarly, composites are no exception; therefore, nanocomposites are recognized as one of the most practical groups of nanomaterials. As previously mentioned, the aim of this project was the preparation of $MoSi_2$-SiC nanocomposites by the Self-Propagating High-Temperature Synthesis method. One of the drawbacks of this method is excessive grain growth, which causes the product to be devoid of nanocrystallites (Yi et al., 1990; Levashov et al., 2017). To solve this problem, it has been proposed that the powder mixture first be milled in a planetary mill before being used to prepare the final samples. This approach is known as mechanical activation, and when the milled samples are subsequently synthesized by the SHS technique, the entire process is referred to as MASHS (Mechanical-Activated Self-Propagating High-Temperature Synthesis) (Levashov et al., 2017; Gras et al., 2001). However, excessive milling time or excessive mechanical work applied to the powder may have adverse effects; therefore, the milling time must be optimized (Gras et al., 2001; Ovali et al., 2017; Aminikia et al., 2013).

The purpose of this paper is to investigate a suitable method for the synthesis of $MoSi_2$-SiC nanocomposites by the Self-Propagating High-Temperature Synthesis method in a single step. In this regard, oven-chemical and mechanically activated technologies were selected from the available options. Finally, the effect of milling time on the phase composition and microstructural properties of the resulting nanocomposites is investigated.

2. MATERIALS AND METHODS

In this study, the reactive raw materials consisted of Mo, Si, and C powders for preparing the main samples, and Mo, Si, and Al powders for preparing the combustion-aid samples. The characteristics of these powders are presented in Table 1.

The raw material powders were weighed according to the desired composite composition ($MoSi_2$-SiC) and the combustion-aid composition ($Mo(Si_{1-x},Al_x)$, x = 0.5) (Tayebifard, 2006). The composite raw materials were then subjected to milling in a planetary mill. Milling times of 0, 1, 3, and 6 h, a ball-to-powder ratio (BPR) of

TABLE 1. Introduction of raw materials

Raw Materials	Symbol	Purity (%)	Size (μm)	Company manufactured	Production Code
Molybdenum	Mo	99.7>	-44	Riedel	13309
Silicon	Si	99>	-150	Merck	12497
Graphite	C	99>	-50	Merck	04206
Aluminum	Al	99	100-200	Fluka	06140

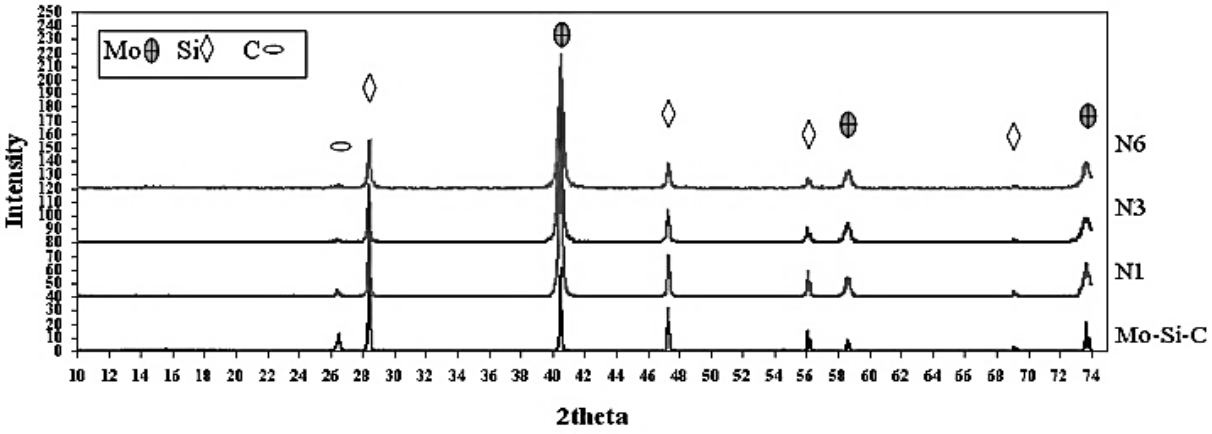

Figure 1. X-ray diffraction patterns of powder mixture before milling (Mo-Si-C), powder mixture after 1 hour milling (N1), powder mixture after 3 hours milling (N3), and powder mixture after 6 hours milling (N6)

10:1, and a rotational speed of 250 rpm were selected as the milling parameters (Zakeri et al., 2006; Mirvakili et al. 2012). The combustion-aid powders were mixed using a heater-magnetic stirrer in an organic medium (methanol). Next, the prepared powder mixtures from the two aforementioned formulations were compacted in a uniaxial press under a pressure of 300 MPa. Finally, each main composition sample ($MoSi_2$-SiC), together with a combustion-aid sample, was synthesized in a combustion reactor (assembled at the Materials and Energy Research Center) at 850 °C under an Ar atmosphere using the Self-Propagating High-Temperature Synthesis method (Tayebifard, 2006). The analyses performed on the samples included phase identification and crystallite size determination by XRD (Philips PW3710), microstructural investigations by SEM (Stereo Scan 360-Leica Cambridge), and microstructural and point chemical analyses by TEM (Philips, 200KV, FEG).

3. RESULTS AND DISCUSSION

Figure 1 shows the X-ray diffraction (XRD) patterns of the main raw materials used for the composite before milling and after 1, 3, and 6 h of milling. According to this figure, the intensities of the C (graphite) (PDF No. 8-0415) and Si (PDF No. 5-0565) peaks decrease rapidly relative to the Mo (PDF No. 4-0809) peaks as the milling time increases. This phenomenon may be attributed to the fact that Si powder is more brittle than Mo, while the C crystallites become broadened during milling. An interesting point is that no additional peaks, resulting from contamination caused by milling or possible reactions, are observed in any of the milled samples, and they are only present in the initial raw materials (Mo, Si, and C).

Furthermore, the crystallite sizes of the aforementioned powders were calculated using the Scherrer equation and peak broadening data obtained from the XRD patterns (Cullity, 2014; Neikov, 2019).

Table 2 shows the results of these measurements and calculations. As shown in the table, the reduction in Mo and Si crystallite sizes is quite evident with increasing milling time. Therefore, the objective of milling was achieved by preparing the raw material powder mixture within the nanocrystallite size range prior to synthesis by the Self-Propagating High-Temperature Synthesis method.

TABLE 2. The crystallite size of Mo and Si before and after different milling times

Milled powder Type	Mo-Crystallite Size(nm)	Si-Crystallite Size(nm)
Unmilled powder(Mo-Si-C)	92	144(>100)
Milled Powder at 1hour(N1)	61	114(>100)
Milled Powder at 3hour(N3)	53	110(>100)
Milled Powder at 6hour(N6)	51	90

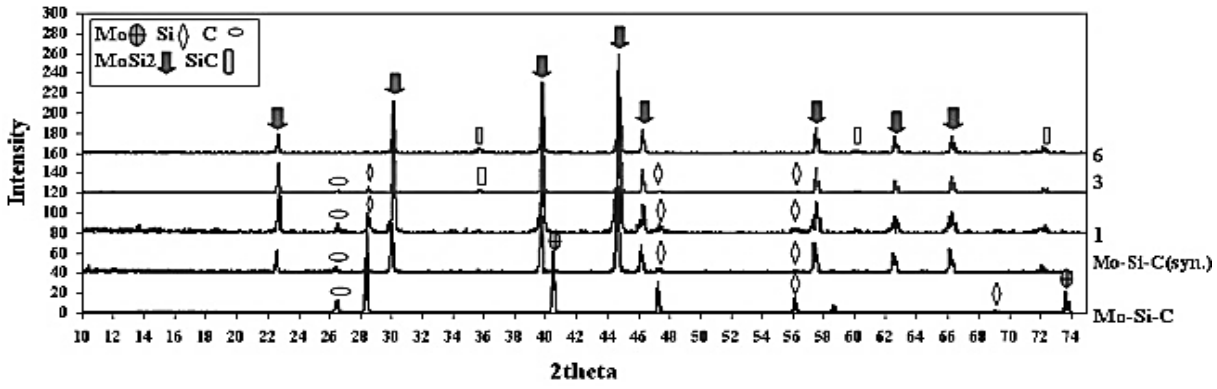

Figure 2. X-ray diffraction patterns (XRD) of: mixed as received powder (Mo-Si-C), synthesized samples: No milling (Mo-Si-C (syn.)), after 1 hour milling (1), after 3 hours milling (3) and after 6 hours milling(6)

In the next step, the diffraction patterns of the synthesized samples prepared from the mixed and milled raw materials at different milling times were analyzed and compared with those of the mixed raw material powders before milling. Figure 2 shows the X-ray diffraction (XRD) patterns of these synthesized samples.

According to this figure, the $MoSi_2$ phase (PDF No. 80-0544) appears as the major phase in all synthesized samples. Increasing the milling time of the mixed raw material powders altered the diffraction patterns of the synthesized samples such that the peaks corresponding to unreacted raw materials became weaker. Consequently, these peaks are not observed in sample (6), whereas the intensity of the SiC peaks (PDF No. 73-1665) has increased. This phenomenon can be attributed to the higher reactivity of the powders with increasing milling time. The Self-Propagating High-Temperature Synthesis reaction was facilitated by this activated powder, particularly in the case of SiC synthesis (Ganjeh et al., 2015; Gras et al., 1999). Therefore, according to the diffraction pattern of sample (6), it can be concluded that the composite consists only of the desired phases, $MoSi_2$ and SiC.

The crystallite sizes of the synthesized samples were calculated using the Scherrer equation and peak broadening data obtained from the XRD patterns (Cullity, 2014). Table 3 presents the results of these measurements and calculations. According to this table, the crystallite sizes of the synthesized samples increased compared with the crystallite size of Mo (Table 2), which in fact forms the main skeleton of the samples after synthesis (Tayebifard, 1999). Despite the inherent nature of the Self-Propagating High-Temperature Synthesis process, the final products, samples (3) and (6), formed crystallites smaller than 100 nm. Therefore, it can be concluded that a $MoSi_2$-SiC nanocomposite was successfully synthesized.

The results of the present research can be compared with those reported by Zakeri et al. In that study, mechanical alloying (MA) was used; however, the raw materials and the final product were similar to those employed in the present work. Although the desired product, a $MoSi_2$-SiC nanocomposite, was obtained in both studies, the present work offers several advantages over that of Zakeri et al., as outlined below. First, the energy applied during milling of the raw powder mixture in Zakeri's study was significantly higher than that used in the present work. For example, the rotational speed (RPM) was selected as 750 and 250, respectively. Similarly, the ball-to-powder ratio (BPR) was 15:1 and 10:1, respectively. Finally, the milling times ranged from 10 to 20 h in Zakeri's study, whereas a maximum milling time of only 6 h was used in the present work. Second, in Zakeri's research, the intermediate Mo_5Si_3 phase was synthesized in addition to the desired phases, whereas only the desired phases ($MoSi_2$ and SiC) were detected in the present study. Third, due to the higher energy input in Zakeri's study, the final product contained a greater amount of Fe impurity (Zakeri, et al. 2012).

TABLE 3. The crystallites size of $MoSi_2$-SiC composite based on milled powders at different milling times

Synthesized sample type	$MoSi_2$-SiC Crystallite size (nm)
Based on N1 (1)	120 (>100)
Based on N3 (3)	95
Based onN6 (6)	86

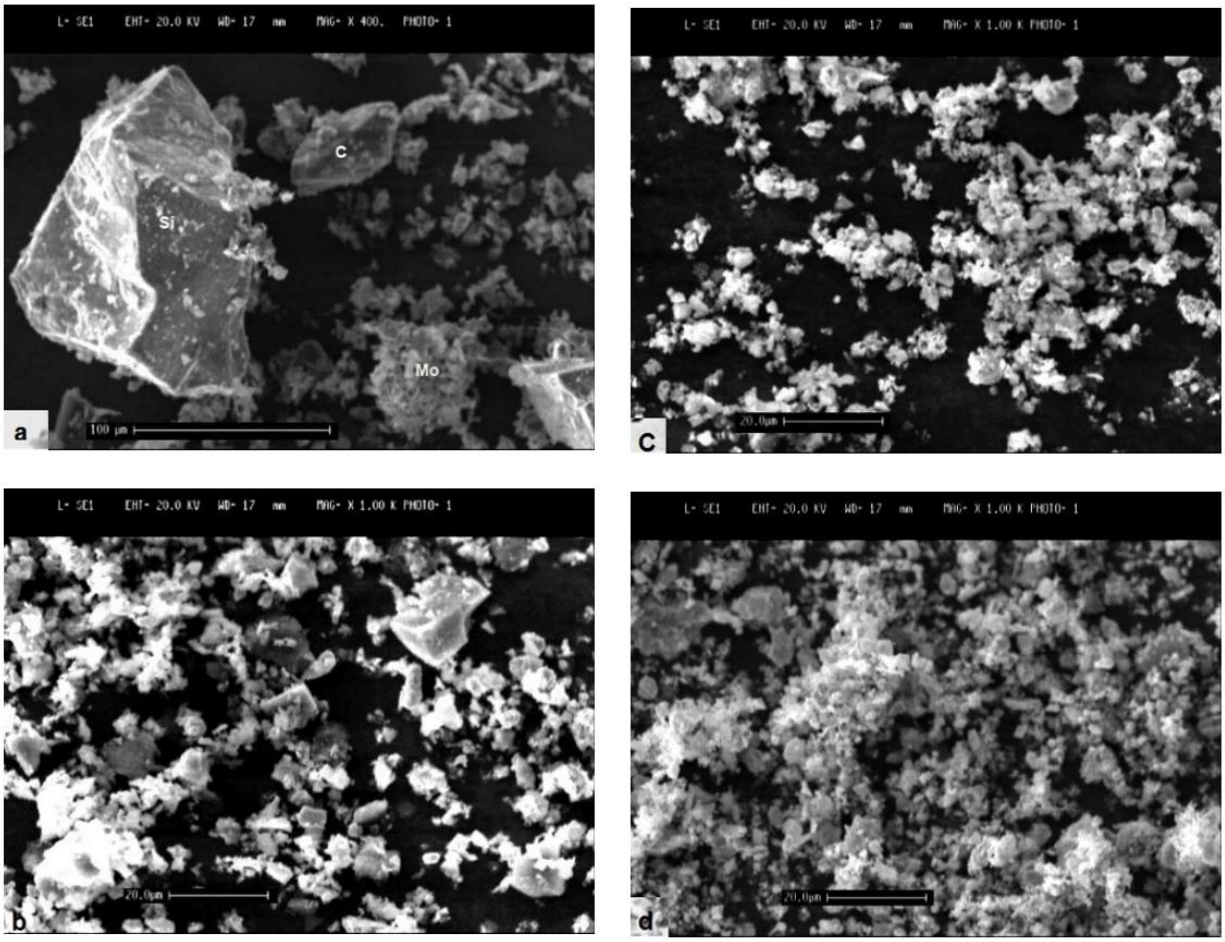

Figure 3. SEM micrograph: a) the mixed powder before milling (Mo-Si-C), b) the mixed powder after 1 hour milling time (N1), c) the mixed powder after 3 hours milling time (N3), and d) the mixed powder after 6 hours milling time (N6)

The green and synthesized samples were analyzed by microstructural examination and point chemical analysis (EDAX). Figure 3 shows the microstructures of the powders before milling and after different milling times. According to this figure, the particle sizes decreased with increasing milling time, although this phenomenon was accompanied by agglomeration. In Figure 3(a), the large particles are Si (Figure 3(b)), the flake-shaped particles are C (graphite), and the small spherical particles are Mo (Figure 3(c)), as confirmed by EDAX analysis.

As mentioned previously, the milled samples were subsequently synthesized. Figure 4 shows the synthesized samples prepared without and with prior milling. Examination of this figure demonstrates that milling the raw materials has a positive effect on the final microstructure of the samples. Accordingly, sample (6), which was milled for the longest duration (6 h), exhibits the finest microstructure among all the samples.

After analysis of the samples by SEM, the TEM technique was also used to confirm the SEM results.

Figure 5(a) shows the microstructure of the sample in bright-field mode. In this figure, it can be seen that the agglomerates are nanosized and are consistent with the results presented in Table 2. Figure 5(b) shows the EDAX analysis pattern of point E7 in Figure 5(a). According to Table 4, the combined XRD and EDS results (based on TEM analysis) indicate the presence of both $MoSi_2$ and SiC phases in the analyzed sample. Therefore, it can be concluded that a $MoSi_2$-SiC nanocomposite was successfully synthesized in this research. In summary, this research is innovative in several respects. First, a $MoSi_2$-SiC nanocomposite with unique properties was synthesized in situ. Second, this nanocomposite was synthesized by the SHS method in a minimum processing time and with the fewest undesirable phases. Third, two advanced approaches, MASHS and the chemical oven technique, were employed to achieve the optimal product at a relatively low synthesis temperature. Fourth, in addition to conventional XRD and SEM analyses, detailed TEM analysis was used to determine the morphology of the synthesized product (Kasraee et al., 2024).

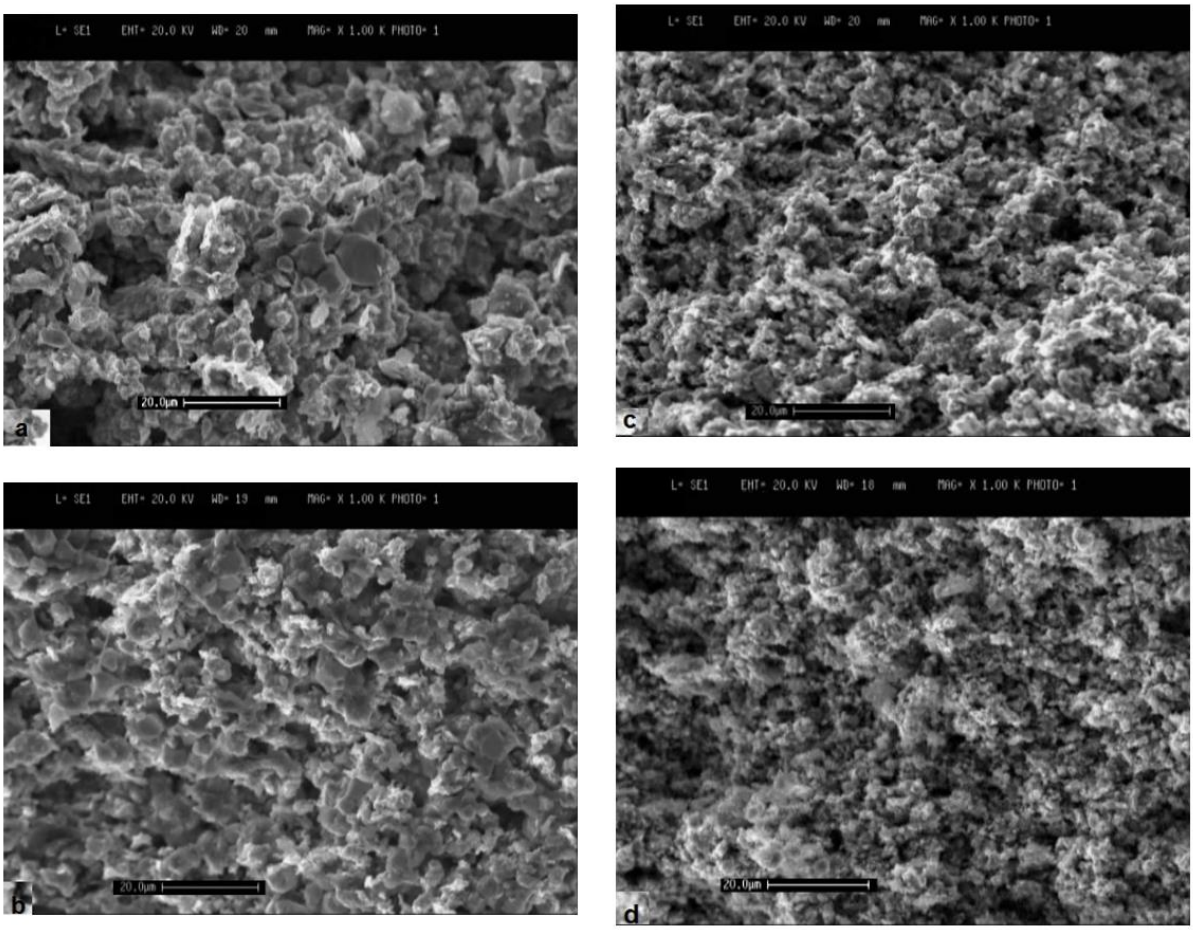

Figure 4. SEM micrograph of the synthesized samples composed of powders including: a- unmilled, b- after 1 hour milling time (1), c – after 3 hour milling time (3), d - after 6 hour milling time (6)

TABLE 4. The point chemical analysis (EDAX) of E7-cited point on figure 5-a and figure 5-b based on K, L picks levels

Element	Cu*	Mo	Si	O	C
Weighting Percent	24.23	1.82	25.60	0.61	47.74

* From Cupper grid

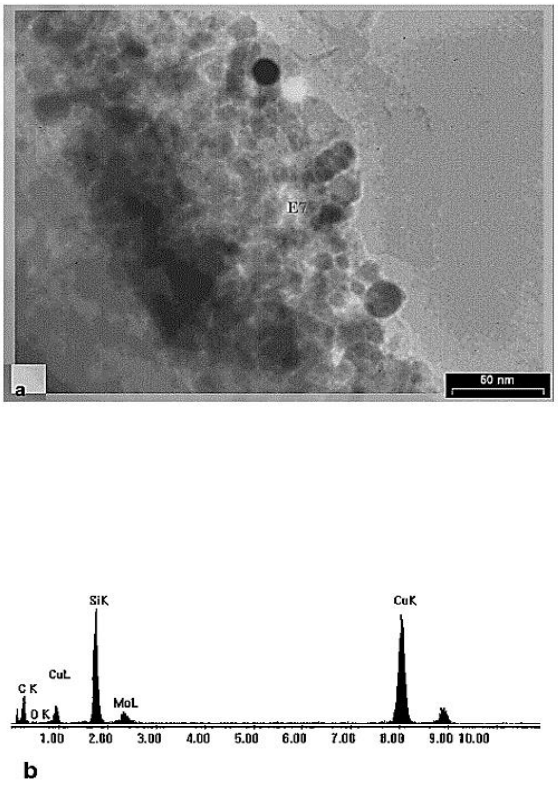

Figure 5. a- TEM micrograph (bright-field) of the synthesized sample (6), b- pattern of EDAX analysis of the point (E7) on micrograph (a)

4. CONCLUSIONS

According to the X-ray diffraction patterns, the following results were obtained: A longer milling time resulted in lower intensities of the Si and C peaks relative to the Mo peaks. No additional peaks associated with impurities introduced during the milling process were observed in any sample. The diffraction patterns of the synthesized samples prepared from powders subjected to longer milling times showed a reduction in the amount of unreacted phases, accompanied by an increase in the intensity of the SiC peaks. As a result, after 6 h of milling, only the $MoSi_2$ and SiC peaks were observed. The measurements of the crystallite sizes of the milled powders and the synthesized samples prepared from these powders showed that, although the crystallites of the synthesized samples exhibited slight growth compared with those of the milled powders, their sizes remained within the nanometer range. According to the SEM analysis, the following result was obtained: With increasing milling time, the grain size of the synthesized samples decreased. According to the TEM analysis, the following results were obtained: The grain size of the milled powder was in the nanometer range, which confirms the XRD results. Based on the results obtained in this research, $MoSi_2$-SiC nanocomposites were successfully synthesized.

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