



Effect of $\text{Al}_{59}\text{Cu}_{25.5}\text{Fe}_{12.5}\text{B}_3$ Quasi-crystals on Microstructure and Flexural Strength of Aluminum Matrix Composites Prepared by Spark Plasma Sintering Method

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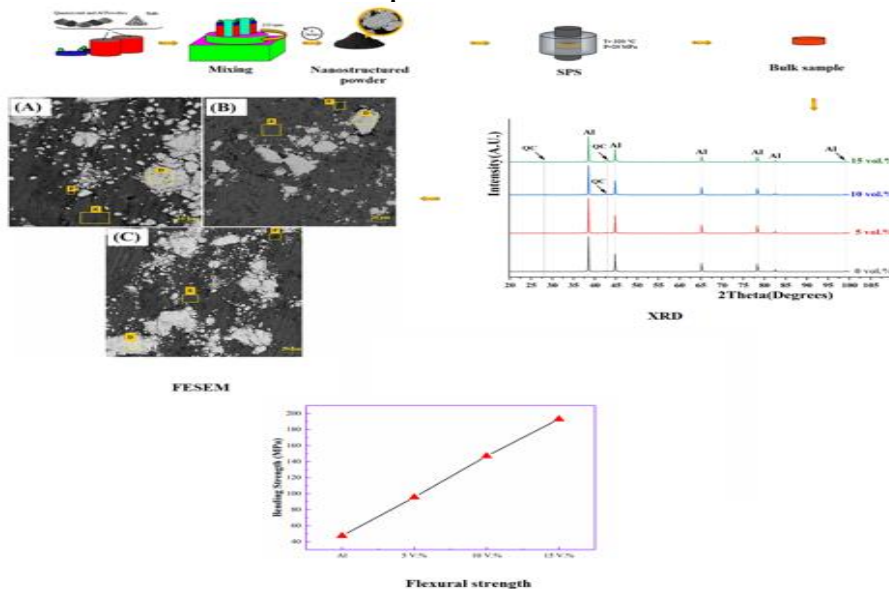
Mechanical Properties

ABSTRACT

In this study, Al-based composites reinforced with $\text{Al}_{59}\text{Cu}_{25.5}\text{Fe}_{12.5}\text{B}_3$ quasicrystal (QC) were prepared by spark plasma sintering (SPS) method. Microstructural and mechanical properties were examined. It is observed that with addition of quasi-crystalline reinforcement the intensity of the quasi-crystalline peak has increased. Also, it is observed that by performing spark plasma sintering, the quasi-crystalline particles maintain their stability. Due to low temperature of the process and the short time of spark plasma sintering, the occurrence of destructive phases within the quasi-crystal has been prevented. Based on field emission scanning electron microscopy (FESEM) images, the distribution of quasi-crystalline particles at the sample level has increased. In addition, the mechanical properties are improved by increasing the quasi-crystalline particles. Therefore, the sample with 15 vol.% of quasi-crystal has better results than other samples in improving microstructural and mechanical properties and it can be considered as an optimal sample with suitable practical properties.

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Graphical Abstract



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1. INTRODUCTION

After classifying solids into two categories of crystalline materials and amorphous materials, quasi-crystals were introduced as the third type of solids by Avar et al. [1] and Shechtman et al. (1, 2). The quasi-crystals are among the new materials that have a non-periodic long-range order (3, 4). There is a repetitive periodicity in the arrangement of atoms with a forbidden rotational symmetry for the crystals in the quasi-crystalline material. Symmetries in quasi-crystals have been observed. Icosahedral, octagonal, decagonal and dodecagonal structural are among the most important quasi-crystals (2, 4, 5). There are several methods for the synthesis of the quasi-crystalline materials, these methods are: melt spinning, casting, mechanical alloying, gas atomization, physical vapour deposition (PVD), sol-gel, electrodeposition, gas evaporation, plasma spray and laser- or electron-beam superficial fusion and electron irradiation (4). One of the most widely used methods for the synthesis of quasi-crystals is mechanical alloying (MA) (3, 6). This method is a powder method that produces homogeneous materials from a mixture of pure powders. Mechanical alloying method has been used over the years as an acceptable fabrication method for the development of a variety of advanced materials such as nanomaterials, quasi-crystals, amorphous materials and nanocomposites (7, 8). The most important mechanisms of this method are repeated welding, fracturing, and re-welding of particles (9). Metal matrix composites (MMCs) consist of two main components including the matrix phase and reinforcement phase. The purpose of placing a high-performance secondary phase in these materials is to create a combination of aspects that would not be possible by the components alone. The reinforcement phase is continuous or discontinuous in the matrix. The reinforcement phases are continuous or discontinuous in the matrix, which may make up about 10 to 60 volume percentage of composite. The purpose of creating reinforcement phase in these materials is to achieve the desired properties that these properties will not be possible by the components alone (10). One of the most important and widely used metal matrix composites are aluminum matrix composites (AMCs) (10). Aluminum matrix composites are advanced engineering materials that have superior properties compared to conventional aluminum alloys (11). Due to high hardness, high thermal conductivity, low thermal expansion coefficient, wear resistance, impact resistance, corrosion resistance, low density, strength to weight ratio, relatively reasonable price and isotropic properties, these composites have many applications in various industries (11, 12). Reinforcements can be particles, layers, fibers or interpenetrating type. Also, according to the type of reinforcement, the composite can be classified into

laminar composite, flake composite, fiber-reinforced composites, filled composite and particle-reinforced composite (10). Particle-reinforced composites are divided into two categories. In the first category, the heat treatment process leads to chemical reactions between the reinforcement phase and the matrix phase, which results in a homogeneous distribution of the stable reinforcement phase in the matrix. In the second category, reinforcement particles are embedded in a metal matrix, and the best way to make this type of composite is to use powder metallurgy (13, 14). Recently, the use of quasi-crystalline materials as reinforcement particles in aluminum-based composites has received much attention (15, 16), because quasi-crystalline alloys and composites containing quasi-crystalline phase improve some properties due to their complex atomic structure, unique mechanical and tribological properties, and special thermal behavior (17, 18). These properties make them suitable for applications such as surface coatings, reinforcement particles in composites and catalytic applications (4, 19, 20). Conventional sintering methods such as hot press, high temperature extrusion, hot isostatic press lead to grain growth which affects the properties of the final product. Spark plasma sintering (SPS) method is one of the techniques using powder metallurgy (21, 22). Compared to conventional sintering techniques, the SPS method can be very effective due to its unique sintering mechanisms. In this method, pressure and pulsed current simultaneously are used to achieve high density components (21, 23, 24). In the SPS method, momentary melting and evaporation on the particle surfaces leads to the formation of necks between the particles (22). When particles are in contact with each other, a high-density electric current passes through these particles, causing a bond between the particles. Uniaxial pressure also causes plastic deformation, which ultimately increases the density (25). Using this method, high density samples can be obtained at very low temperatures and times (26). Excessive interfacial diffusion can be avoided by using this method because the quasi-crystals exist only in a small composite range (27). Amini et al. (3) synthesized $Al_{67}Cu_{20}Fe_{10}B_3$ quasi-crystalline alloys using mechanical alloying method. They studied the microstructural and structural evolutions of these materials at different times of the mechanical alloying process. Their results showed that the stable AlCuFeB single quasi-crystalline phase can be synthesized using high energy planetary ball milling in a short time from mechanical alloying process (around ~ 4 hours). Yong et al. (28) achieved the quasi-crystalline phase using the method of mechanical alloying and heat treatment. Asahi et al. (29) and Tsai et al. (30) also synthesized the quasi-crystalline phase by mechanical alloying method. According to the findings of other researchers, the compositional range from 10 to 14 atomic percent of iron and 20 to 28 atomic percent of

copper is a very suitable range for the formation of icosahedral phase (4, 31, 32). In this compositional range, the peritectic reaction between $\lambda 2$ -Al₃Fe and β -AlFe(Cu) phases with the remaining molten liquid is reported to be the main reason for the creation of this quasi-crystalline icosahedral phase (4). The quasi-crystalline icosahedral phase in aluminum-copper-iron system has high hardness and low fracture toughness (33, 34). It is also reported that adding boron to this alloy system can significantly change the brittleness, hardness and toughness of these alloys (35, 36). In addition to these properties, boron causes more stability of the quasi-crystalline icosahedral phase and reduces the coefficient of friction and electrical resistance compared to alloys without boron (35, 37). Due to the special crystal structure and special properties, quasi-crystals have a wide variety of applications. One of the most important potentials of quasi-crystals is as a catalyst for steam-reforming of methanol (4). Also, quasi-crystals can be used in thermometry and heat flow detection due to their temperature-dependent electrical conductivity (4, 38). Another application of quasi-crystals is light absorption. Thin layers of these materials are suitable as selective absorbers in solar thermal applications. These materials can also be used in glass and window applications, thermal insulation plates, and electrical insulation (4). In another research by Amini et al. [42] the effect of quasi-crystal on solar properties was investigated. In this research, they obtained a quick method for the synthesis of the quasi-crystalline phase, based on which there was no need for annealing operations and complex methods for the synthesis of the quasi-crystalline phase. Also, their results showed that the quasi-crystalline phase increased the amount of sunlight in solar. Addition of quasi-crystals to aluminum alloys leads to new properties in these materials. Lityńska-Dobrzyńska et al. (39) prepared aluminum matrix composites with Al₆₅Cu₂₀Fe₁₅ quasi-crystalline reinforcement phase with different percentages of the quasi-crystalline phase (20, 40 and 60 wt%). In order to prepare these composites, they used the hot vacuum pressing method. Their results showed that with increasing amount of quasi-crystalline phase, hardness and compressive strength of the composite increased (173 HV_{0.5} and 370 MPa, respectively). They also reported that the friction coefficient of these composites depends on the composition (in the range 0.5-0.7). Sainfort and Dubost (40) used icosahedral Al-Li-Cu as reinforcement particles in the aluminium based MMCs. Tsai et al. (41) created composite material that contained quasi-crystals as reinforcement particles using powder metallurgy. It has also been shown that the hardness of this type of composite increased by increasing the quasi-crystalline reinforcement particles up to 5 times compared to pure aluminum. Therefore, the use of quasi-crystalline reinforcement particles in aluminum base composites has been proposed in order to overcome the

low surface hardness of aluminum and increase the wear resistance of these materials (27). In this research, our goal was to synthesize Al₅₉Cu_{25.5}Fe_{12.5}B₃ quasi-crystalline compound using a new method without annealing and then to obtain Al/Al₅₉Cu_{25.5}Fe_{12.5}B₃ composite for the first time using SPS method. Also, according to the research of others, we realize that others use higher percentages of quasi-crystals as reinforcement, but considering that this project is a field for the industrial and practical use of quasi-crystals in the industry, from the volumetric percentages The bottom is used as reinforcement (5, 10, and 15) to investigate the effect on microstructure and mechanical properties.

2. MATERIALS AND METHOD

The raw materials of this research included Aluminum (purity percentage 99% < , particle size = 3 to 5 μ m), Copper (purity percentage 99.8% < , particle size = 14 μ m), Iron (purity percentage 99% < , particle size = 45 μ m >) and Boron (purity percentage 99% < , particle size = 5 μ m) powders supplied by Aldrich company. Also, the Al₅₉Cu_{25.5}Fe_{12.5}B₃ quasicrystal composition used in this study was prepared using the optimal research process by mechanical alloying method (42). Therefore, before the SPS process, first 4 samples were prepared according to the specifications of TABLE 1 and then the powders of quasicrystal and aluminum are mixed using ball-milling at 210 rpm for 4 hours. Composite samples of aluminum matrix were obtained by SPS method at a synthesis temperature of 320 °C and pressure of 20 MPa. Also, Figure shows a schematic of the process of sintering a bulk sample.

Phase analysis was carried out by a Philips MPD 1880 X-ray diffractometer (XRD) system with Cu-K α radiation at 40 mA and 40 kV. The composition and microstructure of the sintered pellets were investigated by a JEOL JSM-5600LV field emission scanning electron microscope (FESEM), and Oxford energy dispersive spectroscopy (EDX) attachments with them. To evaluate the mechanical properties of the prepared samples, three-point flexural strength (Santam-STm 20) test was performed. It should be noted that three samples were used to reduce the measurement error for each test.

TABLE 1. Specifications of samples

Number	Combination
1	Al
2	Al + 5 vol. % Al ₅₉ Cu _{25.5} Fe _{12.5} B ₃
3	Al + 10 vol. % Al ₅₉ Cu _{25.5} Fe _{12.5} B ₃
4	Al + 15 vol. % Al ₅₉ Cu _{25.5} Fe _{12.5} B ₃

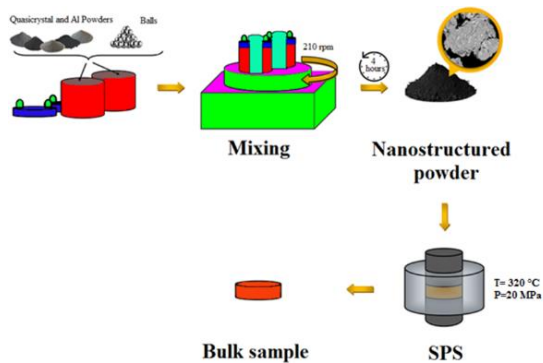


Figure 1. Schematic of the process of sintering a bulk sample

3. RESULTS

Figure 2 shows for samples with different percentages of quasi-crystalline phase, there are only peaks related to the aluminum phase (cubic structure) as well as the quasi-crystalline phase. Therefore, by performing the sintering process by SPS method, the peaks related to the quasi-crystalline phase have not been eliminated and this phase has maintained its stability during the SPS process. In other words, in the SPS process, there is no interfacial reaction for this type of composite. It has been reported that in Al/Al-Cu-Fe composites, the ω -tetragonal phase is also formed due to the reaction between the quasi-crystalline particles and the aluminum phase (43). However, as can be seen, the sintering process using SPS method has prevented the formation of ω phase due to lower temperature and shorter sintering time. These results are consistent with the other researchers (27). Al/QC composites are stable in a large temperature regime and phase transformations occur at high sintering temperatures (43). With the occurrence of these phase transformations, the amount of quasi-crystalline phases also decreases. Kenzari et al. (44) concluded that the quasi-crystalline phase transformations to the ω phase occur at approximately 450°C. It has also been reported that the reaction between aluminum and the quasi-crystalline phase occurs at approximately 482 °C (43).

Figure 3 shows electron microscopy images for composites with different percentages of quasi-crystalline phase. Also, Table 2 summarized the results of EDX elemental analysis from different regions (as shown in Figure 3). As can be seen for the sample with 5 vol% of the quasi-crystalline phase (region D in Figure 3 A), the amount of aluminum is equal to 68.03 at%, and the amounts of copper and iron are equal to 18.55 and 13.42 at%, respectively. Also, for samples with 10 and 15 vol% of quasi-crystalline phase, the amount of elements in regions D are very similar to samples with 5 vol% of quasi-crystalline phase. Therefore, the white areas (regions D in Figure 3) are related to quasi-

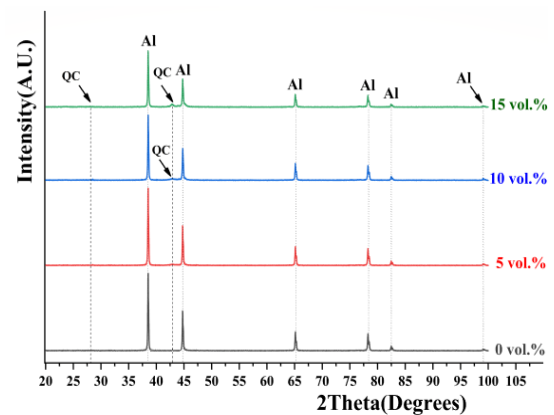


Figure 2. X-ray diffraction patterns for samples with different volume percentages of the quasi-crystalline phase in the aluminum matrix

crystalline reinforcement particles. In region E, for sample with 5 vol% of the quasi-crystalline phase (region E in Figure 3 A), the amounts of aluminum, copper and iron are 96.90, 1.98 and 1.12 at%, respectively. In regions E, the amount of the elements for samples with 10 and 15 at% of the quasi-crystalline phase are almost similar to samples with 5 vol% of the quasi-crystalline phase. Therefore, the dark gray areas (regions E in Figure 3) are related to the aluminum matrix phase. In region F, for the sample with 15 vol.% Of the quasi-crystalline phase (region F in Figure 3 C), the amounts of aluminum, copper and iron are 52.34, 0.33 and 0.31at%, respectively. As can be seen, reinforcement particles are scattered in the aluminum matrix. It can also be seen that the quasi-crystalline particles are agglomerated together. In addition to these elements, the elemental EDX analysis for this region also indicates the presence of the oxygen. For samples with 5, 10 and 15 vol% of the quasi-crystalline phase, the amount of oxygen in the F regions are 39.61, 37.77 and 46.97at%, respectively. As shown in Figure 3, the F regions are mostly formed at the interface between the quasi-crystalline phase and aluminum. It is reported that at the interface between the quasi-crystalline particles and aluminum, an amorphous oxide layer is formed in these composites, which acts as a strong bond between the quasi-crystalline particles and the aluminum matrix (27, 45). According to Figure 3, it is also observed that agglomerates of quasi-crystalline particles have formed in some areas. In addition, pores are observed in some areas, which can be due to the time of the mechanical alloying process and the temperature and sintering time.

The Flexural strength results of the pure Al sample and the samples with different percentages of the quasi-crystalline phase are shown in Figure 4. Due to the fact that aluminum is a soft and ductile material, during the bending test, brittle fracture does not occur in this sample.

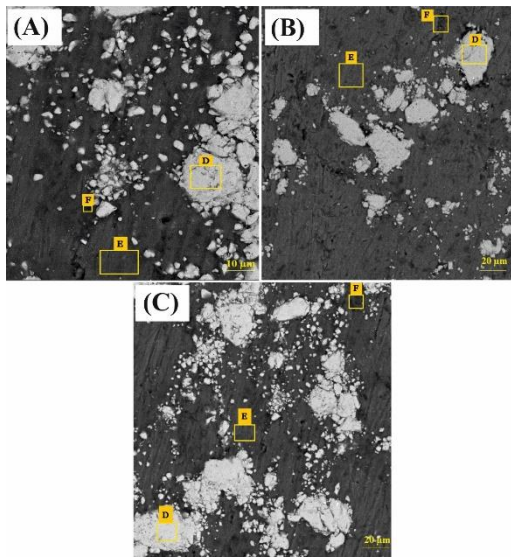


Figure 3. Scanning Electron microscopy images of composites with different percentages of quasi-crystalline phase A) 5vol.% of the quasi-crystalline phase B) 10vol.% of the quasi-crystalline phase and C) 15vol.% of the quasi-crystalline phase

TABLE 2. Results of EDX elemental analysis for samples with different percentages of quasi-crystalline phase

Samples	Phase areas	Al (at%)	Cu (at%)	Fe (at%)	O (at%)
5vol.%	D	68.03	18.55	13.42	
	E	96.90	1.98	1.12	
	F	59.42	0.53	0.44	39.61
10vol.%	D	68.46	18.02	13.52	
	E	97.59	1.39	1.02	
	F	49.57	0.54	0.39	37.77
15vol.%	D	69.03	17.61	13.36	
	E	98.69	0.73	0.58	
	F	52.34	0.33	0.31	46.97

However, in composite samples with different percentages of quasi-crystalline materials, it is observed that by performing the flexural strength test, the samples break brittle. And the fracture in these samples is of the brittle fracture type. Based on this, it is observed that composite samples do not have a plastic region, Accordingly, for the Al sample, the elastic region is examined and compared with other samples. It is observed that in the elastic region, the flexural strength for Al sample is about 47.30 MPa and for composite samples reinforced with 5, 10 and 15% of the quasi-crystalline phase are 95.51, 146.83 and 192.92 MPa, respectively. Therefore, with increasing amounts of

reinforcing quasi-crystalline materials improve the mechanical properties (46). One of the factors affecting the final properties of composites is the interface between the reinforcing phase particles and the matrix phase. In such a way that a joint with strong adhesion leads to the distribution and transfer of force from the matrix to the reinforcing particles, which leads to an increase in modulus and strength. A suitable joint is created by the wetting of the particles by the liquid phase during the sintering process. Other factors affecting the final properties of the composite are the surface reaction at the interface of the reinforcing particles with the matrix phase. This reaction due to its products and factors such as the adhesion of reactive products to the matrix and reinforcing phase, thermal expansion coefficient and strength of reactive products, etc. can have a positive or negative effect on the mechanical properties of the composite (47). Also, the final mechanical properties of the composite are affected by the joint quality season of the reinforcing particles with the matrix phase, which depends on factors such as the adhesion of the phases in the composite, the thickness and amount of reaction products in the joint season, and so on (48).

The level below the applied load diagram in terms of displacement in the 3-point flexural strength test indicates the fracture energy, which is also a measure of toughness for the samples (48-50). Figure 5 shows the changes in applied load in terms of displacement in the flexural strength test of composite samples. Also, Table 3 presents the results related to the flexural strength of the samples. Therefore, it is observed that fracture energy, elastic modulus and stress increase with increasing amount of reinforcement in composite samples (51). But the percentage of elongation in the sample with 10% by volume is higher than other samples. In addition, it is observed that the amount of toughness in samples 10 and 15 is almost equal, while in the sample 15% of fracture energy, elastic modulus and stress is more than 10% of the sample.

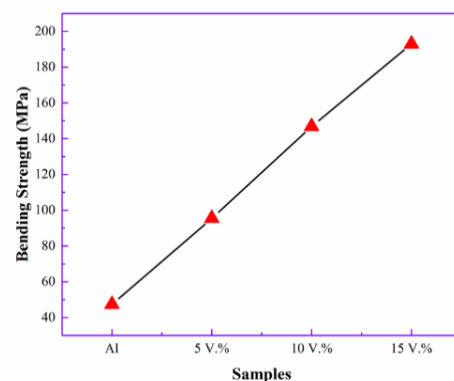


Figure 4. Flexural strength changes in the elastic region for pure aluminum sample and samples with different percentages of quasi-crystalline materials

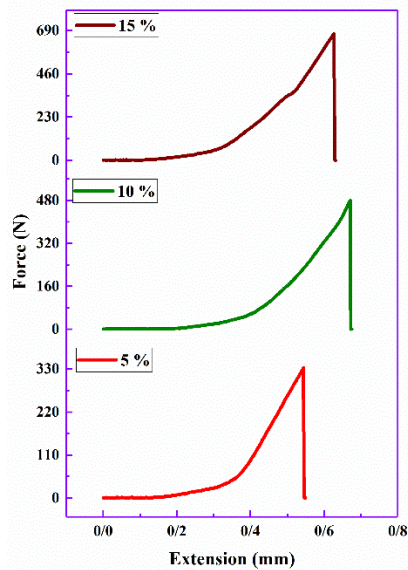


Figure 5. Load changes in terms of displacement in flexural strength test of samples with aluminum reinforcement reinforced with 5, 10 and 15 vol% quasi-crystalline

TABLE 3. Results related to flexural strength for samples with different percentages of quasi-crystalline phase

Sample	Stress (MPa)	Elongation (%)	Module (MPa)	Energy (J)	Elastic Module (MPa)
2	14.74	2.72	541.91	90.63	144.71
3	22.02	3.35	656.47	160.94	171.61
4	29.90	3.31	953.39	210.92	296.70

4. CONCLUSIONS

In this research, the microstructural and mechanical properties of $Al_{59}Cu_{25.5}Fe_{12.5}B_3$ composite samples synthesized by SPS method were investigated and the following results were obtained:

1. According to the XRD results, the quasi-crystalline particles maintain their stability due to the use of the SPS method and prevent the formation of the destructive ω phase in the composite sample.
2. According to the FESEM images, with the increase of quasi-crystalline particles in the sample, in addition to the increase in dispersion, the accumulation of quasi-crystalline particles is observed in some areas, which may be due to short mixing or sintering temperature and time.
3. According to the Flexural strength results, due to the high hardness and excellent properties of the quasi-crystalline particles, it is observed that by adding their amounts in the composite samples, it increases the fracture energy, stress and toughness.

According to the obtained results, it is possible to investigate the practical use cases in the industry and their properties in the future research of quasi-crystals with volume percentages in this research. In addition, other properties such as corrosion and wear can be investigated for this research.

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Persian Abstract

چکیده

در این مطالعه، کامپوزیت‌های زمینه Al تقویت‌شده با ساختار شبه بلور $Al_{59}Cu_{25.5}Fe_{12.5}B_3$ به روش نف جوشی پلاسمای جرقه‌ای (SPS) تهیه شدند و خواص ریزساختاری و مکانیکی آن‌ها مورد بررسی قرار گرفت. مشاهده شد که با افزودن مقدار تقویت کننده شبه بلور، شدت پیک فاز شبه بلور افزایش یافت. همچنین با انجام SPS ذرات شبه بلور پایداری خود را حفظ کردند و به دلیل دمای پایین فرآیند و زمان کوتاه SPS فازهای مخرب در شبه بلور به وجود نیامد. علاوه بر این، با توجه به تصاویر میکروسکوپ الکترونی روبشی (FESEM)، توزیع ذرات شبه بلور در سطح نمونه افزایش یافت. خواص مکانیکی نیز با افزایش درصد حجمی شبه بلور بهبود یافت و نمونه با ۱۵ درصد حجمی شبه بلور به دلیل خواص مکانیکی و ریزساختاری بهتر نسبت به سایر نمونه‌ها، به عنوان نمونه بهینه با خواص کاربردی مناسب انتخاب شد.