

INFLUENCE OF DIRECTIONAL SOLIDIFICATION ON THE MICROSTRUCTURE OF IN-SITU FeSi–NiAl COMPOSITES

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Abstract

This study investigates advanced composite materials consisting of a NiAl matrix reinforced with an FeSi phase, which are considered promising candidates for next-generation tool applications, with the potential to replace conventional high-speed steels alloyed with tungsten and cobalt. The alloys were synthesized via vacuum induction melting followed by centrifugal casting. To further tailor their microstructure, an experimental alloy with a 1:1 mass ratio of matrix to reinforcing phase was subjected to directional solidification using the Bridgman method. The process was carried out at a temperature of 1450 °C with a holding time of 30 minutes, followed by controlled crystallization at a rate of 100 mm/h. Microstructural characterization was performed using the light optical microscopy (LOM) and scanning electron microscopy (SEM). The chemical composition was determined by energy-dispersive X-ray spectroscopy (EDX), and the microhardness was measured using the Vickers indentation method. In the as-cast state, the alloy exhibited a typical in-situ composite microstructure consisting of primary NiAl-based dendrites and interdendritic regions containing lamellar eutectic structures and discrete reinforcing phases. The eutectic structures were composed of alternating FeSi- and NiAl-based phases, while certain interdendritic areas were occupied by an FeSi-based phase with only minor Ni and Al content. Directional solidification via the Bridgman method, which enables controlled solidification through precise regulation of the temperature gradient and solidification front velocity, significantly influenced the morphology and distribution of these phases. As a result, the alloy featured a more refined, oriented, and homogeneous microstructure, which is beneficial for improving mechanical performance and structural integrity in advanced composite applications.

Keywords: Iron silicide, nickel aluminide, tool material, induction melting, directional solidification

1. INTRODUCTION

This study addresses the current challenge of developing in-situ composite alloys based on FeSi–NiAl, with the potential to serve as next-generation tool materials. The primary objective is to design and optimize a new class of tool materials free of tungsten and cobalt, elements identified as critical raw materials [1,2]. A key requirement is that future manufacturing processes be more sustainable, economically viable, and less energy-intensive, while still delivering the required material performance [3,4].

Our research focuses on evaluating the influence of directional solidification using the Bridgman method on the microstructure and microhardness of FeSi–NiAl alloys (mass ratio 1:1) prepared via induction melting.

Previous studies have shown that these alloys can be successfully produced using both vacuum induction melting (VIM) and powder metallurgy (PM) techniques, specifically spark plasma sintering (SPS). The resulting materials exhibit a stable microstructure, high strength, and excellent tribological properties—comparable to

those of Vanadis 6 steel, a high-performance tool steel widely used for machining hardened materials due to its outstanding wear resistance [5].

One of the main drawbacks of these in-situ composites is their inherent brittleness, which correlates with their high microhardness. The aim of this study is to investigate how directional solidification influences the resulting microstructure and mechanical properties, particularly microhardness, in order to enhance toughness and overall performance.

While the effects of directional solidification have been extensively studied in Ni-Al-based alloys (especially the Ni_3Al and NiAl phases) [6], no such research has yet been published for FeSi–NiAl composites or iron silicide (FeSi)-based systems. Therefore, this study offers a novel experimental approach and contributes original insights into the development of advanced tool materials [7].

2. EXPERIMENTAL

The in-situ composite was prepared using high-purity elemental metals: nickel (≥ 99.9 wt%), iron (≥ 99.9 wt%), aluminium (≥ 99.9 wt%), and silicon (≥ 99.9985 wt%). The experimental material was produced in a Linn (GmbH) Supercast-Titan medium-frequency vacuum induction furnace, using a corundum crucible for melting. The alloy was cast centrifugally into a mold made of isostatically pressed graphite, resulting in rods with a diameter of 20 mm and a length of 220 mm. The in-situ composite was prepared with a weight ratio of FeSi to NiAl of 1:1.

Directional solidification by the Bridgman method was conducted in a CLASIC (CZ) crystallization furnace, in employing a corundum crucible under a protective atmosphere of argon 5N. The melting temperature was set to 1450 °C and held at this temperature for 30 minutes. The feed rate of the crystallization zone was maintained at 100 mm/hour. Directionally solidified (DS) composite was subjected to the same observation as the as-cast composite.

The microstructures of the in-situ composites were examined using an Olympus GX51 optical microscope (LOM), equipped with an Olympus DP12 digital camera and Analysis FIVE software. Specimens were etched using nital to reveal the microstructure. Additional microstructural and chemical analyses were carried out using a QUANTA 450 FEG scanning electron microscope (SEM, FEI Company) operated in backscattered electron (BSE) mode. The SEM was equipped with an energy-dispersive X-ray (EDX) analyzer, APOLLO X, for elemental analysis. Vickers microhardness measurements were performed on all prepared composites using a fully automated testing system (FM–ARS900), equipped with the FM–100 microhardness tester and FT–ARS900 software for quantitative analysis.

3. RESULTS AND DISCUSSION

3.1 Microstructure and chemical composition

Figure 1a shows the microstructure of the FeSi–NiAl 1:1 composite captured using an optical microscope (LOM). The composite exhibited a typical dendritic microstructure, characteristic of the alloy in its as-cast state. The eutectic structure is also clearly visible. **Figure 1b** presents SEM images of the composite, highlighting areas where chemical composition was analyzed using EDX. The average of three measurements is summarized in **Table 1**. The chemical analysis revealed the presence of multiple phases within the microstructure. As shown in **Table 1**, Region 1 (dendrites) and Region 2 (part of the eutectic phase) were rich in Ni and Al, with lower contents of Fe and Si. In contrast, Region 3 (interdendritic region) primarily consisted of Fe and Si, with minor contents of Ni and Al.

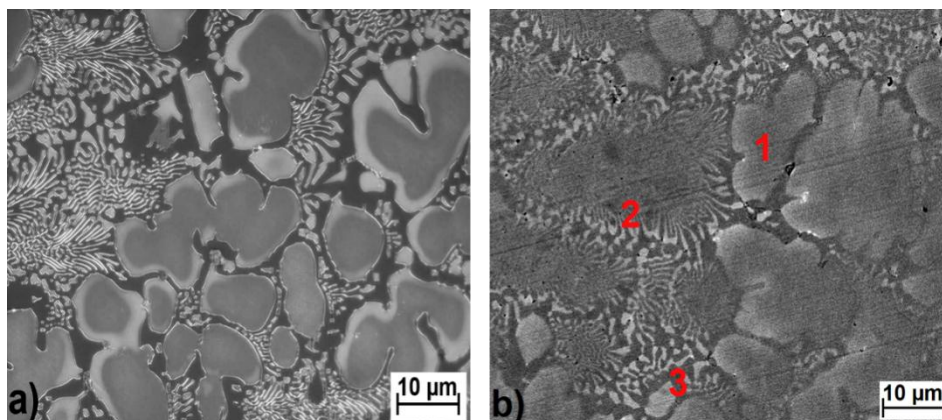


Figure 1 Microstructures of in-situ composite FeSi-NiAl 1:1 in as-cast state: a) LOM, b) SEM

Table 1 Chemical composition (EDX) of selected areas of the composite FeSi-NiAl 1:1

element / area	Al	Si	Fe	Ni
	(at%)			
spot 1 (dendrite)	32.7	19.5	19.6	28.2
spot 2 (eutectic phase)	28.2	21.6	23.5	26.8
spot 3 (interdendritic region)	15.6	36.1	31.3	17.2

Figure 2a shows the microstructure of the FeSi-NiAl 1:1_DS composite in transverse section captured using an optical microscope (LOM). The composite after directional solidification showed a dendritic microstructure, together with a lamellar-type eutectic, both of which are characteristic of the directionally solidified state. **Figure 2b** presents SEM images of the composite, highlighting the areas where the chemical composition was analyzed by EDX. The average of the three measurements is summarized in **Table 2**. The chemical analysis revealed the presence of multiple phases in the microstructure. As shown in **Table 2**, region 1 (dendrites) and region 2 (part of the eutectic phase) were rich in Ni and Al, with lower concentrations of Fe and Si. In contrast, region 3 (interdendritic region) consisted mainly of Fe and Si, with smaller concentrations of Ni and Al. The SEM images also show the modified shape of the dendrites and the presence of a lamellar eutectic, both resulting from the directional solidification process.

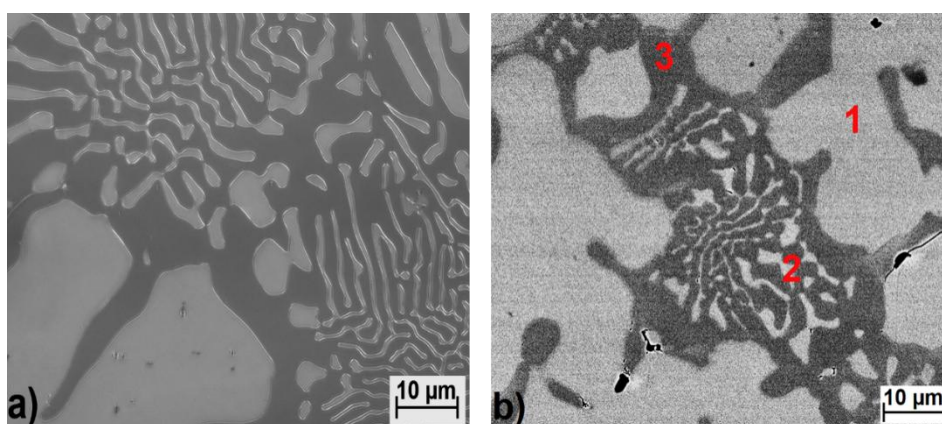


Figure 2 Microstructures of in-situ composite FeSi-NiAl 1:1_DS transverse section: a) LOM, b) SEM

Table 2 Chemical composition (EDX) of selected areas of the composite FeSi–NiAl 1:1_DS

element / area	Al	Si	Fe	Ni
	(at%)			
spot 1 (dendrite)	34.1	19.1	18.2	28.7
spot 2 (eutectic phase)	35.9	16.2	17.6	30.2
spot 3 (interdendritic region)	11.5	42.0	33.5	13.0

Figure 3a shows the microstructure of the FeSi–NiAl 1:1_DS composite in a longitudinal section captured using an optical microscope (LOM). The composite after directional solidification exhibited a dendritic microstructure along with a eutectic of lamellar character, which is typical of the directionally solidified state. The lamellar structure is clearly more pronounced in the longitudinal section. **Figure 3b** presents SEM images of the composite which clearly revealed the formation of the lamellar eutectic structure as a result of directional solidification. This structural refinement contributed to improved homogeneity and increased toughness, potentially reducing the material's brittleness.

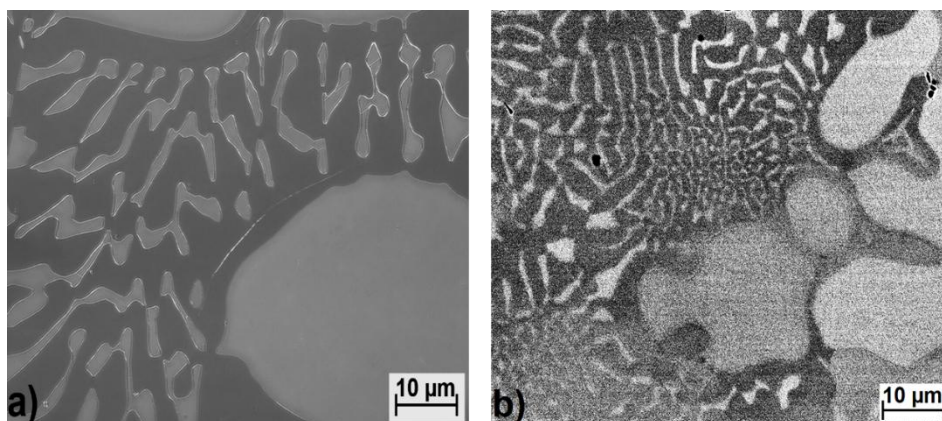


Figure 3 Microstructures of in-situ composite FeSi–NiAl 1:1_DS longitudinal section: a) LOM, b) SEM

3.2 Microhardness

Table 3 presents the results of Vickers microhardness (HV 0.1) measurements for both FeSi–NiAl-based composites with a 1:1 weight ratio, one in the as-cast state and the other after directional solidification. The data clearly illustrate the differences in hardness between the two material states. The conventional as-cast composite exhibited an average hardness of 956 ± 129 HV 0.1, whereas the directionally solidified (DS) composite showed a lower average hardness of 845 ± 12 HV 0.1. Although the DS composite had a slightly reduced hardness, it exhibited significantly lower variability in the measured values. This reduced scatter suggests improved homogeneity and greater stability of the material properties. The microhardness values reported in **Table 3** represent the average of measurements taken from both the transverse and longitudinal sections. As the average values from each section did not differ significantly, a combined average was used for clarity.

The structure of both FeSi–NiAl composites (in the as-cast and directionally crystallized states) is dominated by a dendritic microstructure, with dendrites representing the primary crystallization region of the NiAl phase, while the FeSi phase is concentrated in the interdendritic spaces. In the as-cast state, dendrites grow randomly in all directions, which leads to an irregular distribution of the FeSi phase, a higher incidence of structural defects, and increased local stress concentration. This type of microstructure is often manifested by higher hardness, but at the same time lower homogeneity and a greater dispersion of measured values. On the

contrary, in the directionally crystallized material, the dendrites of the NiAl phase grow in a directed manner in the direction of the temperature gradient, which leads to a more regular and uniform distribution of the FeSi phase. The result is a structure with lower hardness dispersion and higher microstructural stability, because areas with increased internal stress are minimized. Despite a slight decrease in average hardness, the microstructure created in this way is more advantageous in terms of the strength and fracture properties of the composite.

Although the as-cast composite (FeSi–NiAl 1:1) exhibited a higher average microhardness, the large standard deviation (± 129 HV 0.1) indicated considerable structural inhomogeneity and the potential presence of local defects. In contrast, the directionally solidified variant (FeSi–NiAl 1:1_DS) demonstrated a slightly lower average hardness, but with a significantly reduced standard deviation (± 12 HV 0.1), reflecting a much more uniform microstructure. This enhanced consistency is a key advantage in terms of material reliability and performance stability in demanding technical applications. From this perspective, directional solidification improves the reproducibility of mechanical properties and may represent a more suitable processing route in applications where uniform and predictable behaviour is prioritized over maximum hardness.

Table 3 Microhardness values HV 0.1 for all samples of in-situ composites

Composite	HV 0.1
FeSi–NiAl 1:1	956 ± 129
FeSi–NiAl 1:1_DS	845 ± 12

4. CONCLUSION

This work was focused on the study of the influence of direction solidification on the microstructure of in-situ composite and its properties of material based on FeSi–NiAl, prepared by the vacuum induction melting (VIM) in a ratio of 1:1. The study was motivated by the high brittleness of this composite in the as-cast state, which is a current problem in the search for a new type of tool material that could be produced by VIM technology. At the same time, this work was intended as an alternative to the previously mainly investigated procedures based on powder metallurgy. The FeSi–NiAl composite in a weight ratio of 1:1 was investigated both in the as-cast state and after directional solidification performed at a temperature of 1450°C and a crystal growth rate of 100 mm/h.

- Microstructural characterization via LOM and SEM revealed that the as-cast FeSi–NiAl composite consists of coarse primary dendrites surrounded by a randomly oriented eutectic constituent. In contrast, the directionally solidified composite exhibited a refined dendritic morphology and a lamellar eutectic structure with pronounced directional alignment, evident in both transverse and longitudinal sections. This transition from a randomly distributed to a directionally organized eutectic morphology demonstrates the impact of controlled solidification parameters on phase alignment and structural regularity. The observed microstructural refinement and orientation resulting from directional solidification are expected to enhance homogeneity, reduce anisotropy, and improve the mechanical consistency of the material, which are critical attributes for advanced tool applications.
- The results of microhardness measurements show that although directional solidification slightly decreased the microhardness values compared to the as-cast state of the composite, the standard deviations are lower. This means that the dispersion of microhardness values is smaller in the state after directional solidification. It can therefore be stated that directional solidification led to the creation of a very homogeneous and stable material.
- Further investigation will focus on the phase composition, tribological behaviour, and additional processing parameters associated with directional solidification. Specifically, the effects of a reduced

crystal growth rate and an elevated melting temperature will be explored to refine the solidification conditions.

- Based on the current findings, it is evident that the Bridgman directional solidification method has a significant impact on the microstructural development and resulting properties of the FeSi–NiAl in-situ composite.

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