

## IMPROVEMENT OF STRUCTURAL HOMOGENEITY AND WEAR RESISTANCE OF FeSi–NiAl COMPOSITES BY DIRECTIONAL SOLIDIFICATION

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### Abstract

This study deals with the development and characterization of FeSi–NiAl based composite materials, consisting of NiAl matrix and FeSi hardening phase, representing a new generation of tool materials. These composites can compete with conventional materials, such as cemented carbides and high-speed steels, which contain tungsten and cobalt – raw materials classified as critical by the European Union and economically burdened by the current geopolitical situation. The composites were prepared by vacuum induction melting with centrifugal casting in weight ratios of FeSi:NiAl – 1:2 and 2:1. In order to improve structural homogeneity and mechanical properties, the samples were subsequently remelted by the Bridgman directional solidification method (DS) at a temperature of 1450 °C with a holding time of 30 minutes and a crystallization rate of 100 mm/h. The microstructure was analyzed using scanning electron microscopy (SEM) and chemical composition was determined by energy dispersive X-ray spectroscopy (EDX). Furthermore, microhardness was evaluated using the Vickers method along with tribological properties, specifically the friction coefficient and wear rate, which are key for practical tool applications. In the as-cast state, the composites showed a randomly oriented dendritic microstructure. In contrast, DS led to significant straightening of the microstructure and controlled distribution of individual phases. This process resulted in a significant leveling of mechanical and tribological properties in both compositions compared to the as-cast state. This indicates higher structural homogeneity and more stable material behaviour under operating conditions, making these composites suitable for advanced engineering applications.

**Keywords:** Iron silicide, nickel aluminide, sustainability, directional solidification

### 1. INTRODUCTION

In modern engineering, there is a growing need for the development of a new generation of tool materials capable of replacing conventional high-speed steels and cemented carbides [1-2]. These traditional materials rely on tungsten and cobalt, which are classified by the European Union as critical raw materials and whose availability is further constrained by the current geopolitical situation [3]. FeSi–NiAl based composites represent a promising alternative that, owing to the absence of these scarce elements, offer reduced economic burden while maintaining a high potential for demanding engineering applications [4-5].

Previous studies have demonstrated that these alloys can be successfully produced using both vacuum induction melting (VIM) and powder metallurgy (PM) routes, particularly spark plasma sintering (SPS), yielding

materials with a stable microstructure, high strength, and excellent tribological properties comparable to those of Vanadis 6 tool steel [6-7].

One of the principal drawbacks of these composites is their inherent brittleness, which correlates with their high microhardness. Accordingly, the aim of this study is to investigate the influence of DS on the resulting microstructure and mechanical properties to enhance toughness and overall performance. While the effects of DS have been extensively studied in Ni–Al based alloys, particularly those containing the Ni<sub>3</sub>Al and NiAl phases [8–9], its influence on Fe–Si–Ni–Al system has not yet been sufficiently investigated.

## 2. EXPERIMENTAL

The composites were prepared using high purity metals: nickel ( $\geq 99.9$  wt%), iron ( $\geq 99.9$  wt%), aluminium ( $\geq 99.9$  wt%), and silicon ( $\geq 99.9985$  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 composites were prepared with a weight ratio of FeSi to NiAl of 1:2 and 2:1.

Directional solidification by the Bridgman method was conducted in a CLASIC (CZ) crystallization furnace using 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. The directionally solidified (DS) composite was subjected to the same observation as the as-cast composite.

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) analyser, 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 software for quantitative analysis. A corundum friction ball was used to measure the wear rate. The measurement was performed on a ball tribometer TRIBOtester Tt – TRIBOtechnic. The testing conditions were as follows – ball diameter: 6 mm, sliding length: 20 m, sliding speed: 10 mm/s, load: 5 N.

## 3. RESULTS AND DISCUSSION

### 3.1 Microstructure and chemical analysis

**Figure 1** shows the microstructure of the FeSi–NiAl 1:2 composite in both conditions. In the as-cast state (**Figure 1a**), the microstructure exhibits a pronounced dendritic morphology. The primary dendritic regions (spot 1) correspond to an Al and Ni rich phase, which is consistent with the chemical composition determined by EDX analysis (**Table 1**), showing increased contents of Al and Ni. In contrast, the interdendritic regions (spot 2) are enriched in Si and Fe. The microstructure is highly heterogeneous, with sharp interfaces between the individual phases, which is characteristic of solidification under conventional casting conditions. After DS, a significant change in microstructural morphology is observed (**Figure 1b**), where the dendritic structure transforms into coarser, polygonal grains with well-defined boundaries. Despite the pronounced morphological changes, the chemical composition of the individual regions remains essentially unchanged, as confirmed by the EDX results summarized in **Table 1**. DS therefore does not alter the phase composition of the composite but primarily leads to morphological reorganization and increased structural homogeneity.

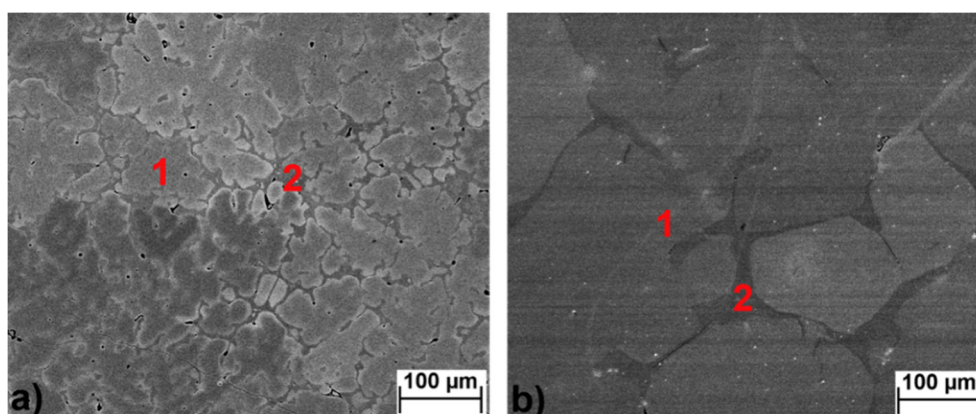


**Figure 1** SEM image of microstructure of composite FeSi–NiAl 1:2: a) as-cast state, b) after DS.

**Table 1** Chemical composition (EDX) of selected areas of the composite FeSi–NiAl 1:2 in both states (at%)

| element / area                 | Composite FeSi – NiAl 1:2 in as-cast state |      |      |      |
|--------------------------------|--------------------------------------------|------|------|------|
|                                | Al                                         | Si   | Fe   | Ni   |
| spot 1 (dendrite)              | 35.5                                       | 17.8 | 15.0 | 31.8 |
| spot 2 (interdendritic region) | 13.3                                       | 40.0 | 30.4 | 16.3 |
|                                | Composite FeSi – NiAl 1:2 after DS         |      |      |      |
|                                | Al                                         | Si   | Fe   | Ni   |
| spot 1 (dendrite)              | 36.4                                       | 17.0 | 15.0 | 31.6 |
| spot 2 (interdendritic region) | 12.3                                       | 41.5 | 31.9 | 14.4 |

**Figure 2** shows the microstructure of the FeSi–NiAl 2:1 composite in both states. In the as-cast state (**Figure 2a**), the microstructure exhibits a complex, heterogeneous morphology consisting of several distinct microstructural regions. The primary dendritic regions (spot 1) are enriched in Fe and Si, corresponding to a Fe–Si rich phase, as confirmed by the EDX analysis (**Table 2**). These dendrites are surrounded by eutectic regions (spot 2) with a mixed chemical composition, indicating the coexistence of FeSi and NiAl phases. The interdendritic regions (spot 3) are enriched in Al and Ni and correspond to a NiAl rich phase (**Table 2**). The as-cast microstructure was characterized by a high degree of heterogeneity and relatively diffuse phase boundaries, which are typical of solidification under conventional casting conditions. After DS (**Figure 2b**), a pronounced modification of the microstructural morphology is observed. The microstructure becomes more ordered and directionally aligned, with clearly distinguishable microstructural regions. In particular, the interfaces between the Fe–Si rich regions and the NiAl containing eutectic areas become significantly sharper and better defined compared to the as-cast state.

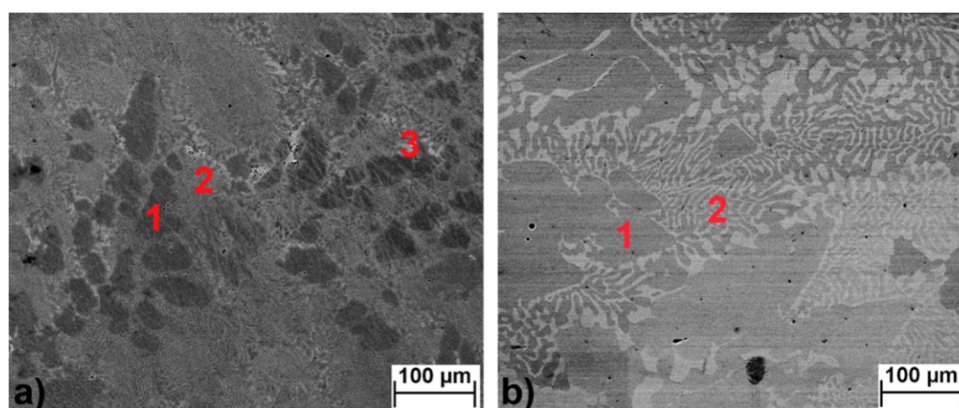


**Figure 2** SEM image of microstructure of composite FeSi–NiAl 2:1: a) as-cast state, b) after DS

**Table 2** Chemical composition (EDX) of selected areas of the composite FeSi–NiAl 2:1 in both states (at%)

| element / area                 | Composite FeSi – NiAl 2:1 in as-cast state |      |      |      |
|--------------------------------|--------------------------------------------|------|------|------|
|                                | Al                                         | Si   | Fe   | Ni   |
| spot 1 (dendrite)              | 10.3                                       | 44.0 | 36.3 | 9.6  |
| spot 2 (eutectic phase)        | 18.2                                       | 35.8 | 30.0 | 16.0 |
| spot 3 (interdendritic region) | 31.7                                       | 19.9 | 21.9 | 26.4 |
|                                | Composite FeSi – NiAl 2:1 in after DS      |      |      |      |
|                                | Al                                         | Si   | Fe   | Ni   |
| spot 1 (dendrite)              | 11.5                                       | 42.4 | 35.1 | 11.0 |
| spot 2 (eutectic phase)        | 34.7                                       | 17.8 | 20.1 | 27.4 |

### 3.2 Mechanical and tribological properties

**Figure 3** shows SEM images of the wear tracks formed during tribological testing of the FeSi–NiAl 1:2 composite against a ceramic ball in both states. In the as-cast state (**Figure 3a**), the worn surface exhibits pronounced surface damage characterized by material transfer, local delamination, and irregular grooves. These features indicate that the dominant wear mechanism is a combination of adhesive and abrasive wear. The presence of adhesive junctions and their subsequent rupture lead to material pull out and contributes to the relatively lower microhardness of the as-cast composite ( $654 \pm 25$  HV0.1), as summarized in **Table 3**.

After DS (**Figure 3b**), the wear track morphology changes significantly. The worn surface is dominated by fine, parallel grooves with minimal evidence of material transfer or delamination, indicating that abrasive wear becomes the prevailing wear mechanism, while adhesive wear is strongly suppressed. This transition in wear behaviour correlates with the increased microhardness of the composite after DS ( $710 \pm 13$  HV0.1) and with the formation of a more homogeneous and structurally ordered microstructure with sharper phase boundaries. Despite the slightly higher friction coefficient and wear rate observed after DS (**Table 3**), the wear mechanism becomes more stable and predictable, governed primarily by abrasion rather than by severe adhesive interactions.

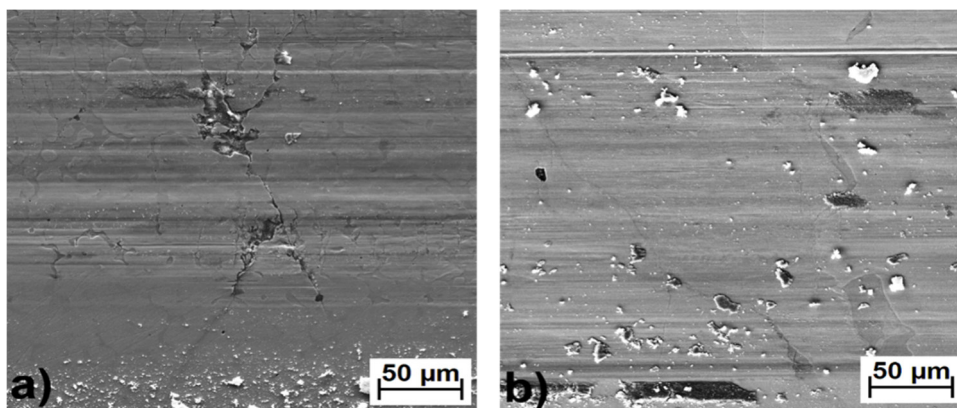


**Figure 3** SEM image of tribological testing with a ceramic ball, composite FeSi–NiAl 1:2: a) as-cast state, b) after DS

**Table 3** Mechanical properties of the FeSi–NiAl 1:2 composite in both states

| Composite                        | Friction coefficient<br>(-) | Microhardness (HV 0.1) | Wear rate<br>(mm <sup>3</sup> /N/m) |
|----------------------------------|-----------------------------|------------------------|-------------------------------------|
| FeSi – NiAl 1:2 in as-cast state | 0.386                       | $654 \pm 25$           | $14.7 \cdot 10^{-6}$                |
| FeSi – NiAl 1:2 in after DS      | 0.435                       | $710 \pm 13$           | $18.9 \cdot 10^{-6}$                |

**Figure 4** shows SEM images of the wear tracks formed during tribological testing of the FeSi–NiAl 2:1 composite against a ceramic ball in both states. In the as-cast state (**Figure 4a**), the worn surface exhibits severe surface damage characterized by extensive plastic deformation, material delamination, and irregular wear scars. These features indicate that the dominant wear mechanism is a combination of adhesive and abrasive wear, with a significant contribution of adhesive interactions. This behaviour is consistent with the relatively high friction coefficient (0.529) and the high microhardness of the as-cast composite ( $1153 \pm 114$  HV0.1), as summarized in **Table 4**.

After DS (**Figure 4b**), a pronounced change in the wear track morphology is observed. The worn surface becomes more uniform and is dominated by parallel grooves aligned with the sliding direction, which are characteristic of abrasive wear. Evidence of adhesive wear, such as material transfer and delamination, is significantly reduced compared to the as-cast state. This transition is accompanied by a decrease in the friction coefficient to 0.438 and a reduction in the wear rate (**Table 4**). Despite a slight decrease in microhardness after DS ( $1086 \pm 35$  HV0.1), the wear resistance improves, indicating that the dominant factor governing tribological performance is the microstructural reorganization induced by DS, particularly the formation of sharper and more stable phase boundaries that suppress adhesive wear.

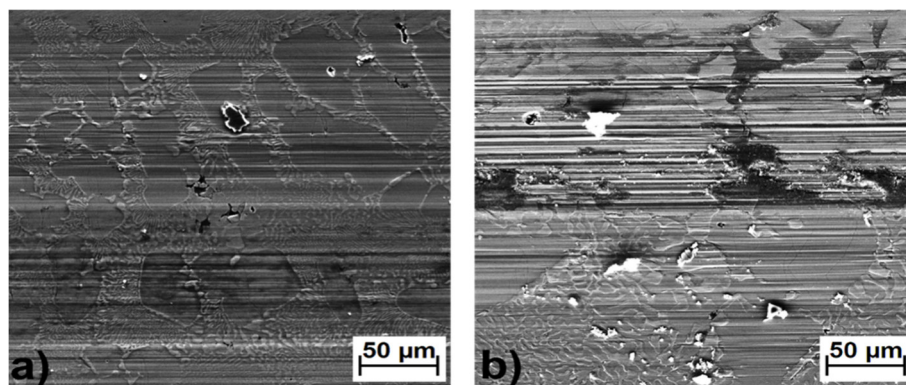


**Figure 4** SEM image of tribological testing with a ceramic ball, composite FeSi–NiAl 2:1: a) as-cast state, b) after DS

**Table 4** Mechanical properties of the FeSi–NiAl 2:1 composite in both states

| Composite                        | Friction coefficient<br>(-) | Microhardness<br>(HV 0.1) | Wear rate<br>(mm <sup>3</sup> /N/m) |
|----------------------------------|-----------------------------|---------------------------|-------------------------------------|
| FeSi – NiAl 2:1 in as-cast state | 0.529                       | $1153 \pm 114$            | $17.8 \cdot 10^{-6}$                |
| FeSi – NiAl 2:1 in after DS      | 0.438                       | $1086 \pm 35$             | $14.3 \cdot 10^{-6}$                |

#### 4. CONCLUSIONS

This work focused on investigating the influence of DS on the microstructure and properties of FeSi–NiAl composites prepared by vacuum induction melting (VIM). The FeSi–NiAl composites were examined in both the as-cast condition and after DS carried out at 1450 °C with a crystal growth rate of 100 mm/h.

- Directional solidification successfully transformed the randomly oriented dendritic microstructure of the as-cast composites into a highly ordered architecture. The process resulted in significant morphological reorganization, leading to coarser, polygonal grains and a more uniform distribution of individual phases.
- EDX analysis confirmed that while DS induces profound morphological alignment, the fundamental chemical composition of the individual regions remains essentially unchanged. However, the process

led to a significant sharpening and better definition of the phase interfaces, which is crucial for stable material behaviour.

- The effectiveness of directional solidification proved to be strongly dependent on the specific weight ratio of the phases. In the FeSi–NiAl 2:1 composite, DS led to a substantial improvement in tribological performance, characterized by a reduction in the friction coefficient from 0.529 to 0.438 and a transition to a stable abrasive wear mechanism.
- In the 2:1 system, the formation of mechanically stable interfaces effectively restricted local plastic deformation and suppressed adhesive junctions. Conversely, in the 1:2 composite, while DS increased microhardness to 710 HV0.1, it also led to increased surface brittleness and a higher wear rate, highlighting the critical role of phase topology in determining material toughness.

Based on the results presented above, it can be stated that FeSi–NiAl composites represent a viable and sustainable alternative to conventional high-speed steels and cemented carbides. By eliminating reliance on tungsten and cobalt raw materials classified as critical by the EU, this research addresses current geopolitical and economic burdens while offering a high potential material for advanced engineering applications. In summary, directional solidification is a powerful tool for optimizing the microstructural architecture of iron silicide and nickel aluminide composites. The FeSi–NiAl 2:1 composition demonstrates a superior balance of structural homogeneity and wear resistance, marking it as a promising candidate for further industrial tool development.

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