# PREPARATION OF COMPOSITES BASED ON FeSi–NiAl BY VACUUM INDUCTION MELTING

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**Abstract**

The work deals with the study of progressive materials based on a matrix of NiAl and a reinforcing FeSi phase. The fabrication is realized through vacuum induction melting and centrifugal casting. In terms of the ratio of the base matrix and the strengthening phase, two experimental alloys were prepared, which were subsequently heat treated at 1173 K/3h. The microstructural characteristics were evaluated using the LOM and SEM methods. The chemical composition of the prepared alloys was determined using EDX. The phase composition was investigated by powder X-ray diffraction analysis. The microstructure of the as-cast FeSi–NiAl (1:1) material consisted of Ni–Al-based dendrites and eutectic. The interdendritic spaces were mainly formed by Fe and Si. As a result of the heat treatment, the dendrites softened and therefore their volume fraction decreased. The microstructure of the FeSi – NiAl (2:1) alloy consisted of dendrites and eutectic. Dendrites contained Fe and Si. Ni and Al were also found in the interdendritic space. All the prepared materials had the character of in-situ composites, which means that the process of their formation occurred during the preparation process. In all samples, the matrix (binder phase) was based on Ni–Al, while the reinforcement was based on Fe–Si. Heat treatment of the samples led to the equalization of the chemical composition and the almost complete disappearance of the $AlNi_2Si$ and $Al_2FeSi$ phases. From the point of view of phase composition, all samples after heat treatment were mostly two-phase and contained phases based on FeSi and NiAl.

**Keywords:** Composite, silicide, aluminide, melting, microstructure

## 1. INTRODUCTION

Currently, there is a wide range of cutting materials – tool steels (high-speed tool steels), cermets (coated), cemented carbides (hard wear-resistant coatings), cutting ceramics (coated) and super-hard diamond-based cutting materials (synthetic diamond, cubic boron nitride), which are most widely used for machining processes in the automotive industry or in the design and manufacture of aircraft or rail vehicles due to their ability to withstand extremely demanding conditions [1]. However, mentioned materials contain tungsten and cobalt, which are included in the list of EU Critical raw materials due to their economic importance and supply risk [2]. Their inclusion highlights the need for sustainable and innovative approaches to reduce reliance on these materials in cutting tools. The ongoing research and innovation in materials science aim to develop new compositions and alternative materials for cutting tools that not only match the performance of traditional materials but also promote sustainability and resource efficiency. One possibility is the preparation of in-situ composites based on FeSi and NiAl intermetallic compounds. Emphasis must be placed here on the appropriate ratio of the individual phases.

The key method of preparation of this system is selected techniques of powder metallurgy [3-5]. Iron and silicon create a total of 6 intermetallic phases – $Fe_2Si$, $Fe_3Si$, $Fe_5Si_3$, FeSi, $FeSi_2$, $Fe_3Si_7$. The mentioned phases have historically been investigated for their exceptional electrical and magnetic properties, but also for their specific strength and resistance [6-7]. The FeSi phase is also known for its excellent magnetic properties [8]. In this work, FeSi fulfill the function of the hard phase, the intermetallic phase of NiAl is intended as the binder material. The work follows on from experiments with the preparation of identical materials using powder metallurgy. In the presented work, the alloys with different FeSi : NiAl ratio were prepared by vacuum induction melting (VIM) in an oxide crucible in combination with centrifugal casting followed by a heat treatment. Microstructural characteristics, phase and chemical composition of prepared alloys in as-cast state and after heat treatment were investigated in detail.

## 2. EXPERIMENTAL

In-situ composites were prepared using of the pure elements - nickel (≥ 99.9 wt%), iron (≥ 99.9 wt%), aluminium (≥ 99.9 wt%) and silicon (≥ 99.9985 wt%). The preparation of the experimental material was carried out in a Linn (GmbH) Supercast-Titan medium-frequency vacuum induction furnace, an $Al_2O_3$ crucible was used for the melting. The casting was performed centrifugally into a mold made of isostatically pressed graphite. Using this procedure, rods with a diameter of 20 mm and a length of 220 mm were prepared. In terms of the ratio of both components, in-situ composites were prepared with ratios (FeSi : NiAl) of 1:1 and 2:1.

Both samples were further subjected to the heat treatment at 1173 K/3 h in an atmosphere of pure Ar (99.999 vol%) in order to homogenize their chemical composition and modify their microstructure using a sintering furnace Xerion Advanced Heating, Ofentechnik (GmbH).

The microstructures of the in-situ composites were studied using an Olympus GX51 optical microscope (LOM) equipped with an Olympus DP12 digital camera and Analysis FIVE software. Etching was done with Nital. The microstructure and chemical composition of the alloy were analyzed by the scanning electron microscope (SEM) QUANTA 450 FEG (FEI Company) used in the backscattered electron (BSE) mode completed by an energy-dispersive X-ray (EDX) analyzer APOLLO X.

The phase composition of the prepared samples was investigated by X-ray powder diffraction (XRPD) method using the X'Pert PRO Powder Diffractometer PANalytical. CoK radiation with a wavelength of $\lambda$ = 0.178901 nm was used in the measurement.

## 3. RESULTS AND DISCUSSION

### 3.1. Phase and chemical analysis

**Figure 1a** shows image of the microstructure of the FeSi-NiAl (1:1) material taken by the optical microscope (LOM). Samples showed a typically dendritic microstructure, which is characteristic for the alloy in the as-cast state. The eutectic was also very well visible here. SEM images of the FeSi-NiAl (1:1) material are shown in **Figure 1b**. The image shows areas where the chemical composition was determined using EDX. The average of three measurements is given in **Table 1.** Chemical analysis revealed that the microstructure of the sample is composed of several phases. From **Table 1**, it can be seen that region 1 (dendrites) and region 2 (part of the eutectic) contained the majority of Ni and Al elements and the minority of Fe and Si elements. Region 3 (a part of the eutectic) was mostly formed by Fe and Si elements and a minor part of Ni and Al elements. The average chemical composition corresponded to the nominal chemical composition of the FeSi – NiAl 1:1 sample.

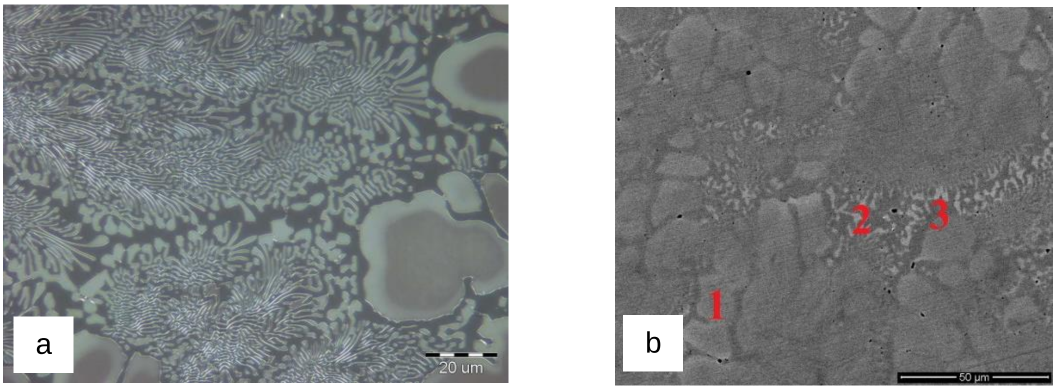


**Figure 1** Microstructures of in-situ composite FeSi-NiAl (1:1) in as-cast state: a) LOM, b) SEM

**Table 1** Chemical composition in wt% (EDX) of selected areas of the sample FeSi-NiAl (1:1)

| element / area | **Al** | **Si** | **Fe** | **Ni** |
|---|---|---|---|---|
| average composition | 16.4 | 17.9 | 32.9 | 32.9 |
| area 1 | 22.6 | 11.6 | 24.2 | 41.7 |
| area 2 | 18.4 | 13.1 | 30.2 | 38.3 |
| area 3 | 8.0 | 26.3 | 44.4 | 21.3 |

**Figure 2a** shows the microstructure of the FeSi-NiAl (1:1) sample after heat treatment taken with the use of the optical microscope (LOM). It was found that the applied heat treatment did not lead to the disappearance of the eutectic, which was still present in the microstructure. The microstructure was further formed by dendrites, which had a modified character compared to the as-cast state. A finer dendritic structure and eutectic can be observed. The SEM image of the FeSi-NiAl (1:1) sample after heat treatment is shown in **Figure 2b**. The areas analysed by EDX are marked here. The average of three measurements is given in **Table 2.**

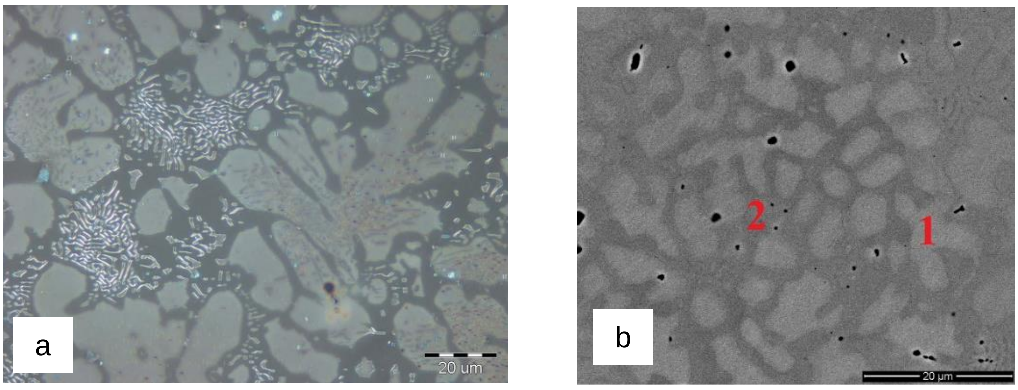


**Figure 2** Microstructures of in-situ composite FeSi-NiAl (1:1) after heat treatment, a) LOM, b) SEM

The sample was found to exhibit a two-phase structure. Area 1 (dendrites) contained mostly elements Ni and Al and minor elements Fe and Si. Area 2 (interdendritic spaces) mainly contained elements Fe and Si and minor elements Ni and Al. The heat treatment of the sample led to a refinement of the dendrites, a reduction in their volume fraction and an equalization of the chemical composition.

**Table 2** Chemical composition (EDX) of selected areas of the sample FeSi-NiAl (1:1) after heat treatment (wt%)

| element / area | **Al** | **Si** | **Fe** | **Ni** |
|---|---|---|---|---|
| area 1 | 23.1 | 11.7 | 25.3 | 40.0 |
| area 2 | 12.0 | 24.4 | 35.4 | 28.2 |

**Figure 3a** shows the microstructure of the FeSi-NiAl (2:1) sample, taken by the optical microscope. It can be seen that the microstructure consisted of dendrites and a high eutectic content. **Figure 3b** shows the SEM image of the microstructure of the FeSi-NiAl (2:1) sample. Areas, where the chemical composition was determined using EDX, are marked on this image. The average of three measurements is given in **Table 3.** The microstructure was composed of several phases. Area 1 (dendrites) was mainly consisted of Fe and Si and a minor part of Ni and Al. Area 2 (interdendritic spaces) was consisted mostly of Ni and Al and to a minor extent of Fe and Si. Region 3 corresponded to the eutectic. The average chemical composition agreed very well with the nominal chemical composition of the FeSi – NiAl 2:1 sample.

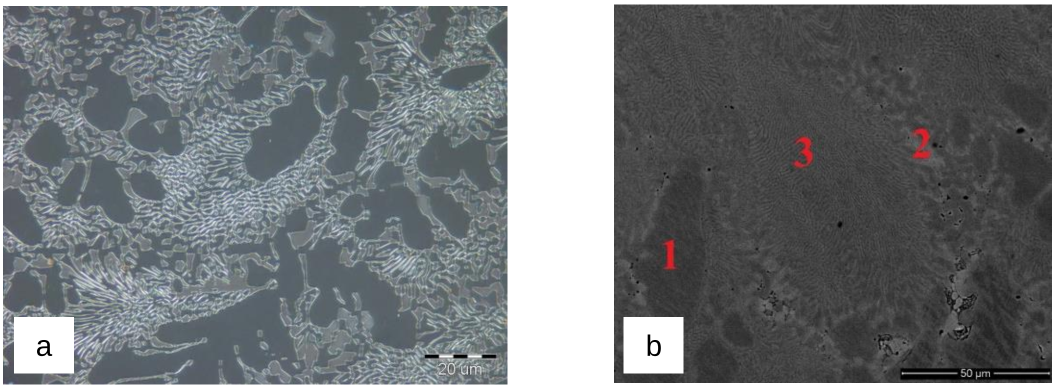


**Figure 3** Microstructures of in-situ composite FeSi-NiAl (2:1), a) LOM, b) SEM

**Table 3** Chemical composition (EDX) of selected areas of the sample FeSi-NiAl (2:1) (in wt%)

| element / area | **Al** | **Si** | **Fe** | **Ni** |
|---|---|---|---|---|
| average composition | 11.9 | 24.5 | 40.9 | 22.7 |
| area 1 | 6.8 | 30.1 | 49.4 | 13.7 |
| area 2 | 20.4 | 13.1 | 29.2 | 36.9 |
| area 3 | 14.2 | 22.2 | 37.8 | 25.8 |

**Figure 4a** shows the microstructure of the FeSi-NiAl (2:1) sample (LOM). As a result of heat treatment, there was a slight reduction in the volume fraction of dendrites, and the eutectic was clearly visible. **Figure 4b** shows SEM images of the FeSi-NiAl (2:1) material after heat treatment. The analysed areas are also marked here. **Table 4** shows the average values from three measurements. It was found that the microstructure of the sample was only two-phase. Area 1 (interdendritic spaces) was formed mostly of Ni and Al elements and to a minor extent of Fe and Si elements. Area 2 (dendrites) mainly contained Fe and Si elements. Region 3 corresponded to the eutectic.

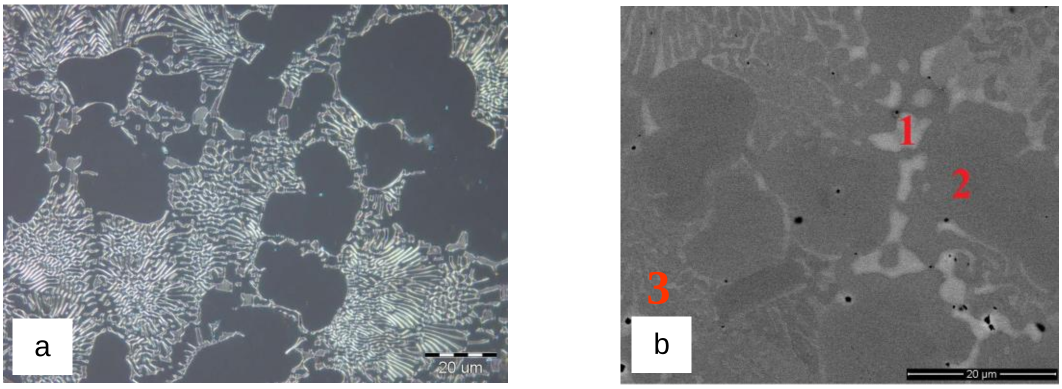


**Figure 4** Microstructures of in-situ composite FeSi-NiAl (2:1) after heat treatment, a) LOM, b) SEM

**Table 4** Chemical composition (EDX) of selected areas of the sample FeSi-NiAl (2:1) (in wt%)

| element / area | **Al** | **Si** | **Fe** | **Ni** |
|---|---|---|---|---|
| area 1 | 22.3 | 10.8 | 29.9 | 37.0 |
| area 2 | 6.4 | 30.3 | 50.3 | 13.0 |
| area 3 | 14.6 | 21.9 | 37.0 | 26.4 |

### 3.2. XRD analyses

**Figure 5a** shows the record from the XRD analysis for samples FeSi-NiAl 1:1 and FeSi-NiAl 1:1 after heat treatment (HT). In the FeSi-NiAl 1:1 sample in the as-cast state, phases with a crystal structure of FeSi, NiAl, $AlNi_2Si$ and $Al_2FeSi$ were found, while after heat treatment the structure was mainly composed of phases based on FeSi and NiAl, which is in good agreement with the SEM/EDX results analysis. It can therefore be concluded that heat treatment led to a substantial reduction in the content of $AlNi_2Si$ and $Al_2FeSi$ phases. From the diffraction records of both samples it is clear that phases with a crystal structure of FeSi and NiAl prevailed in the samples, which is evident from the peaks of the diffraction lines. **Figure 5b** shows the record from the XRD analysis for samples FeSi-NiAl 2:1 and FeSi-NiAl 2:1 after heat treatment (HT). Phases with the crystal structure of FeSi, NiAl and $AlNi_2Si$ were found in the structure of the FeSi-NiAl 2:1 sample in the as-cast state. The diffraction record shows that the sample of this alloy after heat treatment already contained only FeSi and NiAl phases, which is in good agreement with the results of SEM/EDX analysis. The heat treatment therefore led to a substantial reduction in the content of the $AlNi_2Si$ phase. It is clear that in both samples the phases with a crystal structure of FeSi and NiAl prevailed, which is evident from the peaks of the diffraction lines.

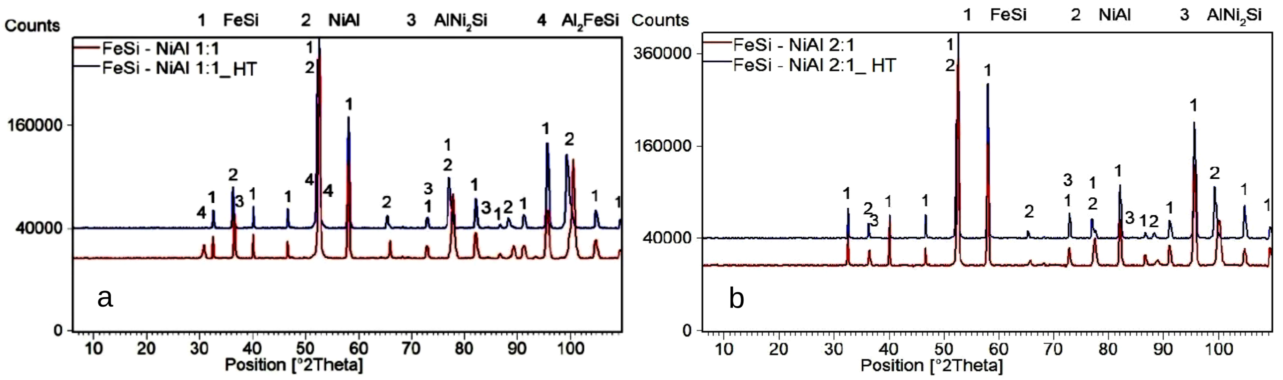


**Figure 5** Diffraction record for samples **(a)** FeSi-NiAl 1:1 (as-cast) and **(b)** FeSi-NiAl 1:1 TZ (after heat treatment)

## 4. CONCLUSION

This work was focused on the preparation of in-situ composite materials based on FeSi-NiAl using VIM, as an alternative to the concurrently investigated preparation procedures using powder metallurgy methods. Two experimental materials with different FeSi : NiAl (1:1 and 2:1) weight ratio were chosen.

- Melting was carried out for both materials on the same amount of charge using the same technology and production procedure. The chosen technology was found to be suitable for the preparation of the investigated materials. In the next part, heat treatment was carried out, here it was stated that it would be appropriate to optimize the regime.
- Metallographic observations were made on prepared metallographic samples. Furthermore, the microstructural characteristics of the prepared materials were evaluated (LOM, SEM) and the chemical composition of the occurring phases was also determined using EDX.
- To deepen knowledge about structural properties, XRD analysis was performed for both types of materials, including materials after heat treatment. It was found that the performed heat treatment caused a modification of the microstructure.
- Subsequent experimental work is focused on determining mechanical characteristics, especially hardness and microhardness, frictional properties will also be given significant attention.

## ACKNOWLEDGEMENTS

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