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EELS and EDS Spectroscopy of FeSi/NiAl Composite

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This article describes details of lamellar eutectic part of FeSi/NiAl (2:1) in-situ composite prepared by induction melting and followed by directional solidification by the Bridgman method. The material was formed by FeSi dendrites, and interdendritic spaces were filled by FeSi-NiAl eutectic, which was proven by EBSD observations. The phase boundaries are clearly visible, but all elements are detected in all phases. The energy dispersive spectroscopy in transmission electron microscope has proven that most significant contamination is by iron in the NiAl phase, where it replaces mainly Ni. The content of iron it the FeSi phase is close to the stoichiometry, and it can be assumed that the nickel is more present is the silicon positions of the FeSi phase. These results were confirmed also by electron energy loss spectroscopy.

Keywords: Iron silicide, Nickel aluminide, Microscopy, Spectroscopy

1 Introduction

Intermetallics are promising materials formed form two or more metals, but their properties significantly differ from usual alloys [1]. The reason is in covalent bounds contained in the material [1]. Intermetallic alloys may be prepared by reactive sintering [2,3], mechanical alloying [1] and with suitable equipment also by casting [4]. Intermetallics are in a centre of attention due to their excellent high-temperature mechanical properties and resistance to high-temperature oxidation. Moreover, they might be possibly used as a replacement for critical raw materials [5,6].

This article describes FeSi/NiAl in-situ composite material. The FeSi: NiAl ration in this material was chosen to be 2:1. The material composed of FeSi dendrites and interdendritic spaces filled with lamellar FeSi/NiAl eutectic [4]. The material was prepared by induction melting and subsequently underwent directional solidification using the Bridgman method. This article focusses on the description of the eutectic part of the systém by spectroscopies included in transmission electron microscope – energy dispersive spectroscopy (EDS) and electron energy loss spectroscopy (EELS). The aim is to study in detail the composition of individual lamellae of FeSi and NiAl.

2 Experimental

The in-situ composite was prepared by melting of 44.4 wt. % of iron (≥ 99.9 wt.%), 22.2 wt. % of silicon (≥ 99.9985 wt.%), 22.9 wt. % of nickel (≥ 99.9 wt.%) and 10.5 wt. % of aluminium (≥ 99.9 wt.%) in a Linn (GmbH) Supercast-Titan medium-frequency vacuum induction furnace. The composition corresconds to weight ratio of FeSi to NiAl of 2:1. 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. The sample was mechanicaly cut followed by mechanical grinding and polishing until final thickness of approximately 100 μm. The 3 mm disc by Leica TXP sample preparation machine. The discs were mechanicaly polished to 70 μm and ion polished by Leica RES 102 ion polisher. The polished sample was observed by transmission electron microscope (TEM, 200 kV) Jeol 2200FS equipped by Oxford Instrument EDS detector and TVIPs camera. The EELS spectroscopy was performed using in-column energy filted by Jeol and were measured by TVIPs software EMenu. The EELS data were obtained with energy dispersion of 400 μm/eV. The EELS data are presented in a native form only with addition of constant for better clarity of presentation. The ion polished sample was also observed by TESCAN MIRA 3 equipped by EDS and EBSD (electron back scattered diffraction) by Oxford Instruments.

3 Results and Discussion

The material composed of FeSi dendrites and FeSi/NiAl lamellar eutectic interdendritic areas. Fig 1

shows the EDS elemental maps and EBSD phase map. The Ni and Al rich regions overlap with NiAl phase, Si ovelaps with FeSi phase but in case of Fe, the situation seems to be more complicated. The presence of Fe is detected also in the NiAl regions.

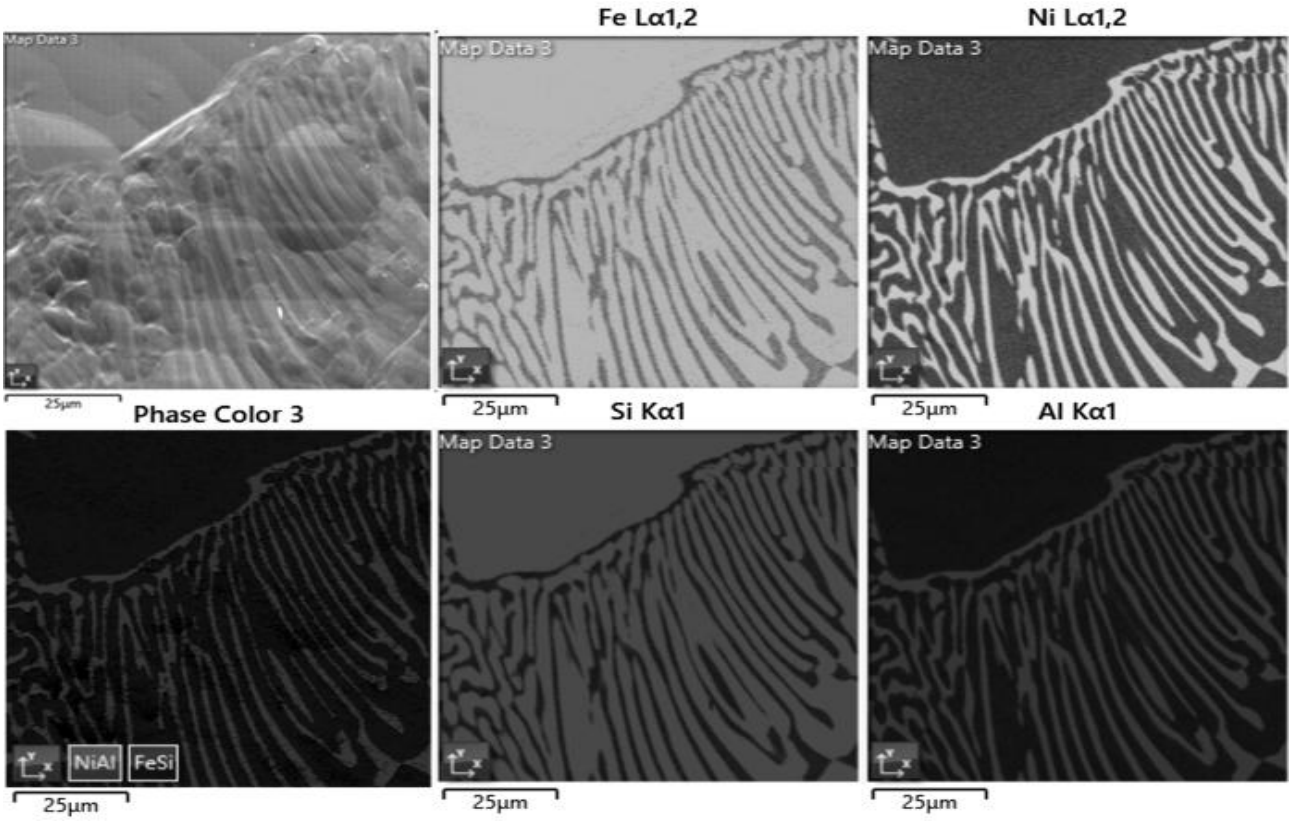


Fig. 1 *Micrograph of the sample with EDS maps and EBSD phase map*

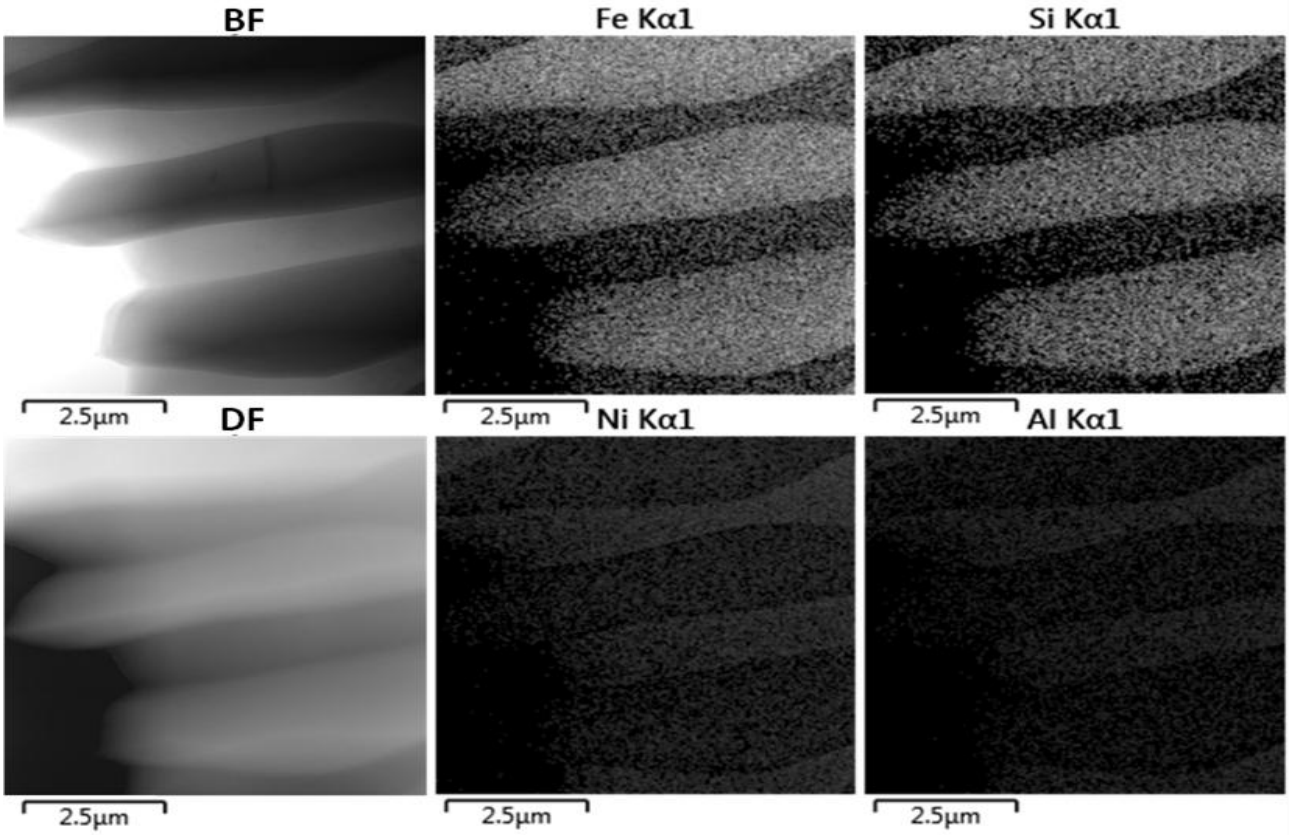


Fig. 2 *Bright and dark field STEM micrographs with EDS elemental maps*

The SEM/EDS resolution is not sufficient for detailed observations. To describe the distribution of individual elements in detail, TEM observation was performed and the result are presented in Fig. 2.

In Fig. 2, lamellaes of FeSi and NiAl can be seen. The contrast in bright field (BF) and dark field (DF) is caused not only by various atomic weight of elements forming the phases but also by different thickness of FeSi and NiAl parts formed by different polishing rates during sample preparation. The TEM/EDS maps indicate contamination of both phases by all elements.

In the bright field image in Fig. 3 are visible dislocations located in the NiAl phase, and they are labelled by red arrows. The elemental maps confirm previous observations. The EDS line scan shows preferential presence of Fe in NiAl phase, but the interpretation is complicated as the results are given only in the intensity units. Moreover, when we focuss on the plot, we can see decreasing of signal in total that corresponds to desceasing thickness of the sample.

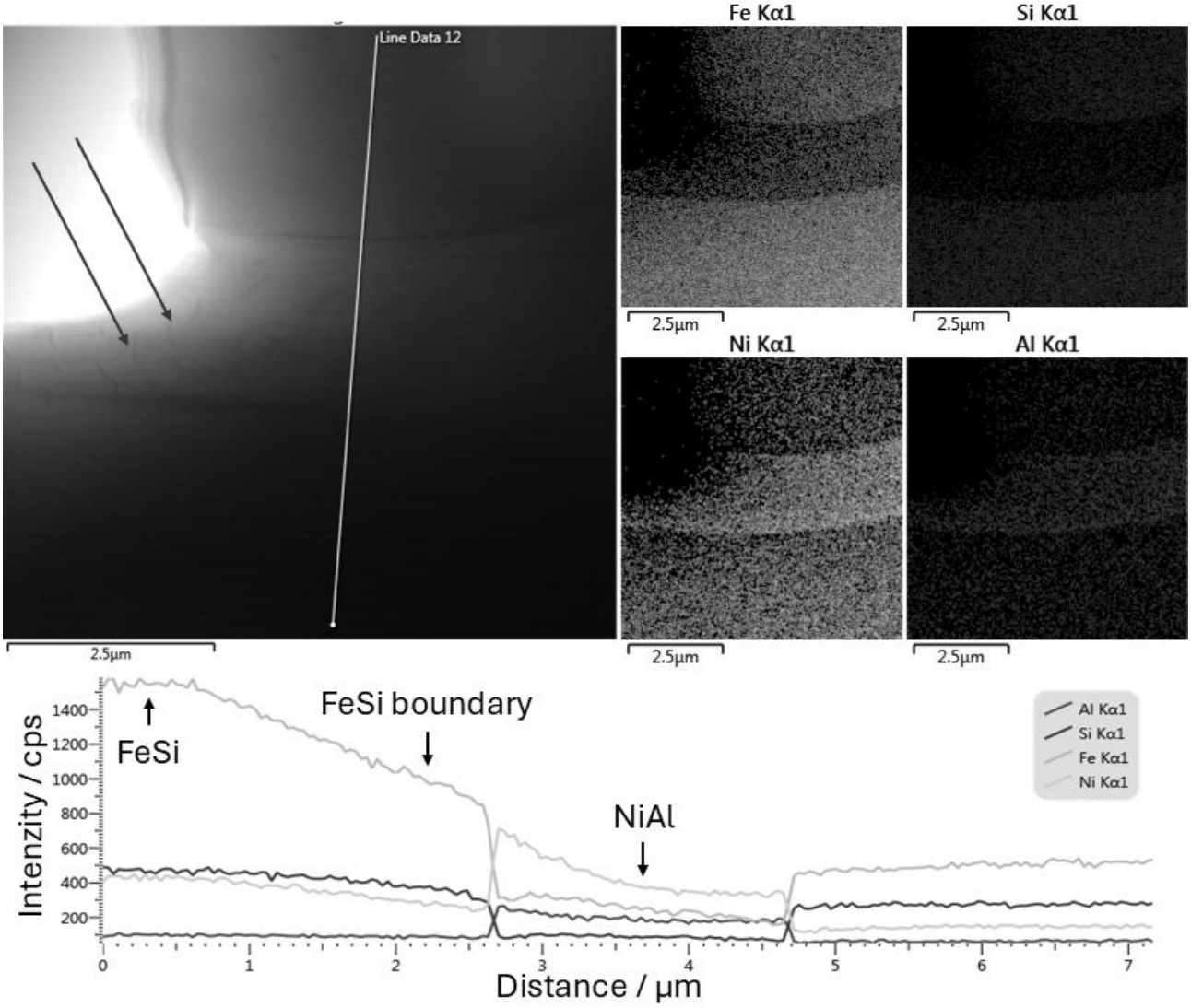


Fig. 3 *Detailed STEM image with EDS elemental maps and EDS line scan, red arrows label dislocations*

Tab. 1 *EDS element composition given in wt. % (average of 3 measurements)*

Position	Fe	Si	Ni	Al
FeSi	62.2 ± 0.2	16.9 ± 0.3	17.7 ± 0.3	3.2 ± 0.2
FeSi boundary	54.9 ± 1.2	23.1 ± 1.0	16.6 ± 0.3	5.4 ± 0.3
NiAl	30.2 ± 1.0	8.8 ± 1.1	43.2 ± 1.9	16.7 ± 1.7

The EDS point analysis was performed in the places labelled in Fig. 3 as FeSi, FeSi boundary and NiAl and the results are given in Tab. 1.

The content of Fe in the center of the FeSi lamellae is approaching the theoretical content of Fe in FeSi (66.35 wt. %). This may indicate that Ni and Al occupy

the Si positions in the lattice. Interestingly, the composition is not the same in the whole cross-section of the lamellae. At the FeSi side of the boundary, lower content of Fe and higher contend of Si was detected with almost the same contents of Ni and Al. Prolonged holding at temperatures above the melting point may lead to changes in the chemical composition of the alloy due to possible elemental losses or redistribution. Such compositional variations may locally affect the phase formation conditions and could contribute to the formation of $FeSi_2$ [6].

To have a closer look at the Ni and Fe in both phases, the EELS measurement was performed, and the results are presented in Fig. 4. Iron was detected both in FeSi and NiAl phase, but the shapes of the spectra differ. The FeSi curve exhibits typical two peak for L2 and L3 edges [8,9]. The Fe L2 edge in NiAl phase is suppressed. There are several explanations: i) more ion character of the bound, ii) less covalent bound, iii) distorture of (crystal) arrangement [8,9]. The observed changes will be caused by combination of previously mentioned reasons (that are anyway not independent). In case of Ni edge this phenomenon is even more pronounced. The Ni L3 edge in FeSi is negligible and the L2 edge is not visible at all. As the Fe L2 edge in NiAl is lower but detectable, we can assume that Fe is mainly replacing Ni (with similar properties). On the other hand, both Ni edges are barely detectable in FeSi. The Ni atoms are probably again replacing Fe in the FeSi phase and the lack of Si atoms is compensated by vacancies in Si position.

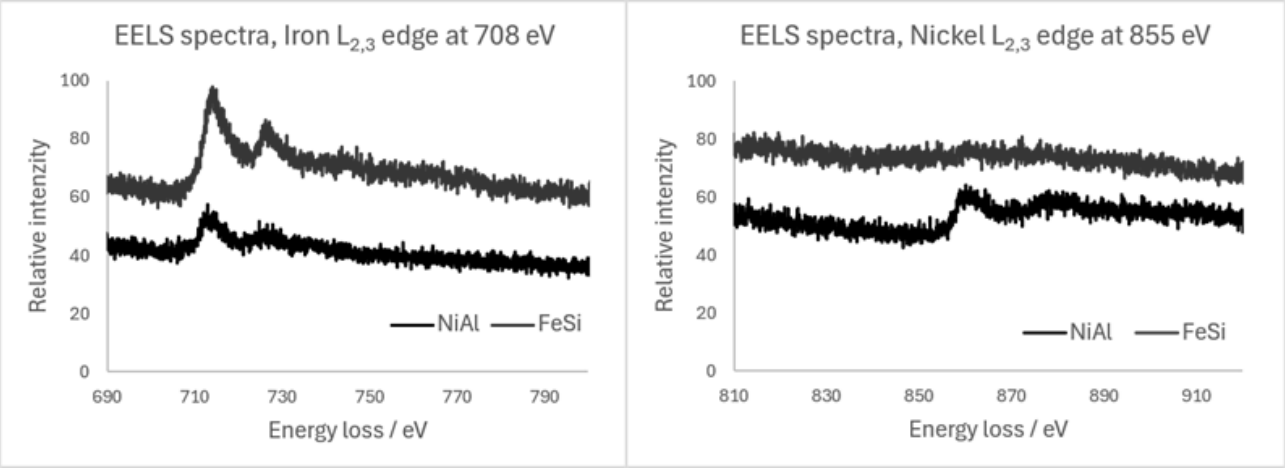


Fig. 4 *EELS spectra on iron and nickel edges*

4 Conclusion

The interdendritic part of FeSi/NiAl (2:1) intermetallic composite was in detail described. It is composed of FeSi and NiAl lamellaes. The NiAl phase is significantly contaminated by Fe presence, and the Fe is most probably replacing Ni in the lattice. On the hand, from combination of EELS and EDS spectroscopies, the Ni atoms are replacing Fe in the FeSi phase and the lack of Si atoms is compensated by vacancies in Si position.

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References

[1] NOVÁK, P., SALVETR, P., PECENOVÁ, Z. (2015). Intermetallics – Synthesis, Production, Properties. In: *Manufacturing Technology*, Vol. 15, No. 6, pp. 1024 – 1028. ISSN 1213–2489. DOI: 10.21062/ujep/x.2015/a/1213-2489/MT/15/6/1024.

[2] MICHALCOVÁ, A., VOJTĚCH, D., KUBATÍK, T. F., NOVÁK, P., DVOŘÁK, P. (2014). Structural Description of Powder Metallurgy Prepared Materials. In: *Manufacturing Technology*, Vol. 14, No. 3, pp. 359 – 362. ISSN 1213–2489. DOI: 10.21062/ujep/x.2014/a/1213-2489/MT/14/3/359.

[3] ŠKOLÁKOVÁ, A., SALVETR, P., NOVÁK, P. (2019). Effect of Heating Rate on the Phases Formation in Ti-20 wt. % Al Powder Mixture. In: *Manufacturing Technology*, Vol. 19, No. 1, pp. 139 – 143. ISSN 1213–2489. DOI: 10.21062/ujep/258.2019/a/1213-2489/MT/19/1/139.

[4] KALOČ, V., SKOTNICOVÁ, K., SZURMAN, I., MALCHARCZIKOVÁ, J., NOVÁK, P., ČEGAN, T. (2025). Influence of Directional Solidification on the

Microstructure of In-Situ FeSi–NiAl Composites. In: *Proceedings 34th International Conference on Metallurgy and Materials*, pp. 458 – 463. ISSN 2694-9296. ISBN 978-80-88365-27-3. DOI: 10.37904/metal.2025.5151.

[5] NOVAK, P., MESTEK, S., MICHALCOVÁ, A., TSEPELEVA, A., SKOTNICOVÁ, K., SZURMAN, I. (2024). How to Determine Possible Interactions of Silicide and Aluminide in Preparation of Composites. In: *Manufacturing Technology*, Vol. 24, No. 1, pp. 104 – 109. ISSN 1213–2489. DOI: 10.21062/mft.2024.008.

[6] NOVÁK, P., MESTEK, S., NEČAS, D., SKOTNICOVÁ, K., DROZDOVÁ, Ľ., SZURMAN, I., SMETANA, B. (2025). FeSi-NiAl composite as a future tool material. In: *Journal of Alloys and Compounds*, Vol. 1010, Article 177227. ISSN 0925–8388. DOI: 10.1016/j.jallcom.2024.177227.

[7] KHAJAVI, L. T., BARATI, M. (2012). Thermodynamics of Phosphorus Removal from Silicon in Solvent Refining of Silicon. In: *High Temperature Materials and Processes*, Vol. 31, No. 4–5, pp. 627 – 631. ISSN 0334-6455. DOI: 10.1515/htmp-2012-0100.

[8] KASATIKOV, S., FANTIN, A., MANZONI, A. M., SAKHONENKOV, S., MAKAROVA, A., SMIRNOV, D., FILATOVA, E. O., SCHUMACHER, G. (2021). Chemical interaction and electronic structure in a compositionally complex alloy: A case study by means of X-ray absorption and X-ray photoelectron spectroscopy. In: *Journal of Alloys and Compounds*, Vol. 857, pp. 157597. ISSN 0925–8388. DOI: 10.1016/j.jallcom.2020.157597.

[9] MIEDEMA, P. S., DE GROOT, F. M. F. (2013). The Iron L Edges: Fe 2p X-ray Absorption and Electron Energy Loss Spectroscopy. In: *Journal of Electron Spectroscopy and Related Phenomena*. DOI: https://doi.org/10.1016/j.elspec.2013.03.005.