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USING SEM AND EDX FOR A SIMPLE DIFFERENTIATION OF α- AND β-ALFESI-PHASES IN WROUGHT ALUMINUM BILLETS

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Abstract

Aluminum 6xxx extrusions have considerable potential to make cars lighter. Thus there is a growing demand for high quality aluminum billets. Additionally there is a requirement to improve the aluminum properties. An important issue to fulfill these increasing requirements is the optimization of the microstructure. While utilizing 6xxx-alloys the formation and optimization of the AlFeSi-phase is important. The determination of α- and β-AlFeSi-phases is a challenge, because β-AlFeSi-phases affect the mechanical properties negatively. A determination in wrought alloys via microscopy is often complicated and leads to questionable results due to the low amount of the phases. TRIMET has investigated one possibility of determining α- and β-AlFeSi-phases with the help of a scanning electron microscope and energy dispersive X-ray. The paper describes the first tests, the phase simulations and the casting experiments. Finally it presents the tests of such phase determinations in the production of 6xxx-alloys like the new TRIMAL 52.

Introduction

As with cast aluminum alloys, intermetallic compounds can form from the melt and from the supersaturated solid solution of the solidified aluminum of wrought alloys. This paper deals with the development and application of a new and simple method for determining the intermetallic AlFeSi-phases and differentiating between α- and β-AlFeSi-phases in wrought aluminum alloys.
Because of deterioration of the mechanical properties the plate like structured β-AlFeSi-phase should be avoided or minimized in extrusion billets as well as in most of the other aluminum products. Instead the curved crystals ("Chinese script") of the α-AlFeSi-phase is preferred, Figure 1 and Figure 2.

A quick determination of the different AlFeSi-phases by considering the structure shape via microscopy as in Figure 1 and Figure 2 is often impossible due to the low amount of AlFeSi-phases and the small dimension of these phases in typical 6xxx alloys. Consultations of universities and institutes yield agreement in principle that the most accurate possibility to differentiate between α- and β-AlFeSi-phases is an EBSD-measuring (EBSD: Electron Backscatter Diffraction). The results are very reliable, but a scanning electron microscope (SEM) with an EBSD is expensive and not always available. Additionally, this method is relatively complex.

Therefore the target of the project described here was to develop a simple and fast method to differentiate between α- and β-AlFeSi-phases with an EDX-measurement of element concentrations (EDX: energy dispersive X-ray) via the relation of silicon and iron content in the AlFeSi-phases.

At first alloy compositions with only α- and only β-AlFeSi-phases respectively were selected via phase simulations and literature. After a microscopy check of the phases the EDX-measurements were carried out.

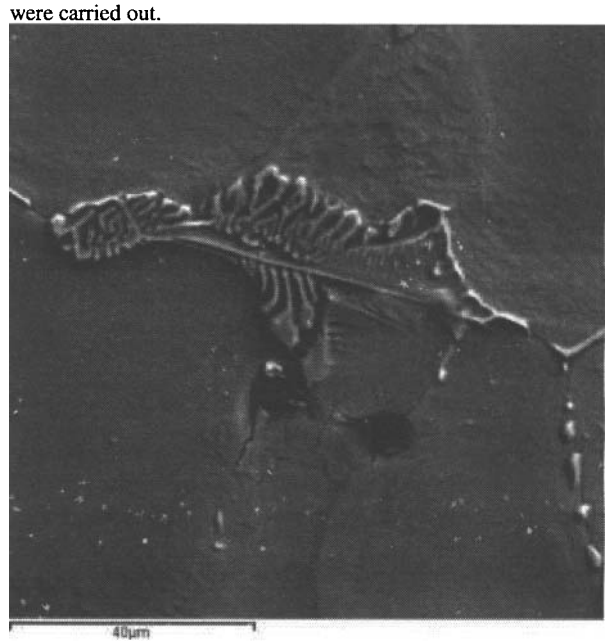


Figure 1. Curved structured α_c-AlFeSi-phase

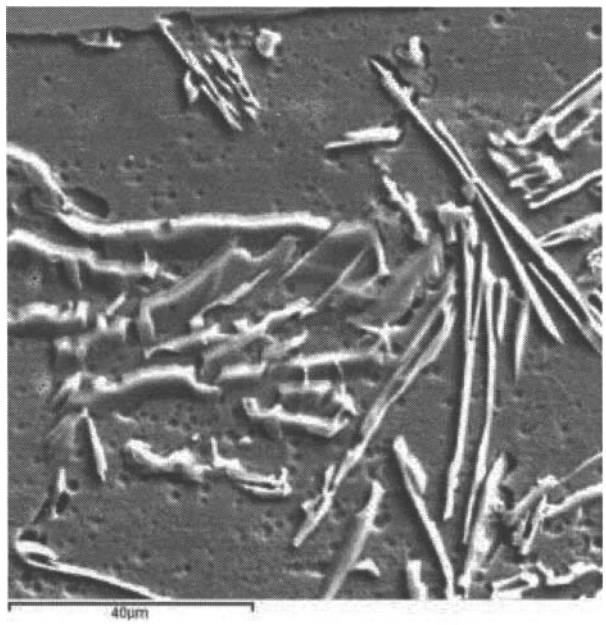


Figure 2. Plate-like β-AlFeSi-phases

α- and β-AlFeSi-Phases

The typical difference between the α- and β-AlFeSi-phases is the structure of the phases as visible in Figure 1 and Figure 2. This is the most convenient method for differentiating between the phases. One of the most important disadvantages of the β-AlFeSi-phase is the sharp and plate-like structure of the β-AlFeSi-phase (Figure 2).

The EBSD-measurement uses the different crystal structures of the phases. In the following table the variance of the prevalent intermetallic phases in the 6xxx series is shown. These different phases have to be differentiated [1].

The stoichiometry of the different phases and especially the relation between silicon and iron in the different AlFeSi-phases are described for wrought alloys in various papers [2, 3]. The different Si/Fe-relations for α_c- and β-AlFeSi-phases À.Griger found out are visible in the diagram in Figure 3. Because the hexagonal α_h-AlFeSi-phase crystallizes only in high purity alloys the investigations focused on the α_c- and β-AlFeSi-phases [2].

Table 1: The variance of the prevalent intermetallic phases in 6xxx series [1]

Phase	Structure	Stoichiometry
$\alpha_c(\alpha)$-AlFeSi	cubic	$Al_{12}Fe_3Si$, $Al_{12-15}Fe_3Si1_{1-2}$
	cubic	$Al_{12}(FeMn)_3Si$, $Al_{15}(FeMn)_3Si_2$
	cubic	$Al_{12}Mn_3Si$, $Al_{15}Mn_3Si_2$, Al_9Mn_2Si
$\alpha_h(\alpha')$-AlFeSi	hexagonal	Al_8Fe_2Si
β-AlFeSi	monoclinic	$Al_{4.5}FeSi$

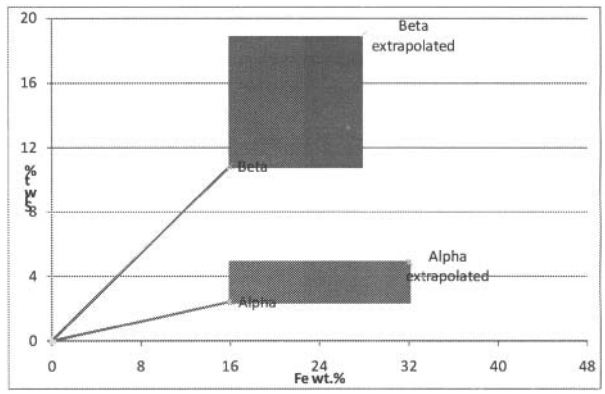


Figure 3: The typical Si/Fe-relation of α_c- and β-AlFeSi-phases [2].

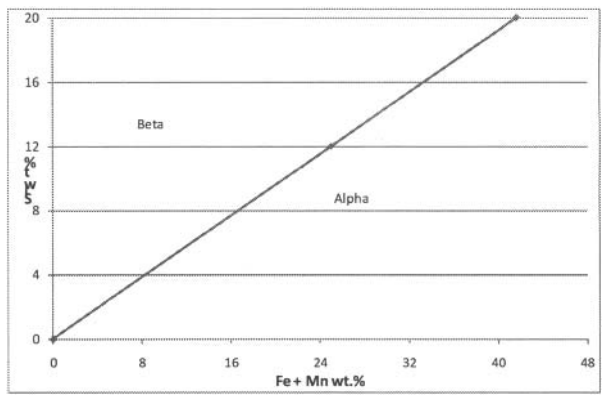


Figure 4: The simplified diagram with the targeted separator between α- and β-AlFeSi-phases

The target of this project was to show that a simplified diagram as in Figure 4 can be used with the EDX-method for the determination of α- and β-AlFeSi-phases. This diagram shows the designated separator between the α- and β-AlFeSi-phases which is expected to be found via EDX-measurements of α- and β-AlFeSi-phases in wrought aluminum alloys. For an accurate analysis the manganese content in the phases is added on the iron

content due to the manganese content of the α_c-AlFeSi-phases, which could be an additional indication to differentiate between the α- and β-AlFeSi-phase.

Investigations

All simulations in this project were carried out at TRIMET with Pandat 8.1 (Version 2009) with the database 8 (Version 2009) from CompuTherm LLC. The main aspects for the alloy composition simulation and development are:

- only α- or β-AlFeSi-phases are present in the alloy to get reference alloys for microscopy, EDX- and EBSD-measuring
- the phases are large enough for a microscopic determination of the different phases.

The first alloy ideas for the starting simulations were taken from different papers [1, 2] and former projects at TRIMET. These alloys are reported or proved to form only α- or only β-AlFeSi-phases. The simulations yielded a couple of alloy compositions with promising simulation results for each phase type (α- and β-AlFeSi-phase respectively). But due to problems with homogenization or other production and casting parameter not all of these alloy composition fulfilled the targets in reality. Below only the best alloy compositions for the α- and β-AlFeSi-phase respectively are described. All simulation diagrams show the last simulation results for the real cast alloy composition. All important alloying elements (11 elements) were considered.

All casting specimens were cast in steel moulds for cylindrical specimens with the following dimensions:

- Diameter: 40mm
- Height: 35mm

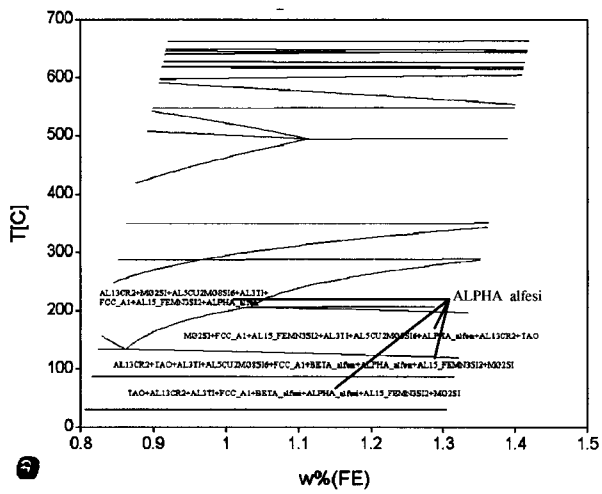
The small dimensions make it easy to control the temperatures during the casting and cooling process.
The target of the casting experiments was to get the reference specimens, which were simulated in the first step. These specimens allow the EDX-measurement method to be proved for the differentiation of α- and β-AlFeSi-phases.

All specimens were analyzed via microscopy and SEM. The phases were analyzed manually via microscopy and with EDX.
As additional specimens, two 6xxx-alloys from TRIMET production were taken. In one alloy predominantly α-AlFeSi-phases were expected, but a minor fraction of β-AlFeSi-phases should be present in some samples. In the other a majority of β-AlFeSi-phases are assumed.

α-AlFeSi-Phase

The alloy presented in Figure 5 is based on previous TRIMET projects. The addition of manganese should favor the crystallization of the α-AlFeSi-phase. After some variations the simulation result concerning the alloy listed below shows a narrow area with only α-AlFeSi. Between approx. 300°C and 120°C only α-AlFeSi is displayed. Based on the theoretical cooling conditions in Pandat, a very fast temperature reduction to 200°C-250°C should give exclusively α-AlFeSi-phases in the specimen.

The specimens for the α-AlFeSi-reference were produced with the parameters shown in Table 2. At 200°C the simulation predicted only α-AlFeSi-phases.
At least the second version (V4) should yield the phase constitution, which is given by the simulation. It was chosen considering the theoretical cooling conditions in Pandat. The predicted forming of the AlFeSi-phases can only be reached with a very slow cooling in the solid state. Therefore a very slow temperature reduction to 200°C was chosen to give exclusively α-AlFeSi-phases in the specimens. Additionally a long holding time at 200°C gives time for forming the predicted α-AlFeSi-phases. After the holding time the phases are frozen by water cooling.



Element	Si	Fe	Mn	Mg	Cr	Zn	Cu
wt.%	0,8299	0,9264	0,1222	0,8403	0,2736	0,0996	0,3446

Figure 5: Pandat simulation and composition of the alloy with the target of only α-AlFeSi-phases in the specimen

Table 2: Casting parameters for the α-AlFeSi-reference specimens

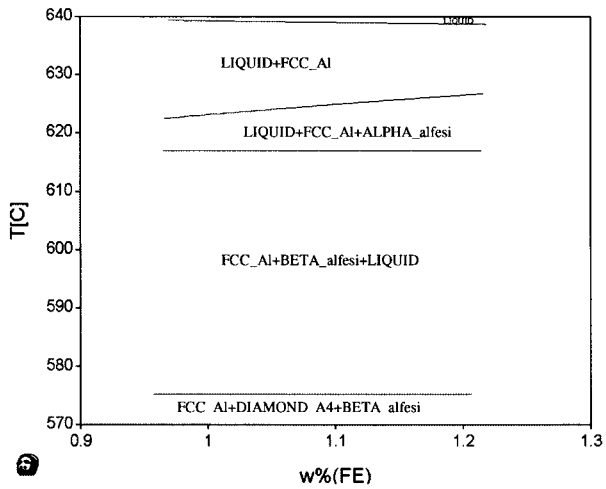
Parameter	Value V3	Value V4
Melt temperature	730°C	730°C
Mould temperature	RT	730°C
Cooling	free air cooling	cooling down in 12h: from 730 to 200°C, Holding time: 12h, 200°C, Water cooling

β-AlFeSi-phase:

The synthetic alloy with only Si and Fe in 99.95% aluminum described in Figure 6 is based on Á.Griger [2]. This alloy is not so complex both concerning alloy composition and simulation result. With the high Si/Fe ratio α-AlFeSi-phases should not be present. The simulation result shows that between 615°C and room temperature, there will be no α-AlFeSi, detail to 570°C in Figure 6.

Summarizing the simulation results we can presume that an exact alloy compositions, adequate casting and cooling parameters should result in reference specimens with only α- and β-AlFeSi-phases respectively.

The casting parameters from Table 3 were used while casting the specimens with the alloy given in Figure 6.
Both versions allow a very fast cooling below 610°C. Below this temperature the simulation gives only β-AlFeSi-phases. Therefore α-AlFeSi-phases should be avoided.



Element	Si	Fe
wt.%	3,0914	0,9802

Figure 6: Pandat simulation and alloy composition of the alloy with the target of only β-AlFeSi-phases in the specimen.

Table 3: Casting parameters for the β-AlFeSi-reference specimens

Parameter	Value V1	Value V2
Melt temperature	730°C	730°C
Mould temperature	RT	590°C
Cooling	immediate water cooling	free air

Results

A brief look at the results found in this project, during the inspection and analysis of the specimens, showed a very good outcome. The first target was to produce reference specimens with only α- and β-AlFeSi-phases respectively. This point was checked via simulation and microscopic analysis. In Figure 7 and Figure 8 it is shown that in the reference specimens only the desired phases are present.

Next important step are the EDX measurements of the reference specimens. In these measurements the Si, Fe and Mn-content was determined. The Si values were plotted against the Fe values and especially against the (Fe+Mn) values, Figure 9. The following facts can be observed:

- the α-AlFeSi points group very well and very closely to the Si/Fe-ratio given by Á.Griger [2]
- the correlation between the β-AlFeSi measurements and the Si/Fe-ratio given by Á.Griger is reasonably visible.
- the spread of the β-AlFeSi measurements is significantly higher
- between the α- and the β-AlFeSi measurements a free area can be found

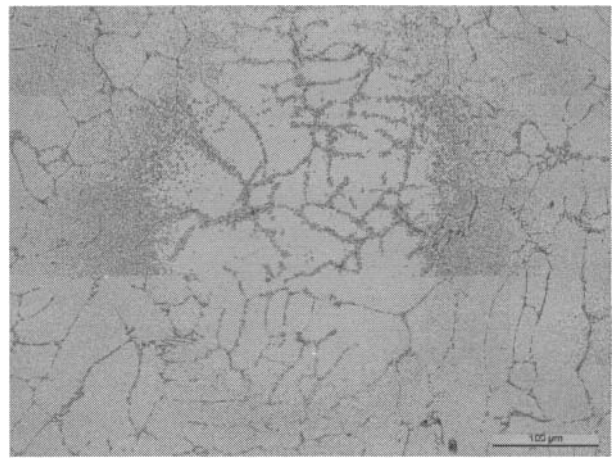


Figure 7: Plate like phases in the β-AlFeSi reference specimen.

The spread of the β-AlFeSi measurements can be explained with the relatively thin phases and the alloy composition. EDX-measurements of element concentrations are imprecise when the target phase is thin. An additional explanation is the free silicon, which could be found in the β-AlFeSi reference especially near β-AlFeSi-phases. Silicon can be measured as a background contamination.

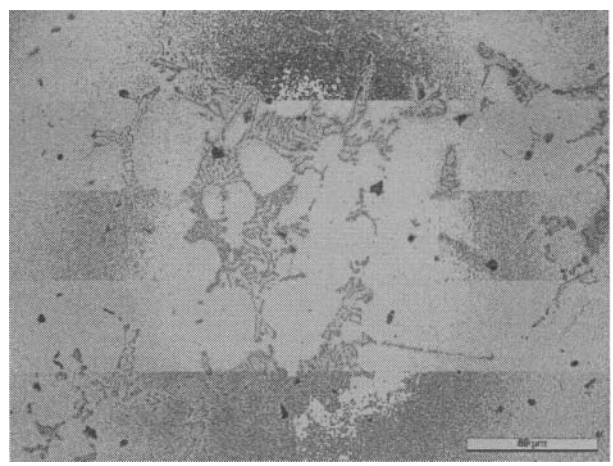


Figure 8: Typical "Chinese script" like phases in the α-AlFeSi reference specimen.

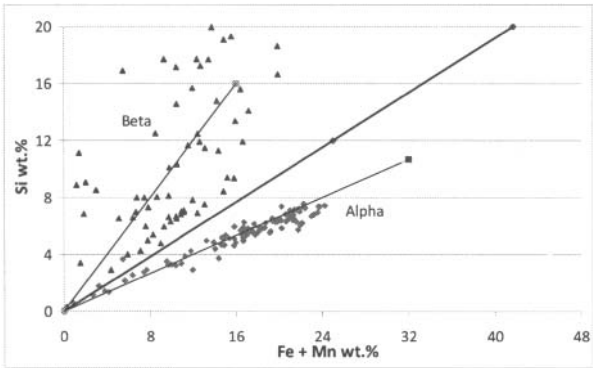


Figure 9: Si/(Fe+Mn) ratio from both reference specimens. Additionally plotted: the Si/Fe ratio for the α- and β-AlFeSi-phase as described by Á.Griger [2], and the targeted separator between α- and β-AlFeSi-phases.

However, of utmost importance is the free and well defined separating area between the α- and β-AlFeSi measurements. Therefore a separator can be defined for the decision, if AlFeSi-phases are the α- or the β-type.

The final step was to verify the new method with the production samples. EDX-measurements were carried out. These measurements are displayed in Figure 10. In alloy A and B predominantly α-AlFeSi-phases were expected. The measurements correlate for most of the AlFeSi-phases. These measurements are on the α-side of the separator. But one can see that a lower content of β-AlFeSi-phases is present in this sample. In alloy C all AlFeSi-phases are from the β-type. For all alloys the measurements concentrates around a straight line. The measurements of alloy C are distributed in higher silicon values, analogue to the reference specimen.

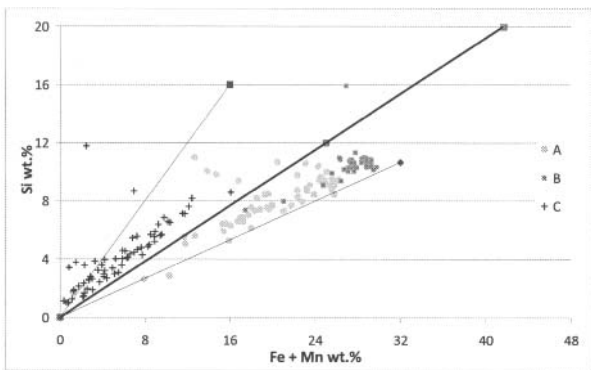


Figure 10: Si/(Fe+Mn) ratio from the 2 production samples. Additionally plotted: the targeted separator between α- and β-AlFeSi-phases.

In summary the method described in this paper is as follows:

- EDX-measurements of the specimen (3-10 measuring points each with 3-10 EDX measurements) resulting in a matrix of points like visible in Table 4
- Plot the Si/(Fe+Mn) ratios of the matrix into a diagram including the separator line
- Determine whether the measured phases are α- or β-AlFeSi-phases

The number of measuring points and measured phases can be low (3 measuring areas each with 3 measured phases) if there is only α- and β-AlFeSi-phases respectively. If the specimen contains α- and β-AlFeSi-phases and a determination of the fraction of phases is desired more measurements are needed. The determination normally takes less than one hour.

Table 4: Detail of a matrix of EDX-measurements (alloy: TRIMAL 52)

TRIMAL 52 114-0153			
Si [wt%]	Fe [wt%]	Mn [wt%]	Fe+Mn [wt%]
6,69	20,27	1,19	21,46
6,7	16,83	2,36	19,19
8,55	21,93	3,52	25,45
4,23	9,75	1,31	11,06
8,07	22,82	2,31	25,13
6,85	17,94	2,17	20,11
3,66	7,6	1,17	8,77
6,09	14,74	2,2	16,94
7,57	20,95	2,53	23,48
4,89	14,91	1,36	16,27
0,67	0,44	0	0,44
6,88	18,93	2,47	21,4
5,67	15,8	2,16	17,96
2,02	3,61	0,9	4,51

This new determination method for α- and β-AlFeSi-phases was used recently for the development of the new TRIMAL 52 alloy [4]. This alloy for high performance space frame constructions was developed to fulfill the demands of the AUDI TL116-C28. Due to the demand for highest mechanical properties β-AlFeSi-phases had to be avoided in the TRIMAL 52, Figure 12. The amount of the β-AlFeSi-phases is one part of the alloy properties, which helps to attain the best mechanical properties.

The main mechanical data are:

- Rp0,2: 281 – 330 MPa
- Rm: > 305 MPa
- A5: ≥ 10%

The crash performance of the alloy is also excellent, Figure 11.

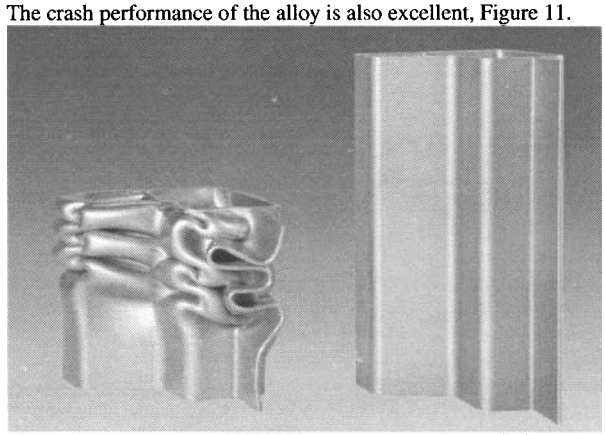


Figure 11: Excellent crush behavior of extrusions with the new high strength aluminum alloy TRIMAL®-52 [4]

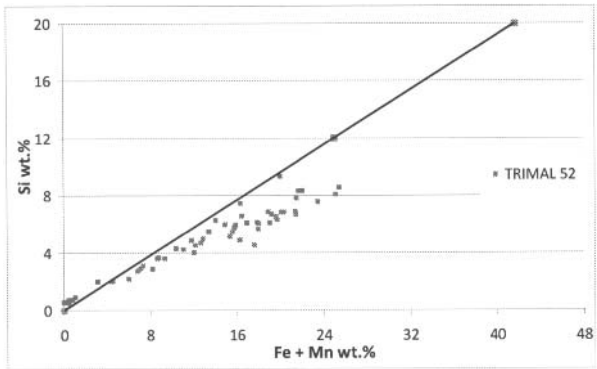


Figure 12: Si/(Fe+Mn) ratio from the new TRIMAL 52 alloy. Additionally plotted: the targeted separator between α- and β-AlFeSi-phases.

Discussion and Conclusions

The EDX-measurement of silicon and iron/manganese concentration is a sure and inexpensive method for a determination of α- and β-AlFeSi-phases in wrought alloys. The method is verified via the simulation results and the microscopic determination of the phases in the reference specimens. Additionally EBSD-measurements of the reference specimens and the production samples are planned for an additional validation.
Limitations of the EDX-measurement principle have to be considered. Because of the relatively inexact element measurement and the penetration of the measurement in the specimens, a statistically relevant number of measurements must be carried out. Measurements with a very low amount of silicon and iron have to be eliminated due to measuring errors. With a higher number of measurements, the straight line of the element relation becomes visible and a determination of the phase character is possible.
Considering these points the EDX-method is an excellent tool for differentiating between α- and β-AlFeSi-phases in wrought alloys.

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