



Refining of Al-Si-Melts by intermetallic iron precipitation – a phase equilibria investigation

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Abstract

The Al-Si-system is the basis of many technological aluminium alloys. Iron-contamination during the processing and metallurgy of recycling materials limits the industrial application of these materials. The presence of numerous ternary phases with iron can be applied for developing a refining technology for Al-Si-melts based on a combination of segregation and mechanical separation of intermetallic phases. The basis for this process is a full understanding of the existent phase equilibria.

According to L.F. Mondolfo [1], there are two ternary phases at room temperature that can be in equilibrium with the aluminium solid solution: $\text{Al}_8\text{Fe}_2\text{Si}$ and Al_5FeSi . O.S. Zarechnik [2] reported a phase distribution of the Al-Si-Fe system at 600°C . According to this work, there are three phases in equilibrium with aluminium: $\text{Al}_8\text{Fe}_2\text{Si}$, Al_5FeSi and Al_4FeSi_2 . In the field of industrial alloys the phase reactions are mainly peritectic. Peritectic reactions are slow because of the diffusion in the solid state. Therefore, it can be expected that the phase distribution in commercial alloys with a high cooling rate differs from the equilibrium distribution. The aim of this work was to determine the metastable phase distribution in alloys with a cooling rate similar to some recycling processes [3].

The phase distribution in alloys crystallised at $10 - 15 \text{ C}^\circ/\text{min}$ in the Al-corner of the Al-Si-Fe system is determined in the following work. For a preliminary analysis the equilibrium state was investigated by triangulation (creation of sub-triangles by use of quasi-binary lines between intermetallic compounds). The results show that four phase areas exist in the Al-corner. Depending on the Fe/Si-ratio of the alloy, it consists of following phases: 1) Al - Al_3Fe - $\text{Al}_8\text{Fe}_2\text{Si}$, 2) Al - $\text{Al}_8\text{Fe}_2\text{Si}$ - Al_5FeSi , 3) Al - Al_5FeSi - Al_4FeSi_2 , 4) Al - Al_4FeSi_2 - Si. Admittedly, in each of the equilibrium areas also phases coexist that do not completely react in corresponding peritectic reactions. The phases were determined by the combined results of metallographic, XRD and SEM analysis.

1 Introduction

In order to reach the new legislative recycling rates (regarding e.g. end-of-life vehicles, 95 % of materials have to be recycled by 2015) the material cycles have to be closed almost entirely. It is not possible to recover all metals in their pure form. Thus, secondary raw materials are often contaminated by each other. The complexity of such materials leads to difficulties in sorting as well as to an impurity pick-up during mechanical treatment processes. Since impurities such as iron accumulate in aluminium secondary materials, it becomes more and more difficult to produce Al-recycling-alloys conforming to standards. Therefore, the refining of aluminium melts is necessary, since the common dilution practise with primary aluminium becomes uneconomical.

The eutectic iron content in a pure aluminium - iron melt is 1,8 % at 655⁰C [1]. Therefore, in the case of hypereutectic alloys (over 1,8 % Fe), the iron content can not be reduced before reaching this value. Iron is present in the form of the intermetallic compound Al₃Fe, if the temperature is reduced below liquidus line (see Figure 1). Since this intermetallic phase has a melting point of 1060 °C and is rather insoluble in molten aluminium, it can be mechanically removed from molten aluminium, e.g. by filtration. In the cast alloy system Al – Si the ternary eutectic iron content is reduced to 0,7 % at 577°C [4]. In this system, besides in the phase Al₃Fe, iron is also present in phases like Al₈Fe₂Si, Al₅FeSi, Al₄FeSi₂. The addition of further alloying elements forms a quaternary or higher alloy with complex phase relationships. Ternary compounds with iron are formed for example in the Al – Si – Fe – Mn-system, and iron can be removed down to 0,29 % [5]. The current German standards regarding the Fe-content in cast Al-Si-alloys range between max. 0,9 % and max. 0,2 [6]. The target of this long-term research project at the IME, Aachen, is the precipitation of Fe from the Al – Si – Fe-system.

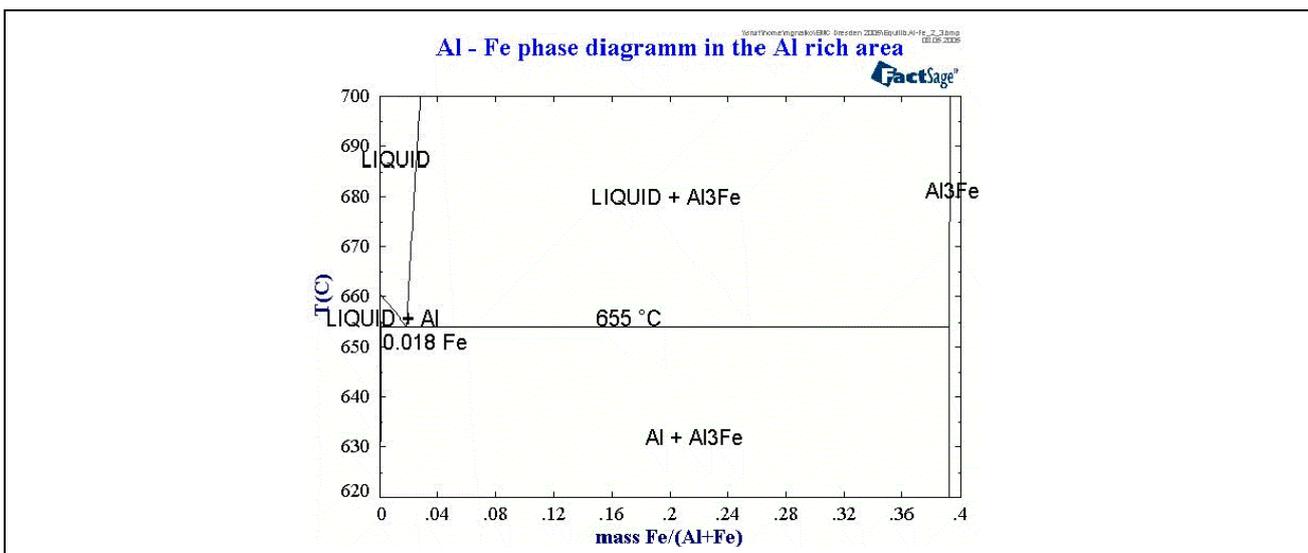


Figure 1: Al-Fe-phase diagram calculated with FactSage[®] thermodynamic program



2 Stable phases in the Al- corner of the Al-Si-Fe -system

The purification of aluminium scrap melts by the removal of intermetallic phases can be successful in melts, if, with falling temperatures, a phase containing impurity elements precipitates. The formation and segregation of the intermetallic particles are often very intensive, so that a concentration gradient in the melt can easily be demonstrated by means of the chemical analysis. The segregation occurs in the temperature interval, in which two phases exist: solid phase and melt. A big temperature interval, in which solid-liquid equilibria exist, is a pre-condition for successful phase separation.

The Al-Si-Fe-system is characterized by a number of ternary phases, and at least nineteen ternary invariant reactions during solidification are known. This makes it difficult to establish and draw the phase diagrams of the system. The complexity is extended by supercooling effects, incomplete reactions and metastability [4].

Commercial aluminium cast alloys contain up to 15 mass.-% silicon. Aluminium scrap, especially the Al-schredder fraction from end-of-life vehicles, is based on the mixture of cast and low alloyed wrought aluminium. This mixture is often contaminated with iron. This makes it necessary to understand the phase equilibria in the Al-corner of the Al-Si-Fe-system, if a purification by the removal of iron containing intermetallic phases is to be successful.

The iron-containing ternary phases present in this system corner are listed in Table 1. They have been assessed and selected from those reported in literature [4][2][7][8][9][10]. Those phases with overlapping composition ranges and the same crystal structure that were described by different authors as individual phases were regarded as one phase.

To define the phases precipitating in the system depending on the Fe/Si ratio, the so-called triangulation method can be applied, which is explained in the following chapter.

Table 1: Ternary phases and elements content (mass.-%) in the Al-corner of the Al-Si-Fe-system, selection based on [4][2][7][8][9][10]

Phase	Fe, Mass.-%	Si, Mass.-%	Al, Mass.-%	Lattice structure and parameter
Al ₈ Fe ₂ Si	30,0	7,4-11,0	56,0-62,6	cubic/hexagonal (after annealing) a=1256 pm/ a=1230 pm, c=2630 pm
Al ₅ FeSi	25,5-27,2	12,1-14,6	59,3-61,6	monoclinic a=b=612 pm, c=4150 pm, β=91°
Al ₄ FeSi ₂	15,0-27,04	20,0-25,0	48,0-65,0	tetragonal a=609 pm, c=944 pm



Phase	Fe, Mass.-%	Si, Mass.-%	Al, Mass.-%	Lattice structure and parameter
Al ₃ FeSi	33,0-38,0	13,0-18,0	44,0-54,0	monoclinic a=1356 pm, b=1025 pm, c=890 pm, β=102,82 °
Al ₂ FeSi	43,0	20,03	36,9	tetragonal a=616 pm, c=949 pm

3 Triangulation method for characterising ternary systems

3.1 Methodology

The triangulation can be applied for a preliminary analysis of phase equilibria in ternary systems, typically preparing experimental work [11]. Triangulation is a subcase of polyhedronation of multi-component systems. This procedure creates phase diagram sub-triangles by dividing the initial diagram by quasi-binary lines connecting the present compounds. The ternary system is broken into a series of sub-systems, whose corner components are compounds of the initial system. These sub-systems are autonomous and show equilibria for its compounds [12].

For example, for the division of a ternary system with two binary compounds in different sub-systems there are two possibilities of triangulation (Figure 2a). The first possibility means that the quasi-binary cuts A_XB-A_YC , A_XB-C divide the system $A-B-C$ into the three sub-systems $A-A_XB-A_YC$, $C-A_XB-A_YC$ and $B-C-A_XB$. In the second possible case the quasi-binary cuts A_XB-A_YC and $B-A_YC$ (dotted line) now divide the system into the three sub-systems $A-A_XB-A_YC$, $B-A_XB-A_YC$ and $B-C-A_YC$. In the first variant, the compound A_YC cannot be in equilibrium with component B, but the compound A_XB is in equilibrium with components B and C. In the second variant, A_YC is in equilibrium with B and C, and A_XB has no equilibrium with component C.

In the case that A_XB-C represents the quasi-binary equilibrium, no direct reaction occurs between C and A_XB , but compound A_YC reacts with B forming A_XB and C. In case of the mixture m1 the entire A_YC , in case of the mixture m2 the entire B is consumed.

If $B-A_YC$ is stable, no reaction occurs between B and A_YC , but compound A_XB reacts with C following the reaction: $A_XB + C = B + A_YC$.

Both types of triangulation of the system $A-B-C$ are correct, yet a mixture is not. Admitting the co-existent cuts A_XB and A_YC leads to the situation, in which four solid phases (B, C, A_XB , A_YC) would be in equilibrium at the crossing of cuts. Based on the phase rule ($F=C-P+1$), this is not allowed at all temperatures. To clarify which of the cuts exist an experimental investigation is neces-



sary. The phase distribution in the investigated alloys defines the existing cut: if A_xB-C is quasi-binary, the alloys of composition of this cut have a two-phase structure (A_xB-C), and the alloys of the A_yC-B cut have a three-phase structure ($A_xB-C- A_yC$ or A_xB-C-B).

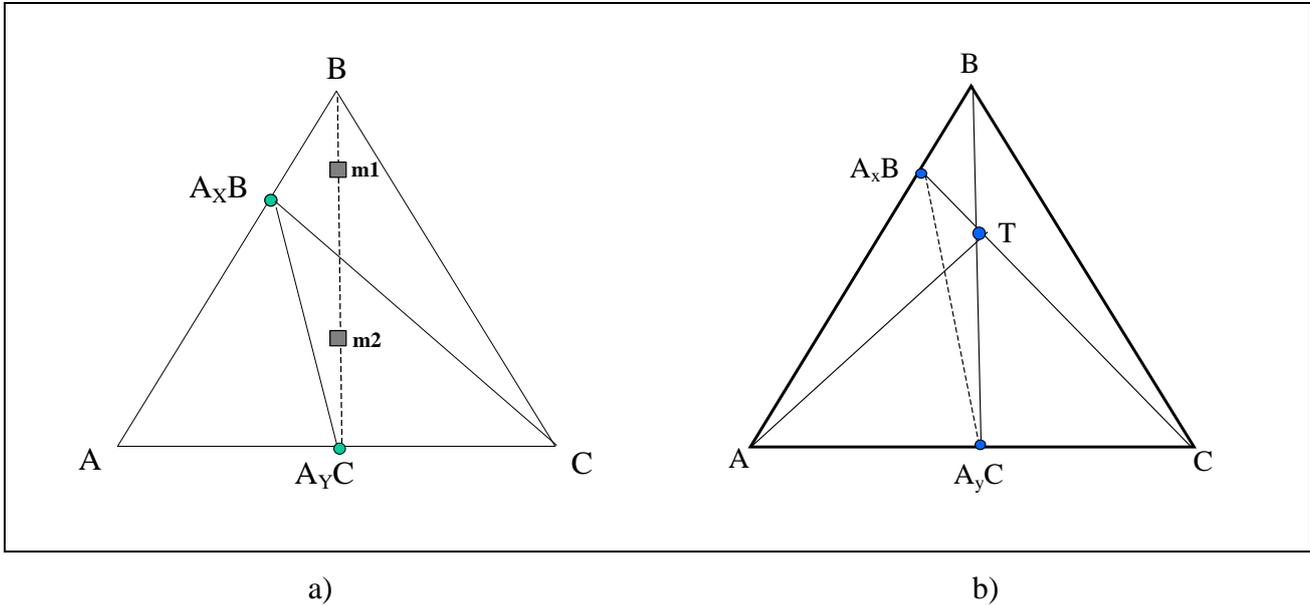


Figure 2: a: Triangulation of a system with the two binary compounds A_xB and A_yC
 b: Triangulation of a system with two binary and one ternary compounds

If the ternary alloy forms an intermetallic ternary compound T at the position of crossing lines (Figure 2b, discussed above), the cuts A_xB-T , $T-C$, $B-T$, $T- A_yC$ and the crossing cuts A_xB-A_yC and $A-T$ are quasi-binary and stable. Concerning the formation of a ternary compound (T), there are again two allowed possibilities to triangulate the ternary system A-B-C: by using quasi-binary cuts: A_xB-T , $T-C$, $B-T$, $T- A_yC$ and $A-T$ or A_xB-A_yC (dotted line). Which cut is quasi-binary, can only be established by experimental investigation of the phase composition of the ternary alloys of the corresponding crossing composition.

The number of the quasi-binary cuts R and of the sub-systems T in a ternary system that derive from the triangulation follow the rules [11]:

$$R = N_b + 3N_t \tag{1}$$

$$T = 1 + N_b + 2N_t \tag{2}$$

With: R – number of developed quasi-binary cuts; N_b – number of binary compounds in the ternary system; N_t – number of ternary compounds in the ternary system; T – number of developed ternary sub-systems.

For multi-component systems ($n>3$), the development of these so-called “clarifying intersections” (concept by W. Guertler [13]) were conducted by L. S. Palantik und L. I. Landau [14]. They, for the first time, performed the tetrahedronisation of quaternary systems. The tetrahedron space of a four-



component system is divided by quasi-binary planes in secondary tetrahedrons (comparable to the triangulation, but with planes instead of lines). For equilibrium conditions, each secondary tetrahedron is a system, in which all components are intermetallic compounds.

This division of multi-component systems in secondary sub-systems can answer the questions about the equilibrium reactions, the concentration areas, in which the reactions proceed, and the possible products that arise from these reactions. It is advisable before the investigation and development of a metallurgical process to start with the triangulation or tetrahedronisation of the respective metal systems.

3.2 Triangulation of the system Al – Si – Fe

It was reported in [15] that the line Al₃Fe – Si refers to a quasi-binary nature. This fact allows the first division of the system and, therefore, the isolation of the subsystem Al - Al₃Fe – Si in the Al-corner. The following ternary phases, reported in [4][2][7][8][9][10], correspond to this subsystem: Al₈Fe₂Si, Al₅FeSi, Al₄FeSi₂, Al₃FeSi (see Table 1). The triangulation (Figure 3a) refers to the complete system, but only the area in the Al-corner is relevant for the subsequent experimental investigation presented in this paper. According to the figure, four phase areas exist in this Al-corner, depending on the Fe/Si ratio of the alloy: 1) Al - Al₃Fe - Al₈Fe₂Si, 2) Al - Al₈Fe₂Si - Al₅FeSi, 3) Al - Al₅FeSi - Al₄FeSi₂, 4) Al - Al₄FeSi₂ – Si.

Figure 3b shows in terms of arrows the shape of the liquidus surface. They represent the surfaces of primary crystallisation of phases such as [4]:

- 1) Al₃Fe: p₁ – P₅ – P₁ – e₁ ,
- 2) Al₈Fe₂Si: P₅ – P₁ – P₂ – P₄,
- 3) Al₅FeSi: P₂ – E – P₄,
- 4) Al₄FeSi₂: P₆ – P₇ – P₃ – P₄,
- 5) (Al): e₁ – P₁ – P₂ – E – e₂.

With e₁: L_{655°C}= Al₃Fe +(Al), e₂: L_{577°C}=(Al)+(Si), p₁: L_{1160°C}+Al₅Fe₂= Al₃Fe,

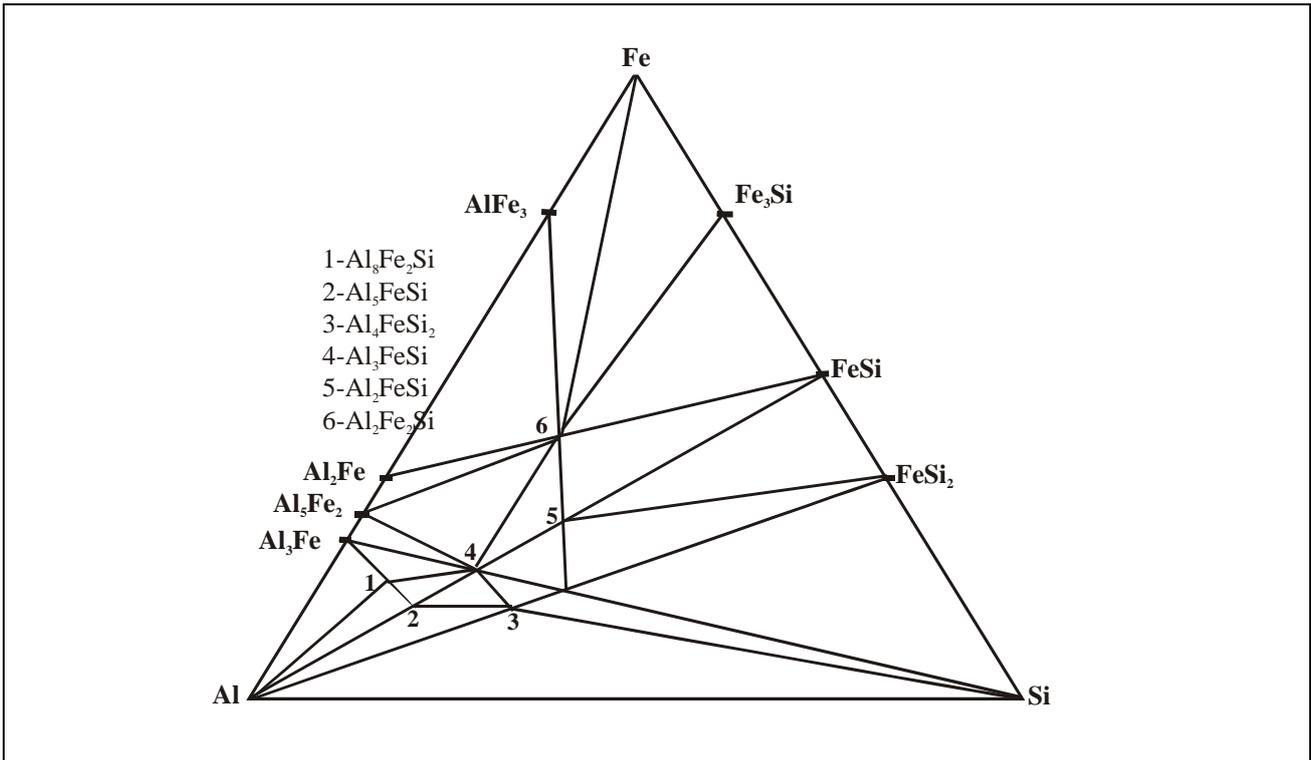
P₁: L_{630°C}+Al₃Fe=Al+Al₈Fe₂Si, P₂: L_{612°C}+Al₈Fe₂Si=Al+Al₅FeSi, P₃: L_{596°C}+Al₄FeSi₂=Si+Al₅FeSi,

P₄: L_{700°C}+Al₄FeSi₂+Al₈Fe₂Si=Al₅FeSi, P₅: L_{855°C}+Al₃Fe+Al₃FeSi=Al₈Fe₂Si,

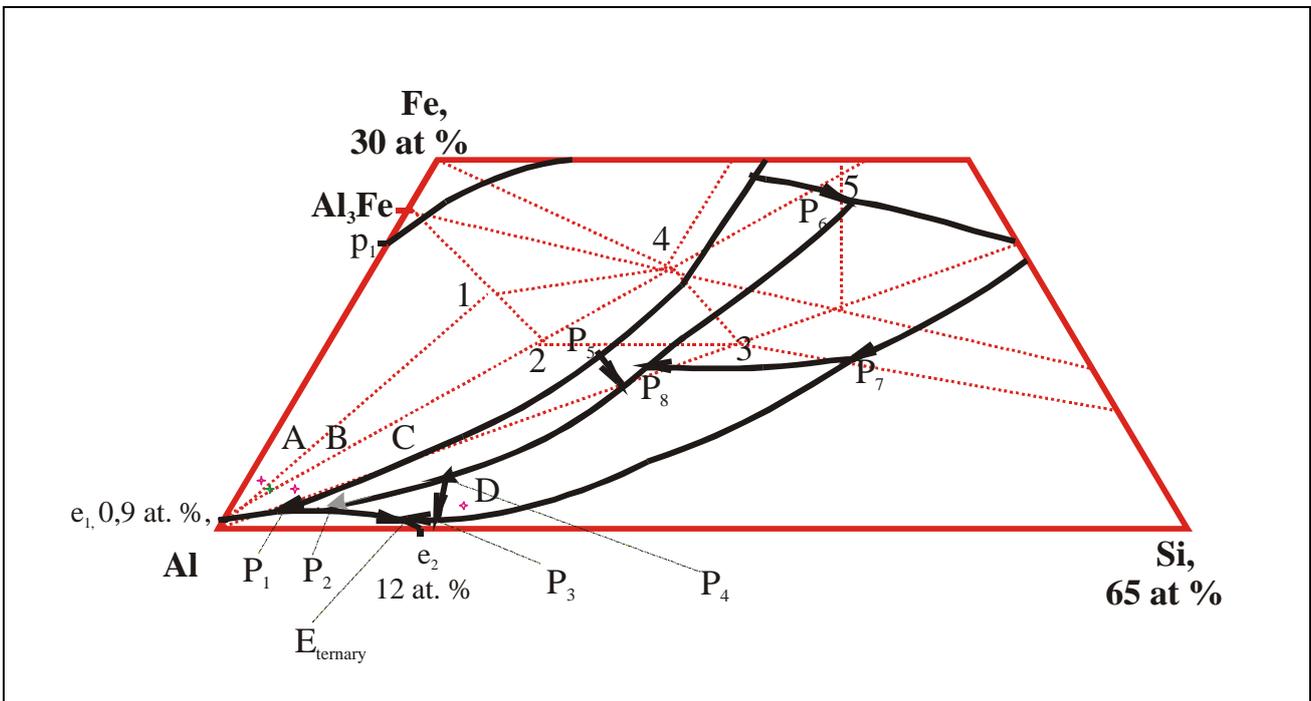
P₆: L_{935°C}+Al₂Fe₂Si+Al₃FeSi=Al₂FeSi, P₇: L_{865°C}+Al₂FeSi+Si=Al₄FeSi₂,

P₈: L_{835°C}+Al₂FeSi=Al₃FeSi+Al₄FeSi₂

The compositions of the investigated experimental samples from corresponding areas are marked by small crosses (+) and letters A, B, C, D, covering the four sub-triangles (phase areas).



a) triangulation of the system



b) liquidus surfaces of primary phase precipitation

Figure 3: Triangulation of the system Al-Si-Fe



4 Experimental

For the experimental investigation of the phase distribution after solidification, according to the four subsystems (points marked A, B, C, D in Figure 3b) samples were synthesised from each area. Table 2 describes the concentration areas and the sample data in details. The Fe:Si-ratio based on the triangulation results of the system Al-Si-Fe. The alloys were prepared by melting in a 100 ml/ 5 kW induction furnace. After casting the 50g samples in a copper mould, they were placed in a resistance furnace and heated to about 40 °C above the liquid temperature, followed by a cooling rate of 5 – 15 °C/min (see Figure 4). The melts were stirred every 3 minutes until solidification. Stirring avoids a separation by segregation of the heavier intermetallic phases from the aluminium matrix. Segregation could lead to a misinterpretation during the phase assessment in a sample. Quenching as an alternative to avoiding segregation was ruled out, because it could disable peritectic reactions, so that the formation of phases resulting from these reactions would not occur. All tests ran in room atmosphere. In all cases graphite crucibles were used. The composition of samples were chemically analysed by ICP. The compositions and Fe/Si-ratios of the four investigated samples are given in Table 2. Subsequently, SEM and XRD analysis were applied.

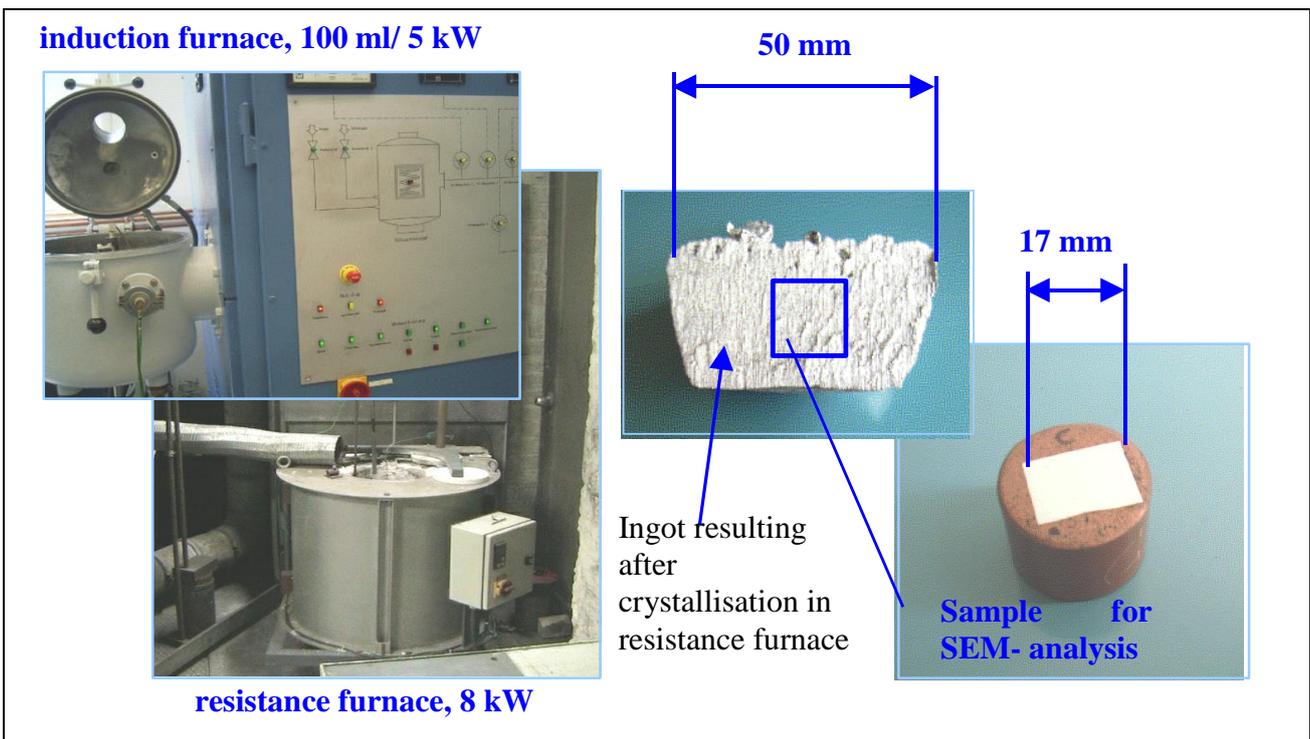


Figure 4: Experimental equipment, ingot after crystallisation and sample prepared for SEM-analysis

Table 2: Areas and investigated samples of the System Al-Si18-Fe6

Area	Al-Al ₃ Fe-Al ₈ Fe ₂ Si	Al-Al ₈ Fe ₂ Si-Al ₅ FeSi	Al-Al ₅ FeSi- Al ₄ FeSi ₂	Al- Al ₄ FeSi ₂ -Si
Point	A	B	C	D
	Fe/Si	Fe/Si	Fe/Si	Fe/Si
Mass.	>4:1	<4:1, >2:1	<2:1, >1:1	<1:1
At.	>2:1	<2:1, >1:1	<1:1, >1:2	<1:2
In the sample	5,68	3,33	1,56	0,16
Fe, mass. %	6,14	5,03	5,27	2,78
Si, mass. %	1,08	1,51	3,37	17,6

5 Results and Discussion

5.1 Fe-precipitation in the area Al - Al₃Fe - Al₈Fe₂Si, sample A

Figure 5 shows the micro-examination of sample A (6,14 mass. % Fe and 1,08 mass. % Si). Based on the SEM analysis, in this area iron precipitates in the phases with the composition shown in Table 3. These precipitations correspond to phases as given in the right column, according to Table 1.

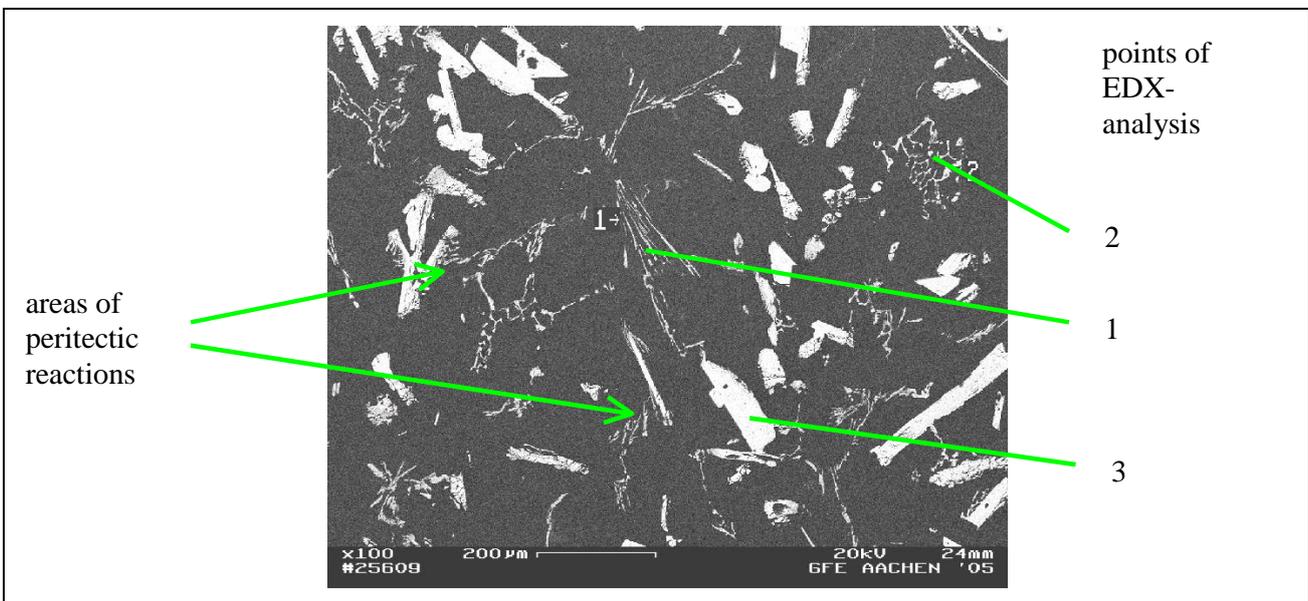


Figure 5: SEM-examination of sample A – iron precipitations in the area Al-Al₃Fe-Al₈Fe₂Si (40 °C overheated, cooling rate 15 K/min, about 20 minutes until solidification)

Table 3: Composition of iron precipitations in the area Al - Al₃Fe - Al₈Fe₂Si (SEM-EDX)



Point of EDX-analysis	Al, Mass. %	Fe, Mass. %	Si, Mass. %	Phase
1	60,92	26,38	12,7	Al ₅ FeSi
2	59,95	32,28	7,77	Al ₈ Fe ₂ Si
3	59,15	39,88	0,96	Al ₃ Fe

Some of the Al₃Fe-crystals show a sharp boundary (see arrows) and nearby net-forming precipitations of Al₈Fe₂Si, resulting from the peritectic reaction $L+FeAl_3=Al+Al_8Fe_2Si$. The occurrence of Al₅FeSi can only be explained by metastability. According to [4], in this system several metastable phases exist in the Al-corner. Their formation is reported to be a function of the Fe/Si-ratio, alloy purity, solidification rate and heat treatment.

5.2 Fe-precipitation in the area Al - Al₈Fe₂Si - Al₅FeSi, sample B

Figure 6 presents the metallographic picture of sample B (5,03 mass. % Fe and 1,51 mass. % Si).

According to the SEM analysis, iron precipitates in phases with the composition shown in Table 4.

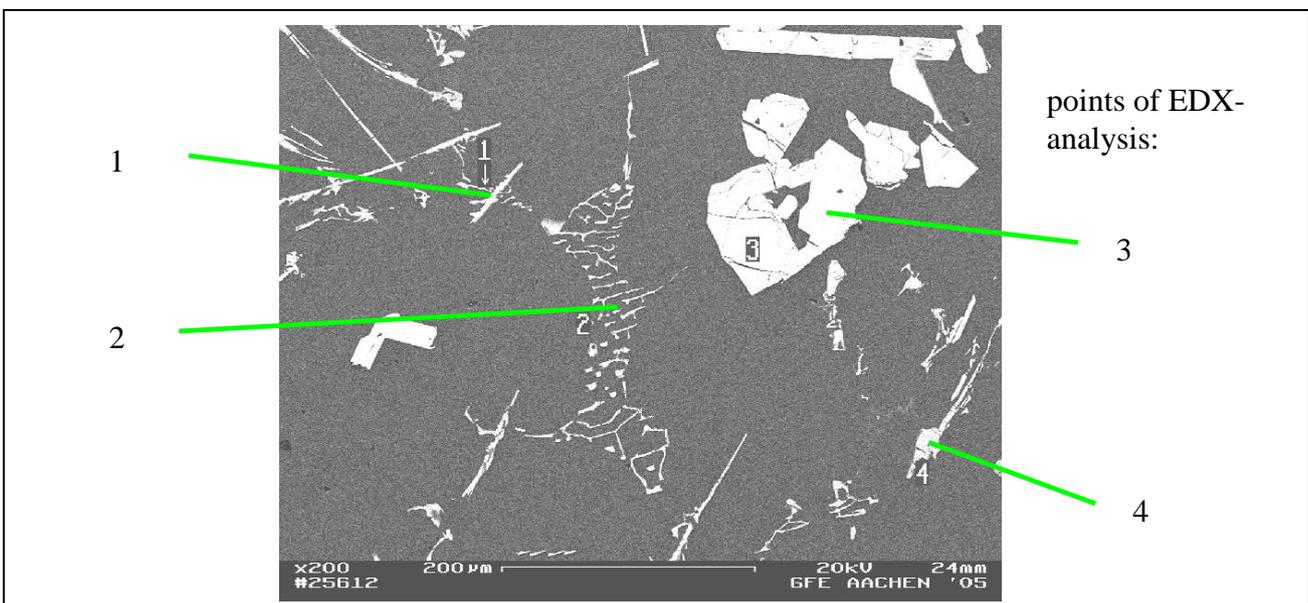


Figure 6: SEM-examination of sample B – iron precipitations in the area Al-Al₈Fe₂Si-Al₅FeSi (40 °C overheated, cooling rate 10 K/min, about 30 minutes until solidification)

Table 4: Composition of iron precipitations in the area Al - Al₈Fe₂Si - Al₅FeSi (SEM-EDX)

Point of EDX-analysis	Al, mass. %	Fe, mass. %	Si, mass. %	Phase
1	58,2	32,26	9,55	$\text{Al}_8\text{Fe}_2\text{Si}$
2	60,19	31,87	7,94	$\text{Al}_8\text{Fe}_2\text{Si}$
3	59,75	40,25	-	Al_3Fe
4	62,77	23,73	13,5	Al_5FeSi

As expected for this area, the phases $\text{Al}_8\text{Fe}_2\text{Si}$ and Al_5FeSi exist. The presence of Al_3Fe can be explained by the fact that the liquidus surface of this phase also overlaps a solid phase area, where it does not exist (Figure 3b, dotted lines). Thus, Al_3Fe primarily precipitates in the area $p_1 - P_5 - P_1 - e_1$. Since the peritectic reaction do not undergo completion (without long-time homogenisation), there could always be residual Al_3Fe -crystals in the alloys.

5.3 Fe-precipitation in the area Al - Al_5FeSi - Al_4FeSi_2 , sample C

Figure 7 shows the micro-examination of sample C (5,27 mass. % Fe and 3,37 mass. % Si). According to the SEM-analysis of this specimen, iron precipitates in the phases with the composition as given in Table 5.

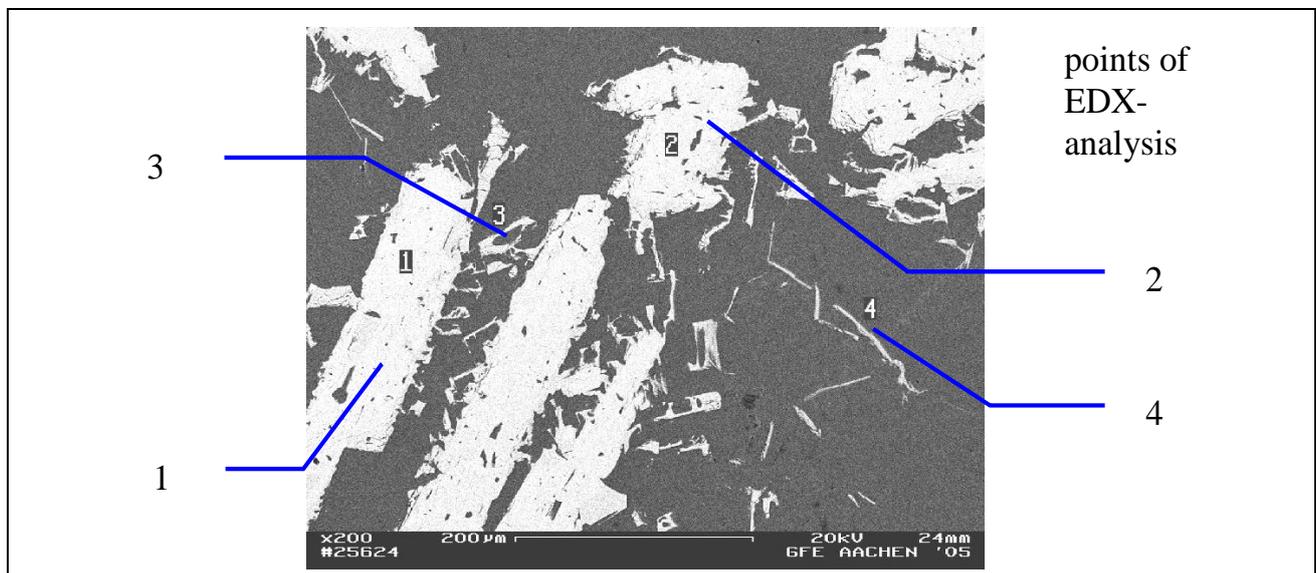


Figure 7: SEM-examination of sample C – iron precipitations in the area Al- Al_5FeSi - Al_4FeSi_2 (40 °C overheated, cooling rate 10 K/min, about 30 minutes until solidification)

The presence of Al_3Fe and $\text{Al}_8\text{Fe}_2\text{Si}$ (point 3) between its crystals points to a primary crystallisation of Al_3Fe , followed by the peritectic reaction $L + \text{FeAl}_3 = \text{Al} + \text{Al}_8\text{Fe}_2\text{Si}$, since the sample C has a com-



position, which is still under the liquidus surface of Al_3Fe (see also 5.2.). The precipitate (point 4) has an intermediate composition of the phases Al_5FeSi and Al_4FeSi_2 . The XRD-analysis of this sample proved the presence of both phases. Therefore, it can be supposed that the phase Al_4FeSi_2 is a result of the increasing Si-content in the phase Al_5FeSi . Diffusion does not completely proceed without homogenisation, and intermediate phases may exist in this area.

Table 5: Composition of iron precipitations in the area Al- Al_5FeSi - Al_4FeSi_2 (SEM-EDX)

Point of EDX-analysis	Al, mass. %	Fe, mass. %	Si, mass. %	Phase
1	58,43	39,65	1,92	Al_3Fe
2	58,04	41,04	0,92	Al_3Fe
3	57,26	34,69	8,05	$\text{Al}_8\text{Fe}_2\text{Si}$
4	56,73	25,52	17,75	$\text{Al}_4\text{FeSi}_2/\text{Al}_5\text{FeSi}$

5.4 Fe-precipitations in the area Al - Al_4FeSi_2 – Si, sample D

According to the SEM-analysis of the phases shown in Figure 8, iron precipitates in phases with the composition presented in Table 6.

(40 °C overheated, cooling rate 15 K/min, about 20 minutes until solidification)

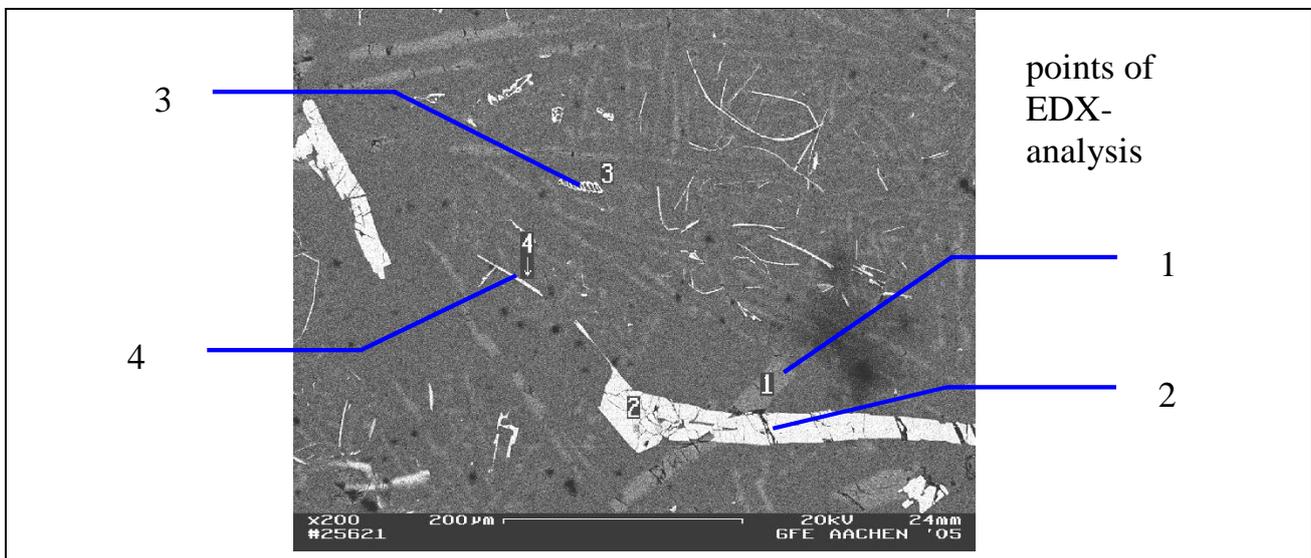


Figure 8: SEM-examination of sample D – iron precipitations in the area Al - Al_4FeSi_2 – Si

Dark grey crystals of Si (point 1) can easily be seen. The precipitation with number 3, a composition near that of the phase $\text{Al}_8\text{Fe}_2\text{Si}$, is reported to be metastable [4]. This phase can only be seen in a few points of the specimen. The needle-like white crystals with a content close to the phase Al_5FeSi



(and also Si-crystals) can result from the reaction $L + Al_4FeSi_2 = Al_5FeSi + Si$ at 600 °C [4]. But it is also possible that the phase Al_4FeSi_2 has a bright concentration range overlapping the composition of the phase Al_5FeSi in specific temperature areas. G. Phragmen [9] noticed three phases in the system with the compositions and lattice parameter shown in Table 7. It can be seen that the t-phase shows the tetragonal structure similar to that of Al_4FeSi_2 , The difference is based on the Fe- and Si-content. It can be supposed that they represent the same phase with a wide composition range, but precipitate under different conditions, thus resulting in the composition difference.

Table 6: Composition of the precipitations in the area Al - Al_4FeSi_2 - Si

Point of EDX-analysis	Al, mass. %	Fe, mass. %	Si, mass. %	Phase
1	-	-	100	Si
2	56,74	28,42	14,84	Al_4FeSi_2/Al_5FeSi
3	64,02	28,13	7,86	Metastable/ Al_8Fe_2Si
4	58,16	24,22	17,63	Al_4FeSi_2

Table 7: FeAlSi-phase compositions and lattice parameters reported by G. Phragmen [9]

Phase designation	Fe	Si	Al	Lattice parameter
c - phase	31,9	5,6	62,5	cubic: a=1254,83 pm
m - phase	27,2	13,5	59,3	monoclinic: a=b=612,23 pm, c=4148.36 pm, $\beta=91^\circ$
t - phase	27,8	14,0	58,2	tetragonal: a=612,23 pm, c=947,91 pm

6 Summary and Conclusion

The number of research reports about ternary phases in Al-Si-Fe-alloys is rather large. The phases often have a wide composition range. The composition of precipitated phases depends on the Fe/Si-ratio of the alloy. These factors probably explain the occurrence of so many ternary phases, as stated by different authors. In this work, the phases with overlapping composition ranges and the same



crystal structure, that were reported as individual phases by different authors, were regarded as one phase. Based upon this assumption, a triangulation of the Al-Si-Fe-system was applied for a preliminary analysis of the phase equilibrium. The results showed four areas: 1) Al - Al₃Fe - Al₈Fe₂Si, 2) Al - Al₈Fe₂Si - Al₅FeSi, 3) Al - Al₅FeSi - Al₄FeSi₂, 4) Al - Al₄FeSi₂ - Si.

Experimental investigations of the alloys of the system Al-Si-Fe, up to 18 mass. % Si and 6 mass. % Fe show the presence of the phases: Al₃Fe, Al₈Fe₂Si, Al₅FeSi, Al₄FeSi₂ and Si. Based upon the present experiments, the alloys, crystallised at a cooling rate of approximately 10 - 15 K/min do not reach the equilibrium state, as it happened by triangulation. In fact, some alloys also contained phases that do not correspond to the equilibrium state because the peritectic reactions did not undergo completion under the conditions of the experiments. Al₃Fe, present in the alloys from the areas Al - Al₈Fe₂Si - Al₅FeSi and Al - Al₅FeSi - Al₄FeSi₂, and Al₈Fe₂Si in the alloys from the area Al - Al₅FeSi - Al₄FeSi₂ are residues of uncompleted peritectic reactions. The precipitation of Al₃Fe is more favourable, since this phase contains more iron than another phases, which must be removed from the melt. It must be considered that a melt composition also differs from the equilibrium. Determining the melt composition of Al-Si-melts by crystallisation under non-equilibrium conditions is the following and final step of examining the Al-Si-Fe system in terms of the refining by intermetallic Fe-precipitations.

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