Aluminium – Calcium – Silicon

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Literature Data

The liquidus surface of the Al-corner has been studied and fundamental agreement was obtained between most of the earlier experimental work [1926Doa, 1927Gro, 1928Shi] and reviewing papers [1934Fus, 1943Mon, 1952Han, 1960Spe, 1969Wat]. Samples were made from Al-Ca and Al-Si master alloys under a $CaCl_2$ or KCl-NaCl-$BaCl_2$ protective layer. Their composition was checked by chemical analysis, and thermal and metallographic analyses were performed [1926Doa]. A dominating $CaSi_2$ liquidus surface extending close to the Al-corner and an eutectic type Al-$CaSi_2$ pseudobinary system was deduced from these data [1926Doa] and essentially confirmed by similar studies [1928Shi, 1962Kol] and [1966Tom]. The coexistence of (Al) and a compound, assumed to be $CaSi_2$, was also seen in micrographs of Al-rich samples that were cast, forged or hot rolled, annealed for 30 min at 460°C and then used for Brinell and tensile tests [1927Gro]. Similar interpretations are given in [1934Fus, 1943Mon] and also by [1952Han] who made additional experiments to quantify the location of ternary eutectics and accepts the binary phase $CaAl_4$ instead of $CaAl_3$ as given in previous literature. The interpretation of an (Al)-$CaSi_2$ equilibrium is, however, inconsistent with reports on some ternary compounds that form after annealing for a long time. [1967Gla] studied 39 samples in the Al-Si-$CaSi_2$-$CaAl_2$ subsystem at 400°C by X-ray methods and metallography and detected the compound $CaAl_2Si_2$ (β) in equilibrium with (Al), (Si), $CaSi_2$, $CaAl_4$, $CaAl_2$ and another ternary compound, $CaAl_{1-x}Si_{1+x}$(γ) [1965Bod]. [1967Gla] also prepared single crystals of β and performed a detailed X-ray structure analysis. The β phase was also observed by electron microprobe in Al-20 mass% Si samples containing 0.13 to 2.6 mass% Ca, which were equilibrated at 800°C, cast and found to consist of (Al)+(Si)+β [1976Tag]. The precipitation of β and Ca_2Si was found in metallurgical grade silicon (< 0.5 mass% Al, <0.3 mass% Ca) by optical metallography and electron microscopy (SEM, TEM, EMPA) [1990Ang].

The $CaAl_{1-x}Si_{1+x}$(γ) phase was also prepared at $x = 0$ by [2002Ima] and in the range $-0.4 < x < 0.2$ by [2002Lor], studied by X-ray diffraction and also for their superconducting behavior with a transition temperature of 7.8 K for CaAlSi [2002Ima].

The congruent melting point of β was reported at 975°C by DTA [1994Ang] and the pseudobinary eutectic L⇌β+(Si) at 927°C, and the "pseudobinary" eutectic L⇌β+$CaSi_2$ at 925°C. [1994Ang] also performed a brief Calphad-type thermodynamic analysis of the ternary system and produced calculated isopleths. This included the modelling of the Ca-activity in ternary liquids on the $Al_{0.5}Si_{0.5}$ - Ca section at 1350°C. This Ca-activity was experimentally determined by [1975Sch] with 11 alloys at 1350°C and 3 alloys at 1210°C using the boiling point method. The activity coefficients of Al and Ca in molten Si at 1450-1550°C were determined by the Knudsen effusion method and also by a chemical equilibrium technique from the distribution between liquid silicon and lead at 1450°C [1999Mik]. Two other compounds, $Ca_3Al_6Si_2$ (δ) and $Ca_2Al_4Si_3$ (ε) were prepared by pressing $CaAl_2$-Si mixtures, heating to 700-1000°C, quenching and studying by X-ray analysis [1955Chr, 1956Chr]. The reaction was complete only above 900°C and the mutual equilibria $CaAl_2$+δ, δ+ε and ε+(Si) were observed at 1000°C, but not the β phase located between ε and (Si). The δ phase completely decomposes into ε and some Al and Ca at 1200°C, while the ε phase only starts to decompose at this temperature, probably due to Ca-loss to the gas phase [1956Chr]. The most Ca-rich compound, $Ca_3Al_2Si_2$ (α) was fused from the elements in corundum crucibles under argon, continuously agitated for 4h at 1150-1200°C and slowly cooled to room temperature within 12h. Single crystals could be extracted from the sample and a detailed X-ray structure analysis was performed [1977Wid]. The composition change during preparation was found to be negligible by chemical analysis of a similarly prepared $Ba_3Al_2Si_2$ compound [1977Wid].

The present evaluation continues and updates the one published in [1990Sch] with respect to both new ternary and binary data.

Binary Systems

The Al-Si system is accepted from [Mas2]. The Al-Ca system is taken from [2002Ted]. The Ca-Si system is accepted from [2000Man], however, the shape of the liquidus lines may need some revision as indicated by thermodynamic calculations [2003Gro].

Solid Phases

Data on all solid phases reported in this system are given in Table 1.
Electronic structure calculations of the stability of $Al_2Si_2^{6-}$ chains in the compound $Ca_3Al_2Si_2$ suggest that all Si atoms reside in the twofold sites and all Al atoms in the threefold ones [1988Li].
Bonding in the $CaAl_2Si_2$ (β) structure type was studied theoretically [1988Zhe]. The same structure type was experimentally found to form also in 13 different, though related, ternary systems [1980Klu].
[1975Eml] mentioned the possible existence of a phase $Ca_{0.8}Al_{1.2}Si$, which is not included in Table 1.

Pseudobinary Systems

The Al-$CaSi_2$ section has been quoted as a eutectic pseudobinary system in the earlier literature [1926Doa] to [1952Han]. The primary crystallization of $CaSi_2$ from Al-rich liquids is probably metastable in view of the formation of β on that section. The β phase may have been misinterpreted as $CaSi_2$ in the metallographic examination, since the conclusions in the basic early work on Al-Ca-Si [1926Doa] rely on the assumption that no ternary compounds exist. The calculated isopleths Al-$CaSi_2$ and Si-$CaAl_2$ [1994Ang] contain the β phase, however, they are based on old versions of the Ca-Si and Al-Ca binaries and cannot be accepted. In addition, the phases α, γ, δ and ϵ had not been considered by [1994Ang]. Thermodynamic calculations performed in the present assessment were based on the recent binaries and they show that both sections are not pseudobinary systems. The Al-poor side of the Al-$CaSi_2$ section exhibits phase fields of liquid with CaSi and $Ca_{14}Si_{19}$. However the partial section Al-$CaAl_2Si_2$ (β) is a pseudobinary eutectic as shown in Fig. 1, according to the present calculation. Similarly, the Si-poor side of the Si-$CaAl_2$ section exhibits phase fields of liquid with CaSi. However the partial section Si-$CaAl_2Si_2$ (β) is also a pseudobinary eutectic as shown in Fig. 2, according to the present calculation. The calculated eutectic temperature 933°C is in close agreement with the experimental value 927°C [1994Ang].

Invariant Equilibria

The three invariant equilibria of the Al-corner, given in Table 2, are from the present thermodynamic calculation. Earlier work [1952Han] assumed the participation of $CaSi_2$ instead of β in the equilibria max, E and D (with reported temperatures of 637, 615.8, and 576.5°C). This cannot be accepted as described in the previous section. Also a eutectic reaction L⇌(Al)+(Si)+β was given with a liquid composition of 0.7 at.% Ca [1966Tom]. This is virtually impossible based on the thermodynamic calculation, which shows that this invariant (D) is essentially degenerate to the binary Al-Si eutectic. [1966Tom] also reported from microradiograph examinations that the Ca-content in (Si) is much smaller than in (Al).

Liquidus Surface

The liquidus surface of the Al-corner given in Fig. 3 is from the present thermodynamic calculation. It deviates from that given by [1952Han] as discussed in the previous section.

Isothermal Sections

An isothermal section at about 400°C is given in Fig. 4 [1967Gla, 1955Chr] and [1956Chr]. The equilibria around the ε and δ phases are estimated by dashed lines. Both phases form above 900°C with appreciable reaction rates [1955Chr] and are presumably stable down to 400°C. The reported ε - (Si) equilibrium at 1000°C [1955Chr] cannot be accepted in view of the congruent melting point of β at 975°C.
The equilibria above 33 at.% Ca have not bee studied experimentally. The dashed tie line Ca_5Si_3-$CaAl_2$ (and the three more Ca-rich ones) given dashed in Fig. 4 are based on the present thermodynamic

calculation, disregarding the α and γphases and the mutual solubilities along the $CaAl_2$-$CaSi_2$ section. No tie lines can be given around these phases.
The precipitation of β and Ca_2Si in metallurgical grade silicon (< 0.5 mass% Al, <0.3 mass% Ca) [1990Ang] supports the existence of the (Si)+ β+Ca_2Si equilibrium with negligible solubility in (Si).

Thermodynamics

The measured Ca-activities [1975Sch] in the ternary liquid phase along the equal molar fractions of Al and Si at 1350°C are well represented by the thermodynamic calculation of [1994Ang] shown in Fig. 5

Notes on Materials Properties and Applications

Si and Ca are important additions to Al-alloys. The $CaAl_{1-x}Si_{1+x}$(γ) phase shows superconducting behavior [2002Ima, 2002Lor]. The de-oxidation of steel using complex Ca-Al-Si de-oxidizers was discussed by [1983Gho].

Miscellaneous

The dissolution of $CaSi_2$ from Al-Ca-Si alloys in HCl was discussed by [1953Tou].

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Table 1: Crystallographic Data of Solid Phases

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
(βAl)	*hP*2 $P6_3/mmc$ Mg	a = 269.3 c = 439.8	at 25°C, 20.5 GPa [Mas2]
(αAl) < 660.452	*cF*4 $Fm\bar{3}m$ Cu	a = 404.96	at 25°C [Mas2]
(γCa)	?	?	at 25°C, 1.5 GPa [Mas2]
(βCa) 842-443	*cI*2 $Im\bar{3}m$ W	a = 448.0	[Mas2]
(αCa) < 443	*cF*4 $Fm\bar{3}m$ Cu	a = 558.84	at 25°C [Mas2]
(Si) < 1414	*cF*8 $Fd\bar{3}m$ C-diamond	a = 543.06	at 25 °C [Mas2]
$CaAl_4$ (h) 700 - 170	*tI*10 *I4/mmm* Al_4Ba	a = 435.3 b = 1107	[V-C2]
$CaAl_4$ (l) < 170	*m**10 ? ?	a = 615.26 ± 0.15 b = 617.30 ± 0.13 c = 632.90 ± 0.14 β = 118.026 ± 0.016	[1993Mil]
$CaAl_2$ < 1086	*cF*24 $Fd\bar{3}m$ Cu_2Mg	a = 804.0	[V-C2] Powder X-ray diffraction
$Ca_{13}Al_{14}$ < 633	*mC*54 *C2/m* $Ca_{13}Al_{14}$	a = 1555.1 ± 0.4 b = 987.3 ± 0.2 c = 972.6 ± 0.2 β = 108.09 ± 0.02°	[1998Hua] Single-crystal X-ray diffraction

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
Ca_8Al_3 < 578	*aP*22 $P\bar{1}$ Ca_8In_3	*a* = 948.4 ± 0.3 *b* = 959.2 ± 0.3 *c* = 967.1 ± 0.3 α = 99.02 ± 0.03° β = 101.13 ± 0.03° γ = 119.55 ± 0.03°	[1998Hua] Single-crystal X-ray diffraction
$CaSi_2$ < 1030	*hR*18 $R\bar{3}m$ $CaSi_2$	*a* = 386.3 *c* = 3071.0	[2000Man]
$Ca_{14}Si_{19}$ 1085- ≈900	*hR*198 $R\bar{3}c$ $Ca_{14}Si_{19}$	*a* = 867.2 *c* = 6844.5	[2000Man]
Ca_3Si_4 <≈ 910	*hP*42 $P6_3/m$ Ca_3Si_4	*a* = 854.1 *c* = 1490.6	[2000Man]
CaSi < 1320	*oC*8 *Cmcm* CrB	*a* = 459 *b* = 1079.5 *c* = 391	[2000Man]
Ca_5Si_3 < 1240	*tI*32 *I4/mcm* Cr_5B_3	*a* = 764.1 *c* = 1487.6	[2000Man]
Ca_2Si < 1270	*oP*12 *Pnma* anti-$PbCl_2$	*a* = 766.7 *b* = 479.9 *c* = 900.2	[2000Man]
* α, $Ca_3Al_2Si_2$	*oI*14 *Immm* $Ca_3Al_2Ge_2$	*a* = 400 ± 1 *b* = 1824 ± 2 *c* = 457.6 ± 1.0	ordered variant of Ta_3B_4 [1977Wid]
* β, $CaAl_2Si_2$ < 975	*hP*5 $P\bar{3}m1$ La_2O_2S	*a* = 413 ± 1 *c* = 714.5 ± 1.5	La_2O_3-type superstructure [1967Gla]. Congruent melting [1994Ang]
* γ, $CaAl_{1-x}Si_{1+x}$	*hP*3 AlB_2	*a* = 419.05 *c* = 439.92	at *x* = 0 [2002Ima] composition range *x* = -0.35 to + 0.55 [1967Gla] or *x* = -0.4 to + 0.2 [2002Lor]
* δ, $Ca_3Al_6Si_2$ <≈ 1150	*hP*	*c*/*a* = 1.64	[1956Chr]
* ε, $Ca_2Al_4Si_3$	*cP*18	*a* = 715	[1956Chr]

Table 2: Invariant Equilibria in the Al-rich Corner

Reaction	*T* [°C]	Type	Phase	Composition (at.%)		
				Al	Ca	Si
$L + CaAl_2 \rightleftharpoons \beta + CaAl_4$	688	U	L	85.4	11.7	2.9
			$CaAl_2$	66.7	33.3	0
			β	40	20	40
			$CaAl_4$	80	20	0
$L \rightleftharpoons (Al) + \beta$	649	e_{max}	L	97.96	0.65	1.39
$L \rightleftharpoons (Al) + \beta + CaAl_4$	610	E	L	94	5.4	0.6
			(Al)	99.99	0	0.01
			β	40	20	40
			$CaAl_4$	80	20	0
$L \rightleftharpoons (Al) + (Si), \beta$	577	D	L	87.9	0	12.1
			(Al)	98.5	0	1.5
			(Si)	0	0	100
			β	40	20	40

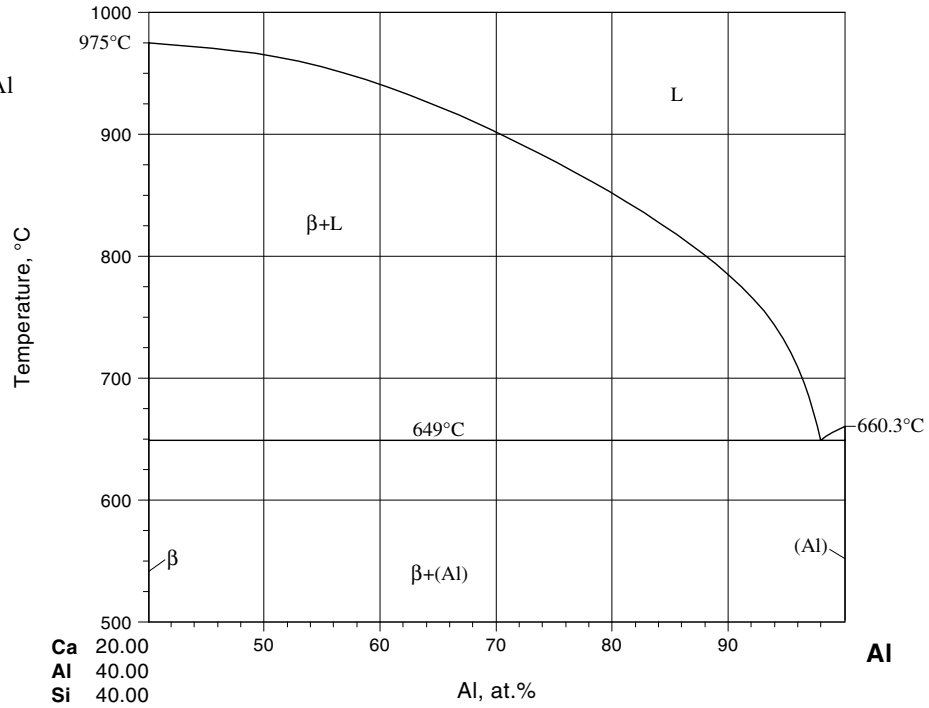

Fig. 1: Al-Ca-Si. The pseudobinaty section $CaAl_2Si_2$ - Al

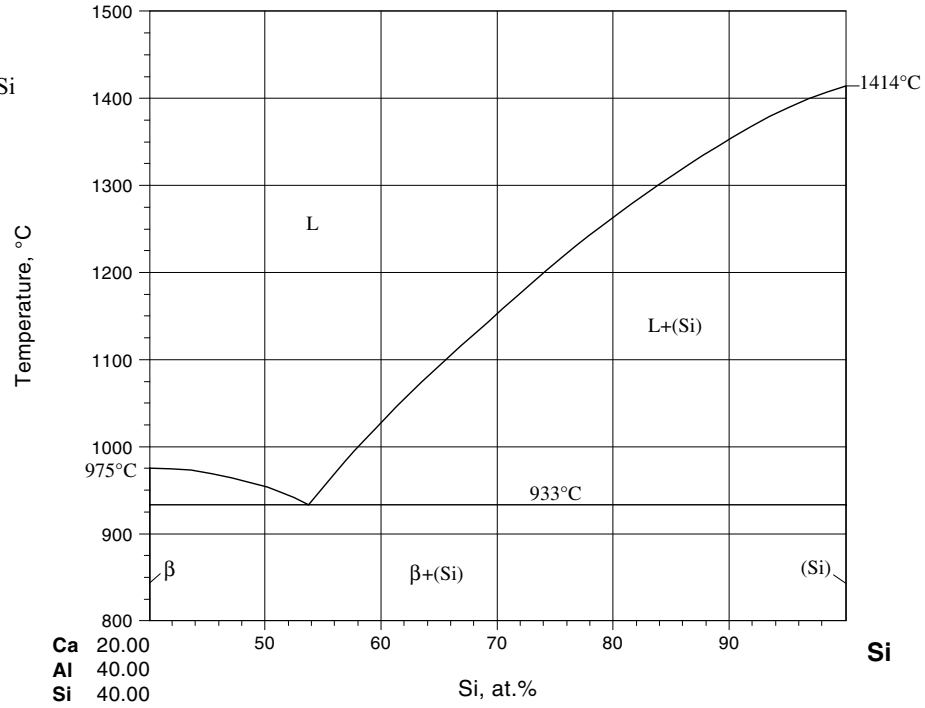

Fig. 2: Al-Ca-Si. The pseudobinary section $CaAl_2Si_2$ - Si

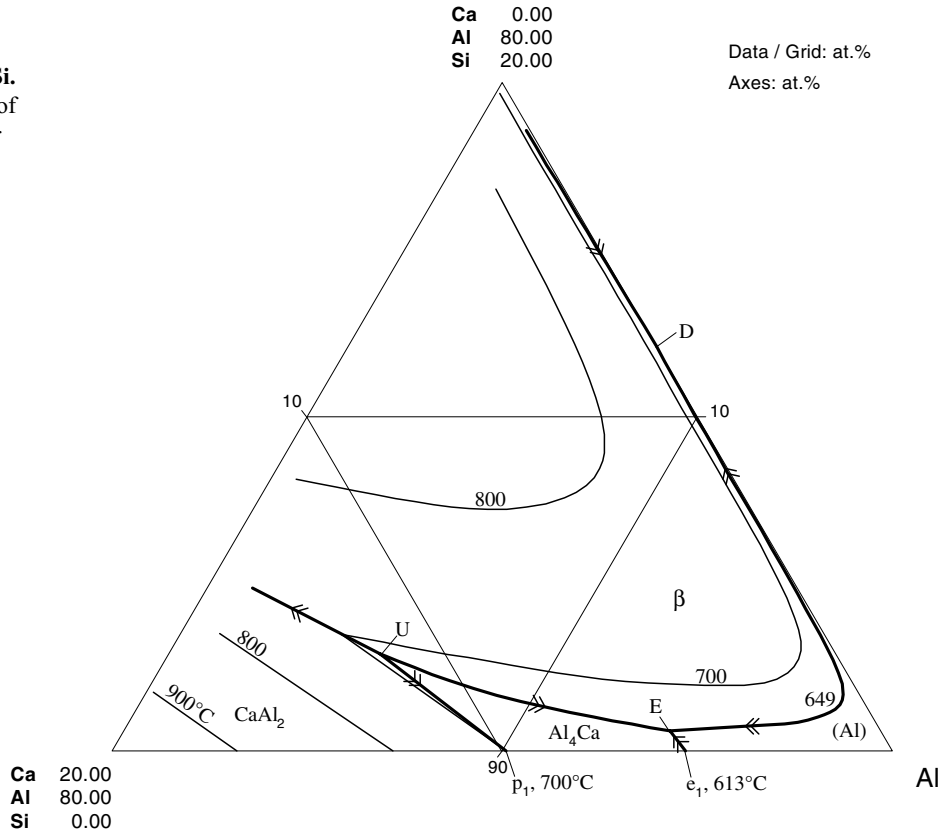

Fig. 3: Al-Ca-Si. Liquidus surface of the Al-rich corner (>80 at.% Al)

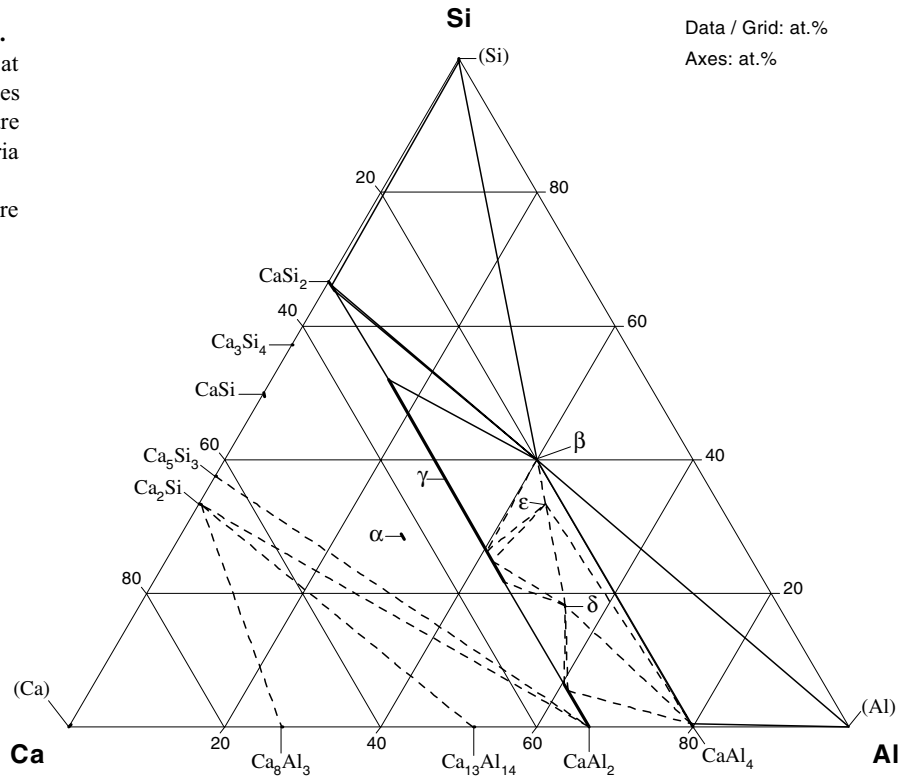

Fig. 4: Al-Ca-Si. Isothermal section at 400°C; some tie lines above 33 at.% Ca are estimated. Equilibria with the phases α, CaSi, and Ca_3Si_4 are not given

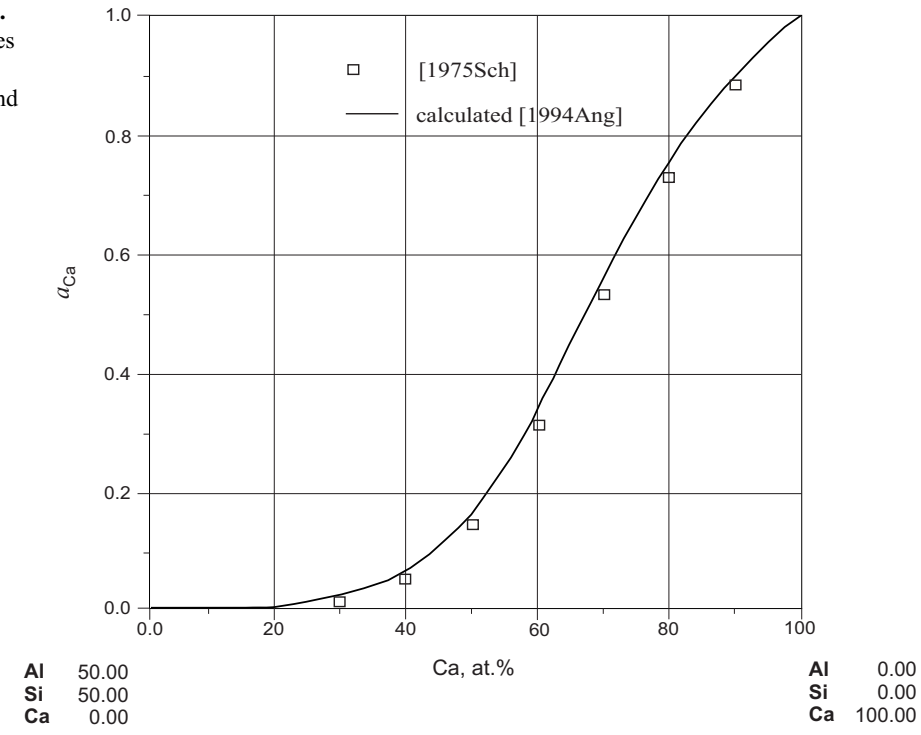

Fig. 5: Al-Ca-Si. Calculated activities of Ca in the liquid phase at 1350°C and $x_{Al} = x_{Si}$