Inclusion of CaF_2 and P_2O_5 in the GTT Oxide database

GTT-Technologies, Herzogenrath
Forschungszentrum Jülich

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- Addition of CaF2
- Addition of P_2O_5
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- Future developments

Elephant's wedding

Al_2O_3-K_2O-Na_2O-SiO_2

Al_2O_3-CaO-CrO_x-FeO-Fe_2O_3-MgO-SiO_2

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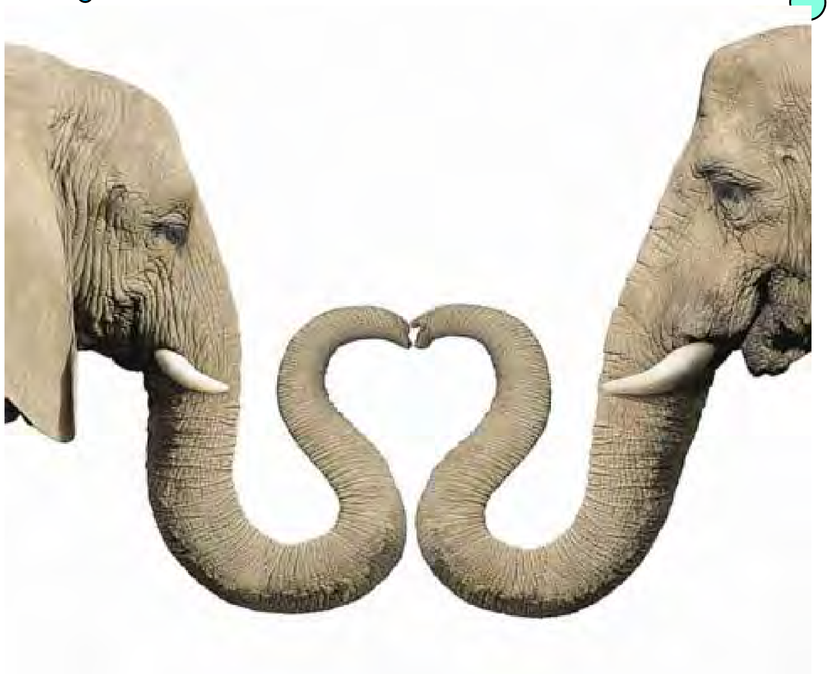


GTT-TECHNOLOGIES

Introduction

CaF_2

The addition of CaF_2 (Fluorspar) to the flux decreases melting point.

Fluorspar decreases the viscosity of the slags.

Fluorspar improves the fluidity of molten slags.

P_2O_5

The dephosphorization is important in the iron and steel industry.

The phospates are of interest in connection with soil-fertilizer relationships.

Addition of CaF_2

- The Al_2O_3-CaF_2 binary system
- The CaF_2-CaO binary system
- The CaF_2-FeO-Fe system
- The CaF_2-Fe_2O_3-O_2 system
- The CaF_2-MgO binary system
- The CaF_2-SiO_2 binary system

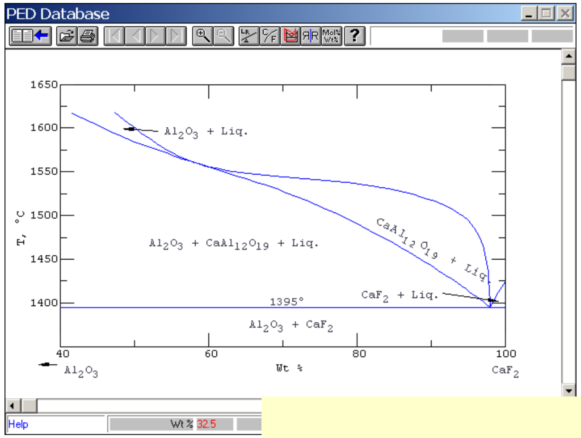
- The Al_2O_3-CaF_2-CaO ternary system
- Liquidus surface in Al_2O_3-CaF_2-MgO system
- Miscibility gaps in Al_2O_3-CaF_2-SiO_2 system
- Liquidus surface in CaF_2-CaO-MgO system
- The CaF_2-CaO-SiO_2 ternary system

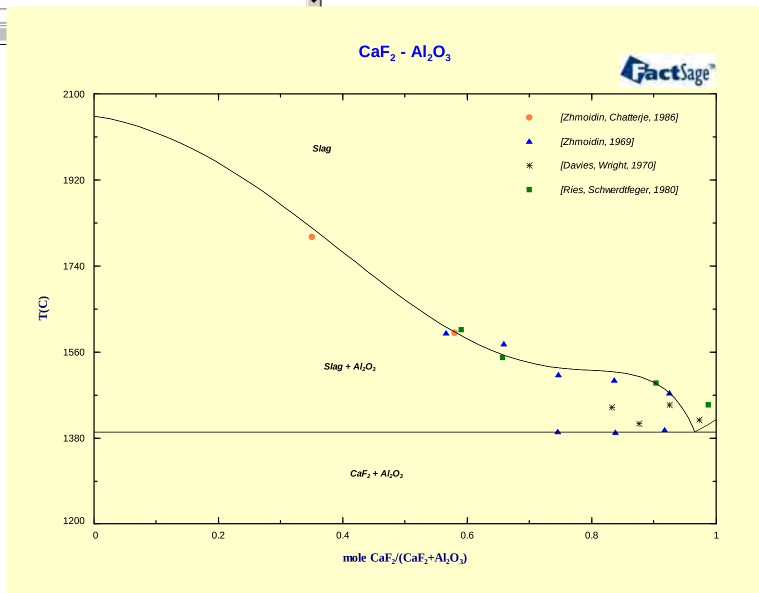
Ternary stoichiometric phases modelled by GTT

Name	Phase	System	Description
C3A3F	3CaO $3Al_2O_3$ CaF_2	Al_2O_3-CaO-CaF_2	$Ca_4Al_6F_2O_{12}$
C11A7F	11CaO $7Al_2O_3$ CaF_2	Al_2O_3-CaO-CaF_2	$Ca_{12}Al_{14}F_2O_{32}$
C3S2F Cuspidine	3CaO $2SiO_2$ CaF_2	CaF_2-CaO-SiO_2 Heat of formation [Fukuyama, Tabata,2003] is used	$Ca_4Si_2F_2O_7$
C4S2F	4CaO $2SiO_2$ CaF_2	CaF_2-CaO-SiO_2	$Ca_5Si_2F_2O_8$
C9S3F	9CaO $3SiO_2$ CaF_2	CaF_2-CaO-SiO_2	$Ca_{10}Si_3F_2O_{15}$

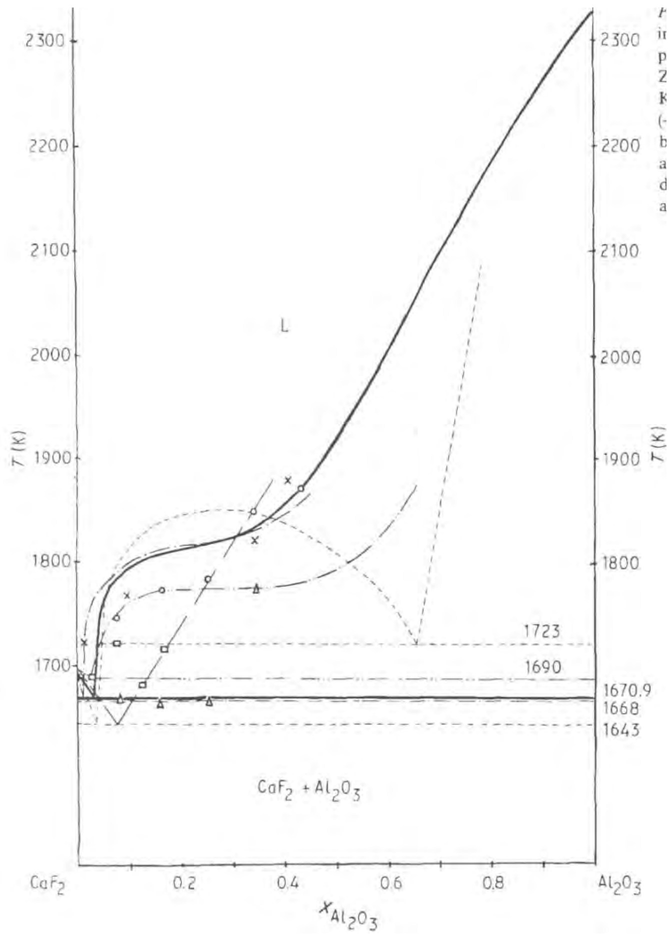
Binary Al_2O_3-CaF_2 phase diagram

A.K. Chatterjee, G.I. Zhmoidin, J. Mater. Sci., 7 [1], (1972), pp. 93-97.



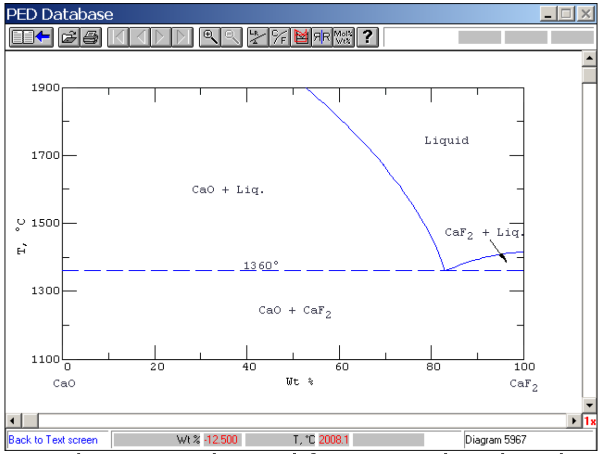


A,I, Zaitsev, N.V. Korolyov, B.M. Mogutnov, Journ. Mater. Scien., 26 (1991), pp.1588-1600.

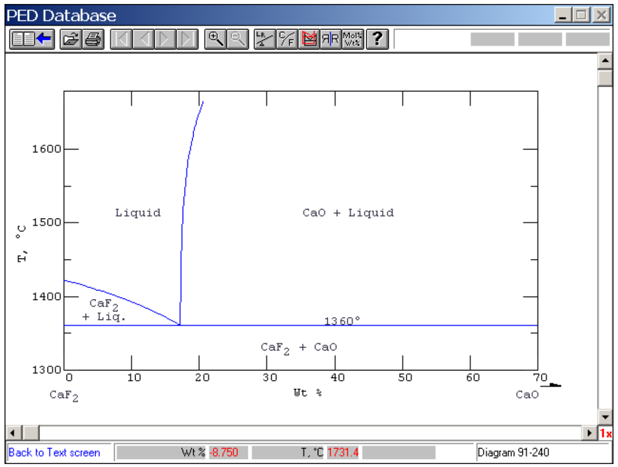


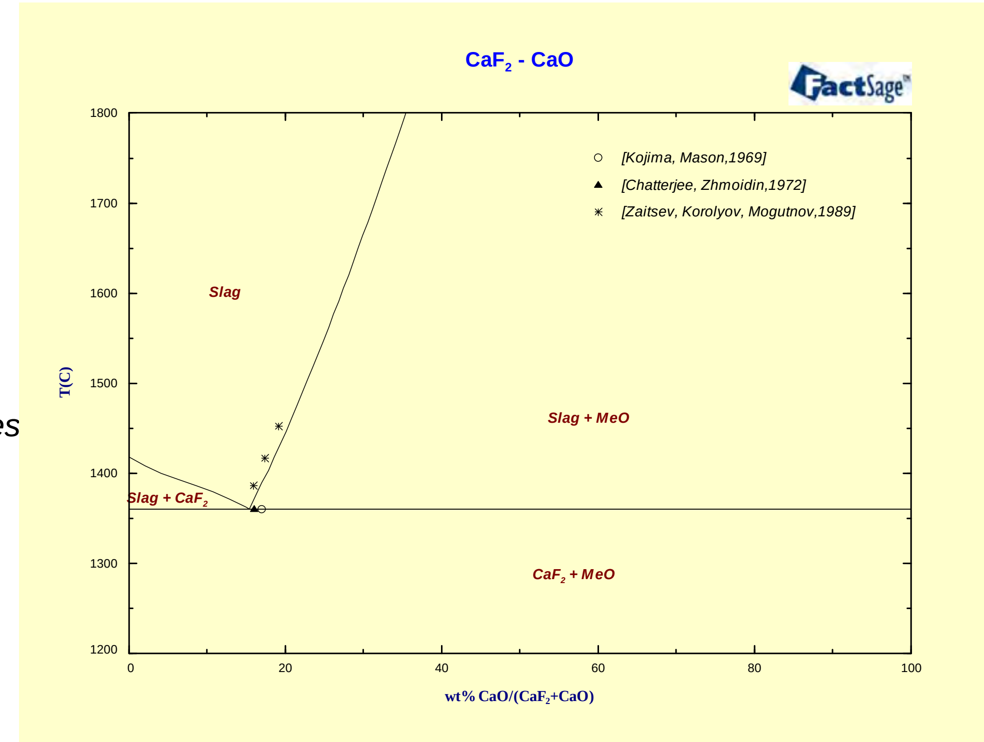
Binary CaF_2-CaO phase diagram

A.K. Chatterjee, G.I. Zhmoidin, J. Mater. Sci., 7 [1], (1972), pp. 93-97.



R. Ries, K. Schwerdtfeger, Arch. Eisenhuettenwes [4], (1980), pp. 123-129.





CaF_2-FeO phase diagram for equilibrium with Fe

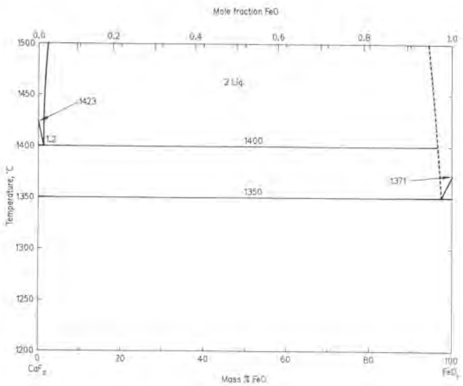
Slag Atlas, 2nd Ed., Verlag Stahl-Eisen, Düsseldorf, 1995.

CaF_2-FeO_x

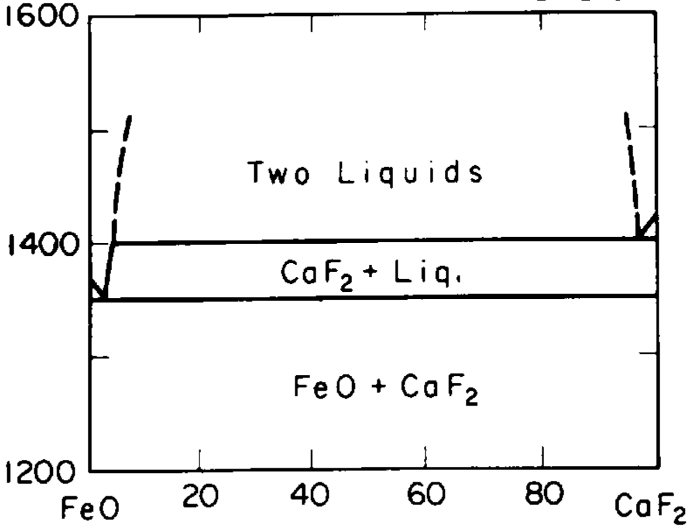
Fig. 3.413. CaF_2-FeO system in contact with metallic iron after Körber, Oelsen [1], Oelsen, Maetz [2] and Kay et al. [3] (extension of the miscibility gap in the CaF_2-rich region of the system between 1420 and 1500 °C). The solubility of FeO in CaF_2-rich melts at 1450 °C up to approx. 2 mass % FeO has been reported by Hawkins, Davies [4]. After Mitchel [5], the miscibility gap at 1500 °C range from 15 to 90 mass % FeO.

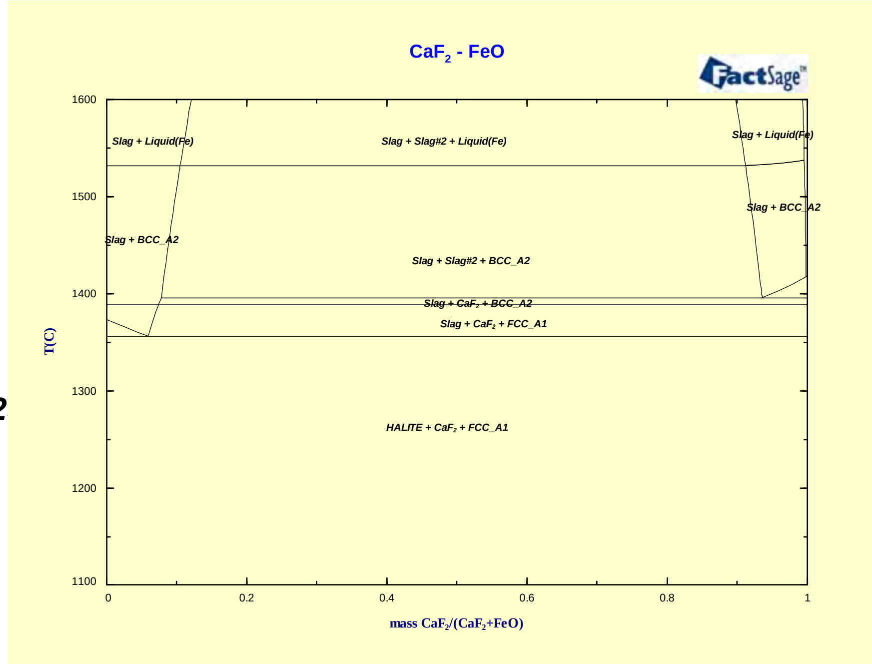
References

[1] Körber, F., W. Oelsen: Stahl Eisen 60 (1940) No. 42, p. 921/9
[2] Oelsen, W., H. Maetz: Mitt. Kaiser Wilhelm Inst. Eisenforsch.,Düsseldorf 23 (1941) No. 12, p. 195/207
[3] Kay, D. A. R., A. Mitchel, M. Ram: J. Iron Steel Inst. 208 (1970) No. 2, p. 141/6
[4] Hawkins, R. J., M. W. Davies: J. Iron Steel Inst. 209 (1971) No. 3, p. 226/30
[5] Mitchel, A.: Can. Metall. Q. 20 (1981) No. 1, p. 101/12



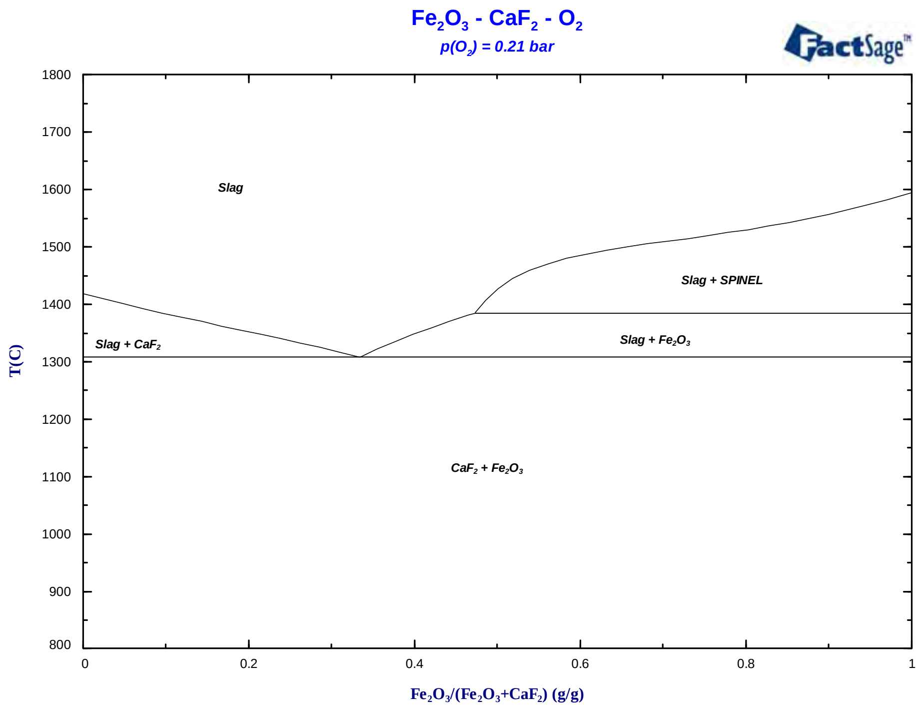
F. Koerber, W. Oelsen, Stahl Eisen, 60[42], (1940), pp.921-92





Predicted CaF_2-Fe_2O_3 phase diagram in air

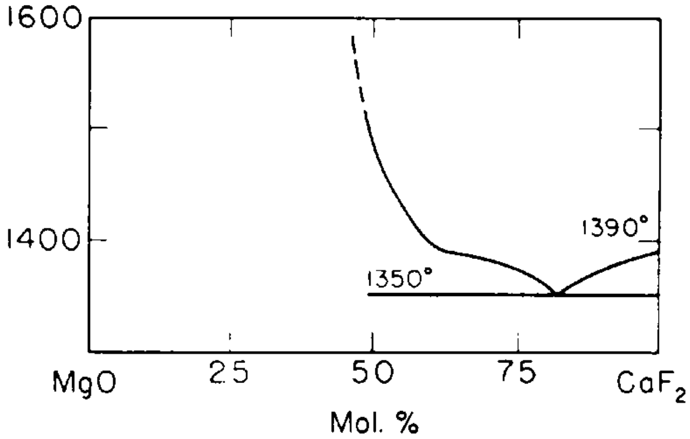
No experimental data are available for this system.



Binary CaF_2-MgO phase diagram

GTT-Technologies

P.P. Budnikov, S.G. Tresvyatskii, Ukr. Khim. Zh. (Russ. Ed.), 19 [5], (1953), pp.552-555..



Slag Atlas, 2nd Ed., Verlag Stahl-Eisen, Düsseldorf, 1995.

CaF_2-MgO

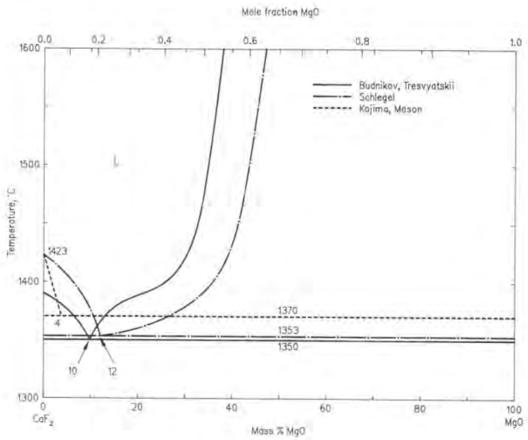
Fig. 3.414. CaF_2-MgO phase diagrams after Budnikov, Tresvyatskii [1], Schlegel [2] and Kojima, Masson [3].

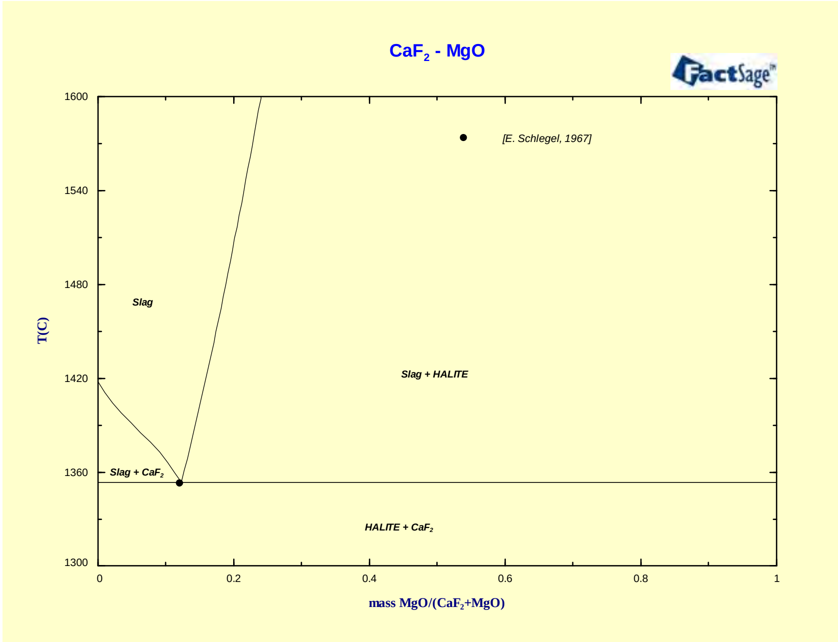
References

[1] Budnikov, P. P., S. G. Tresvyatskii: Ukrain. Khim. Zhur. 19 (1953), p. 552/5

[2] Schlegel, E.: Cercetari Metallurgice Bucureshi 9 (1967), p. 785/91

[3] Kojima, H., C. R. Masson: Canad. J. Chem. 47 (1969), p. 4221/8

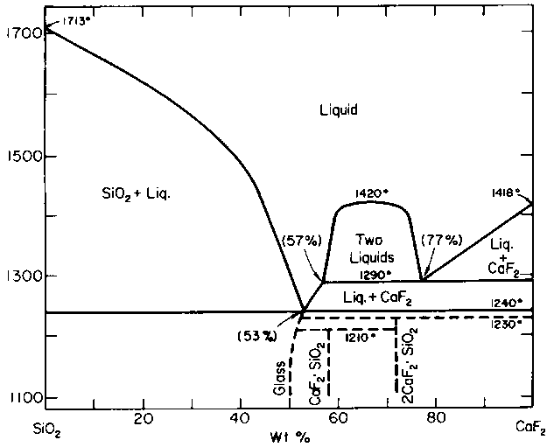




Binary CaF_2-SiO_2 phase diagram

GTT-Technologies

L. Hillert, Acta Chem. Scand., 18 [10], (1964), pp. 2411-2411.



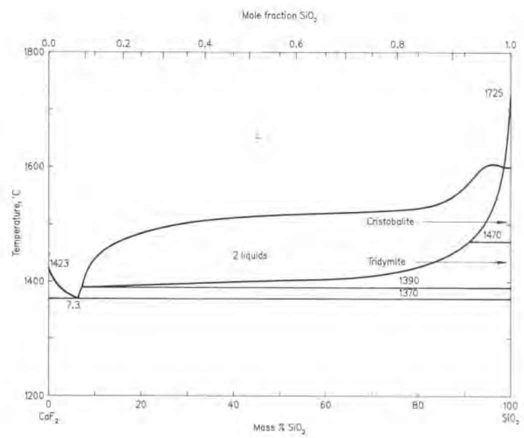
Slag Atlas, 2nd Ed., Verlag Stahl-Eisen, Düsseldorf, 1995.

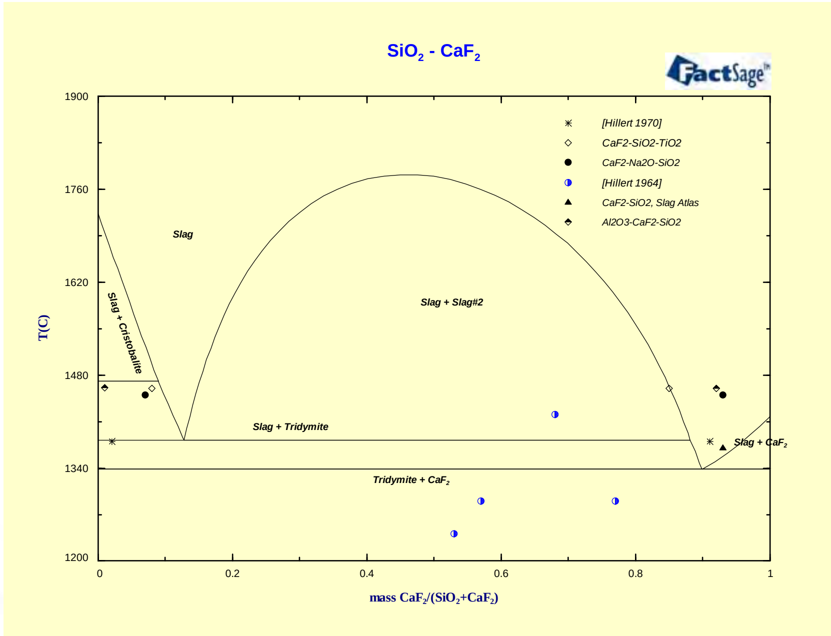
CaF_2-SiO_2

Fig. 3.416. CaF_2-SiO_2 phase diagram after Hillert [1]. The system CaF_2-SiO_2 has also been investigated by Putilin et al. [2], however, the final compositions of the investigated samples belong rather to the ternary system CaF_2-CaO-SiO_2.

References

[1] Hillert, L. H.: Acta Polytech. Scand., Chemistry Including Metallurgy, Series No. 90, (1970)
[2] Putilin, Y. M., A. D. Romanova, A. I. Milov: Ionnye Rasplavy (1976) No. 4, p. 79/83





Isothermal section at 1100°C in Al_2O_3-CaF_2-CaO

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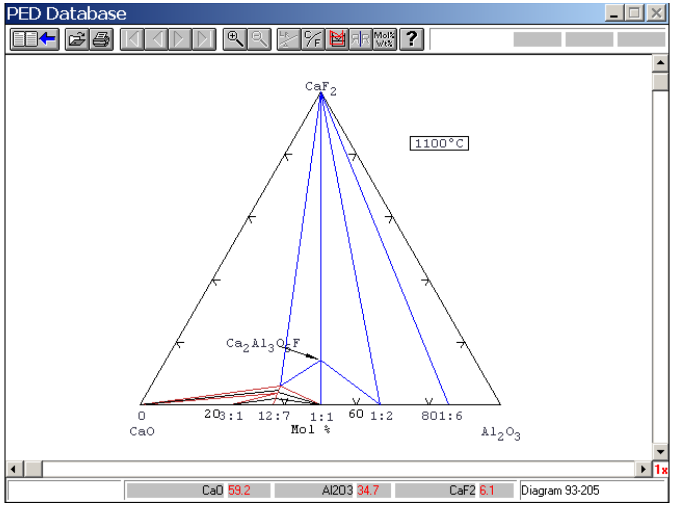
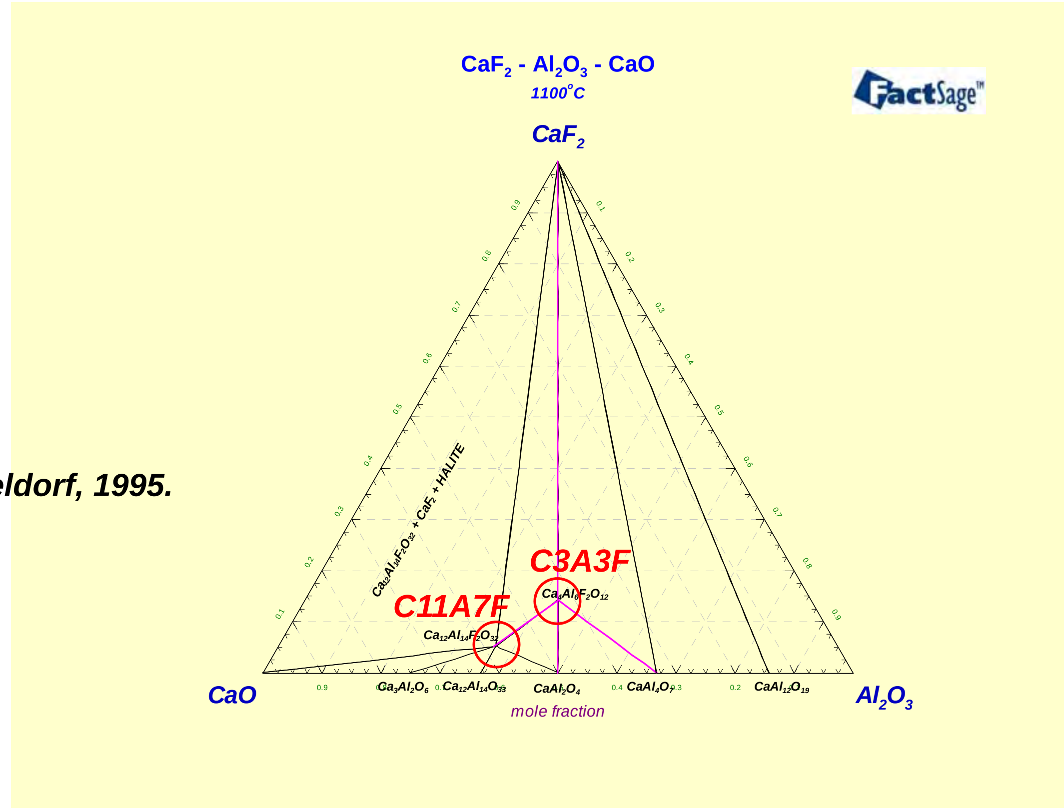
C. Brisi, P. Rolando, Ann. Chim. (Rome), 57 [11], (1967), pp.1304-1315.





Slag Atlas, 2nd Ed., Verlag Stahl-Eisen, Düsseldorf, 1995.

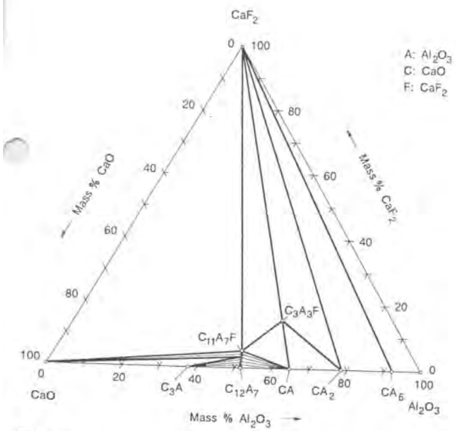
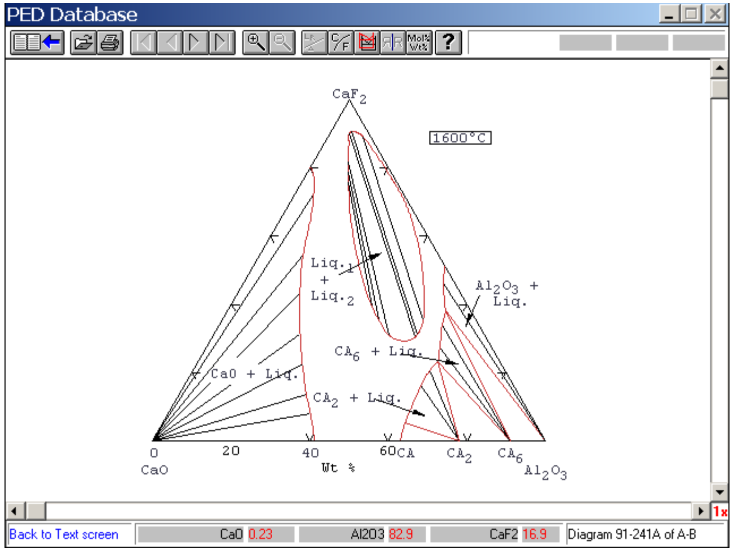


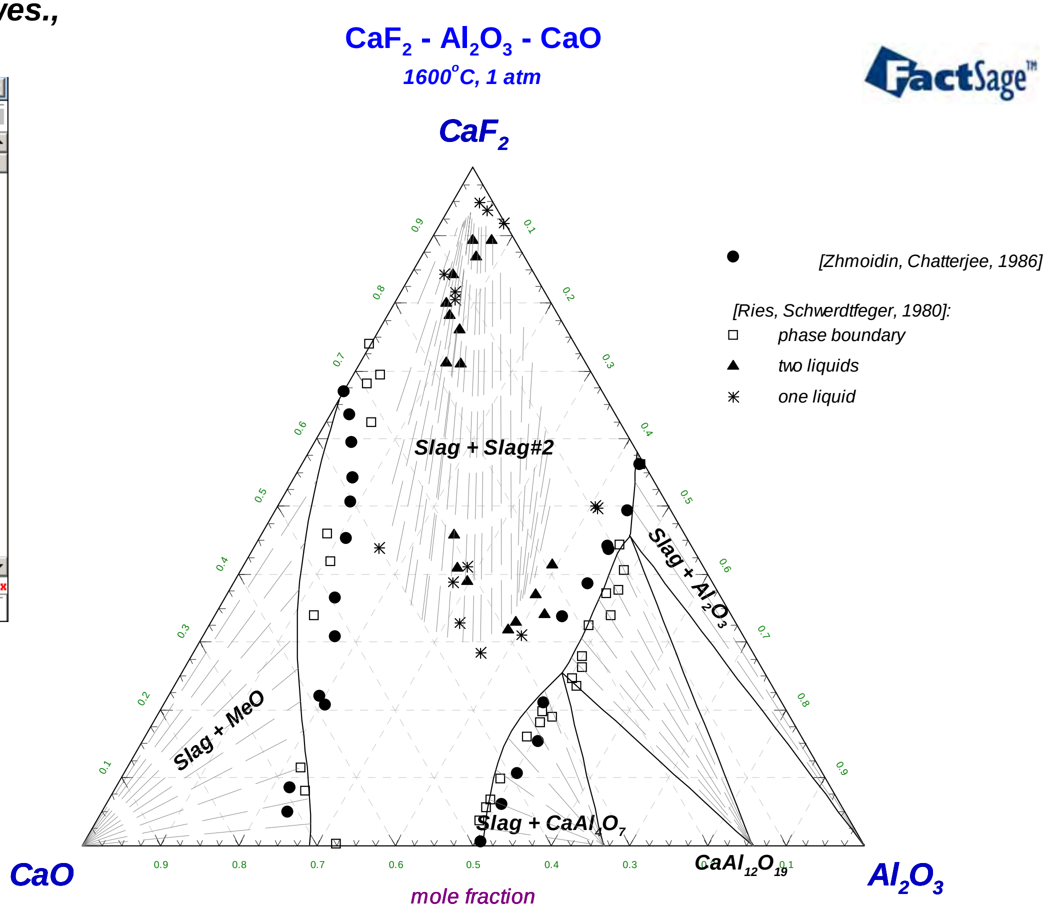
Fig. 3.421. Sub-solidus equilibria in the Al_2O_3-CaF_2-CaO system as determined by Brisi, Rolando [8] and confirmed by Chatterjee, Zhmoidin [1].

Isothermal section at 1600°C in Al_2O_3-CaF_2-CaO

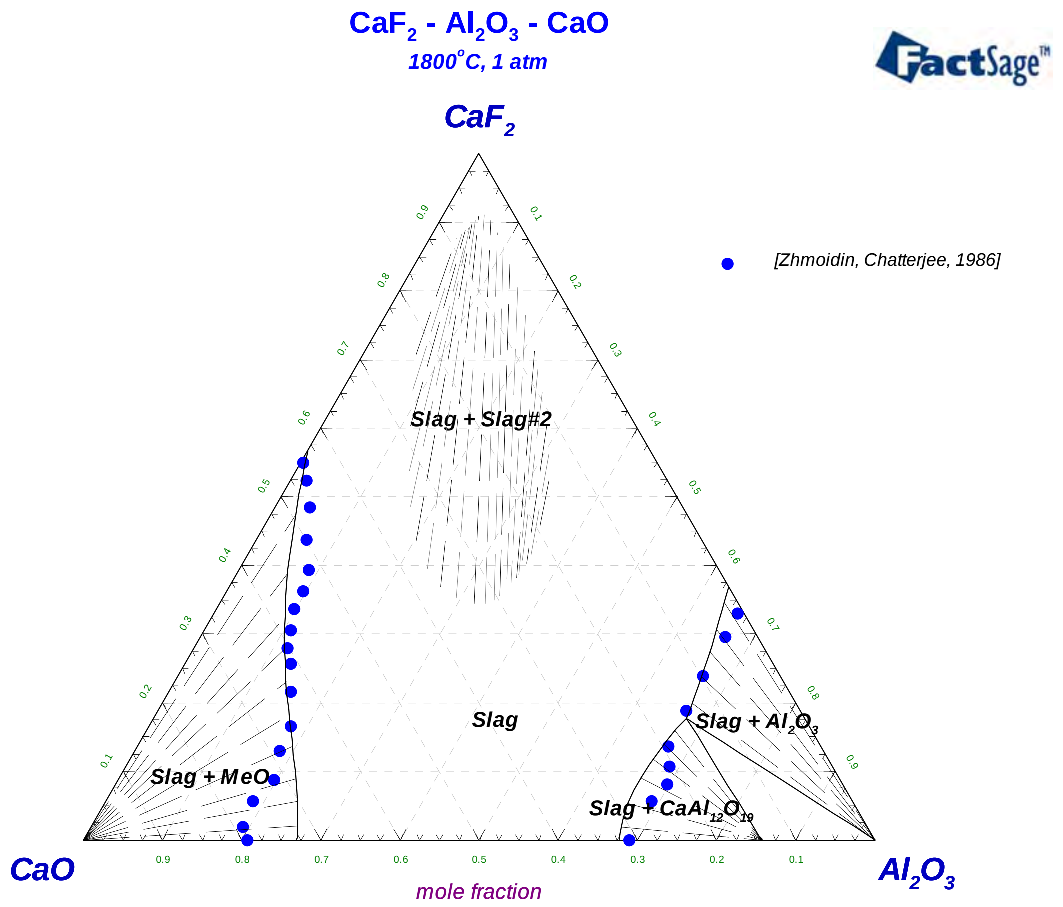
GTT-Technologies

R. Ries, K. Schwerdtfeger, Arch. Eisenhüttenwes., 51, (1980), pp.123-129.



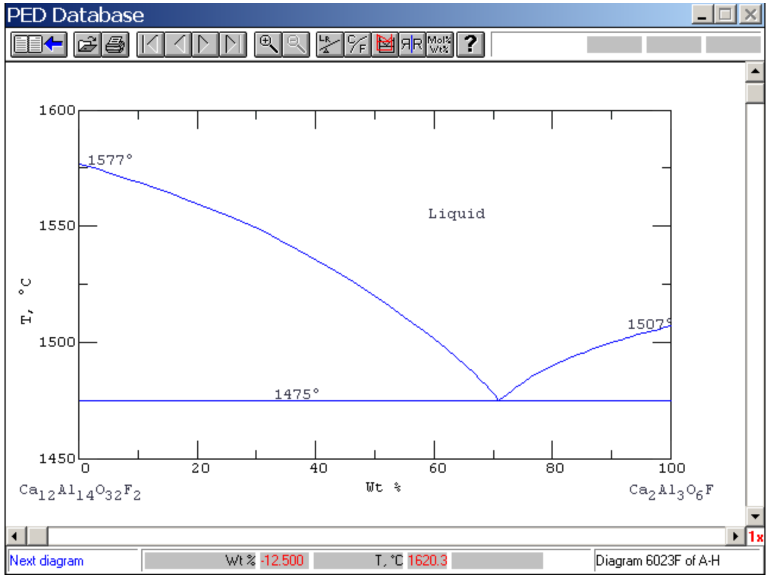


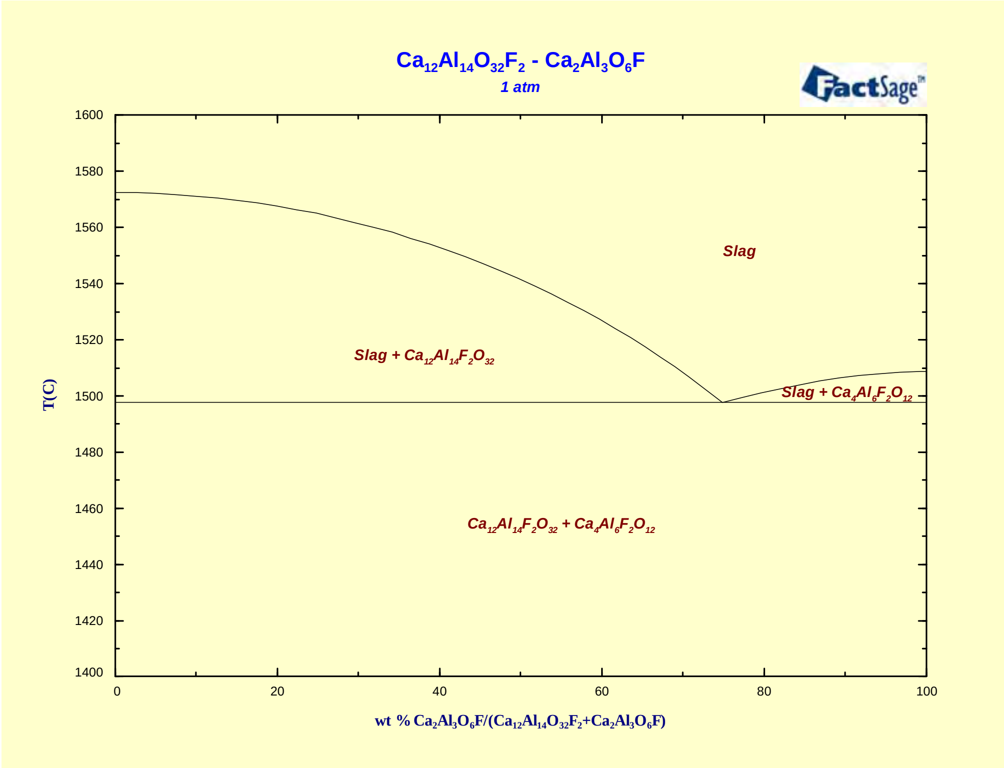
Isothermal section at 1800°C in Al_2O_3-CaF_2-CaO



Isopleth section C11A7F– C3A3F

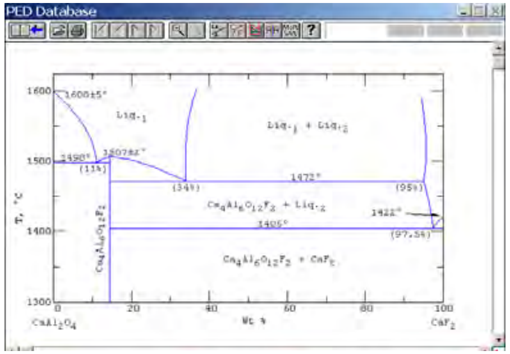
A.K. Chatterjee, G.I. Zhmoidin, Izv. Akad. Nauk SSSR, Neorg. Mater., 8 [5], (1972), pp.886-892.



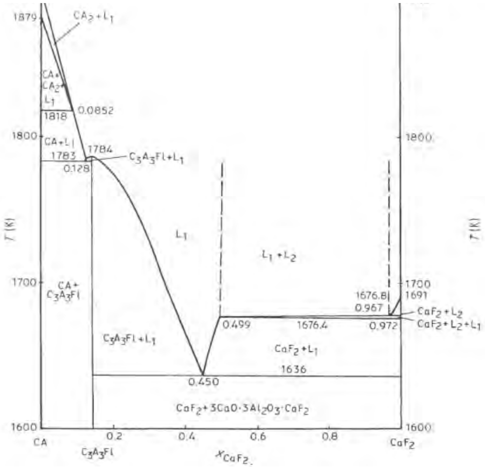


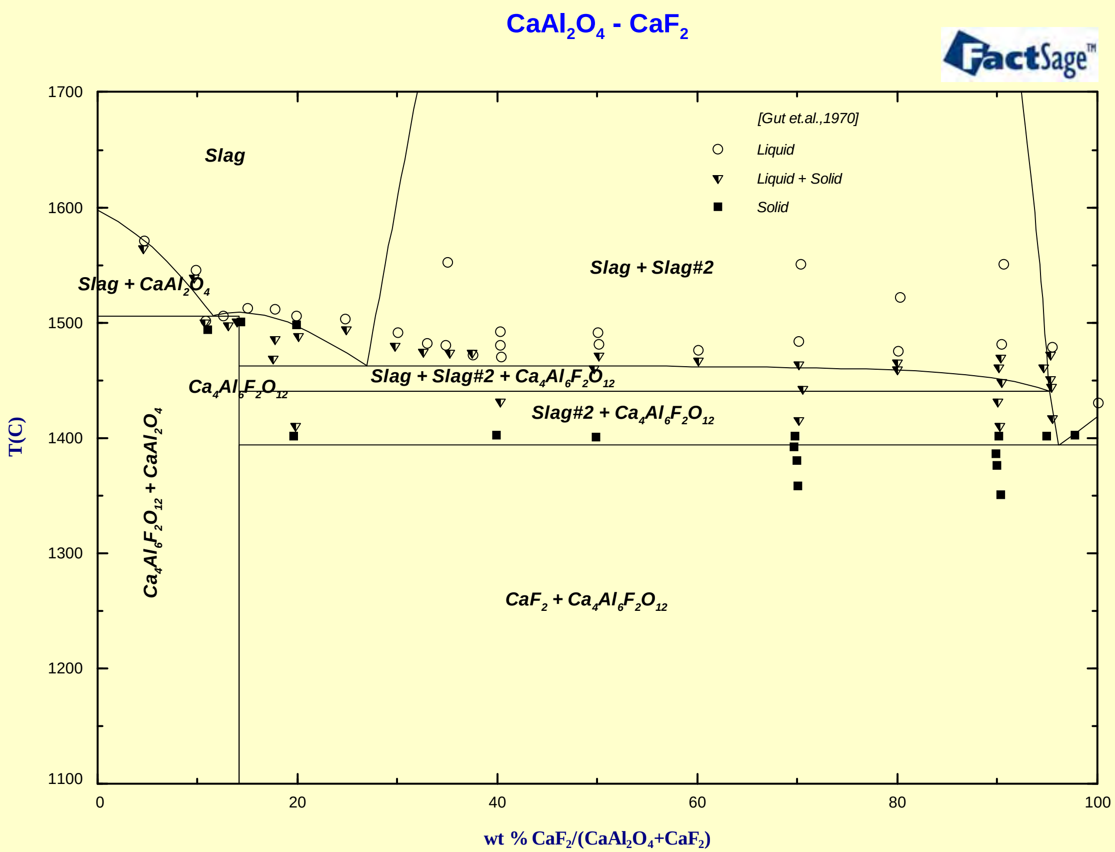
Isopleth section $CaAl_2O_4$ – CaF_2

A.K. Chatterjee, G.I. Zhmoidin, Izv. Akad. Nauk SSSR, Neorg. Mater., 8 [5], (1972), pp.886-892.



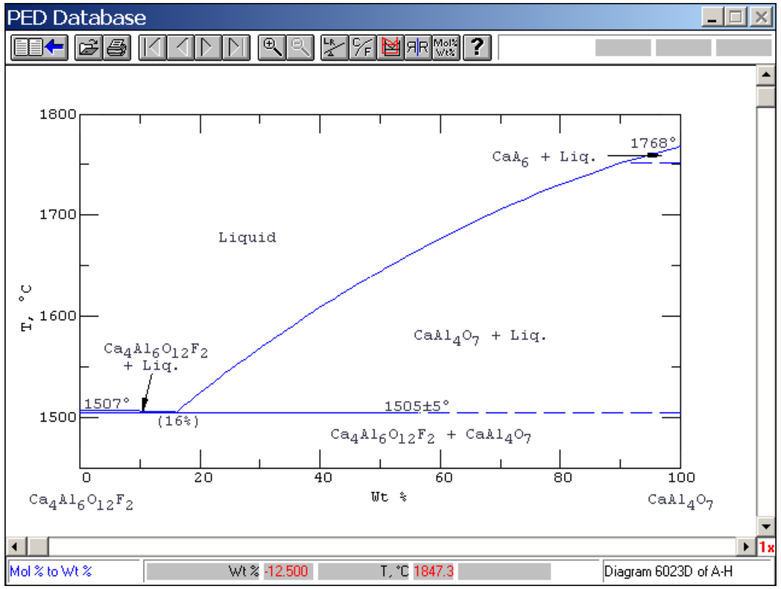
A,I, Zaitsev, N.V. Korolyov, B.M. Mogutno Journ. Mater. Scien., 26 (1991), pp.1588-1

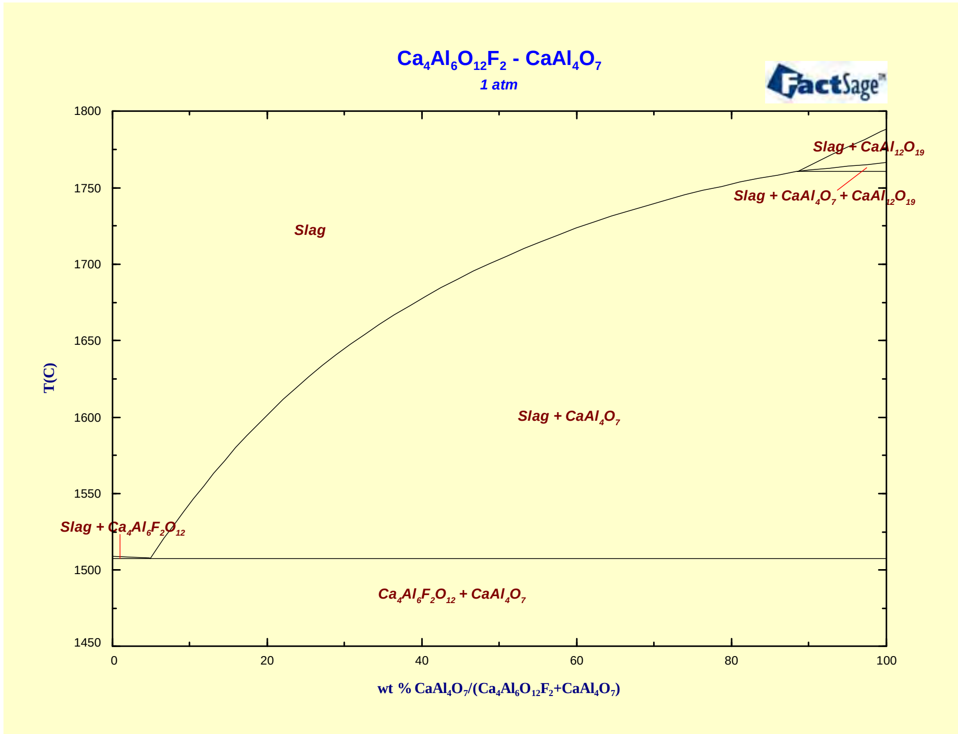




Isopleth section C3A3F – $CaAl_4O_7$

A.K. Chatterjee, G.I. Zhmoidin, Izv. Akad. Nauk SSSR, Neorg. Mater., 8 [5], (1972), pp.886-892.





Liquidus surface in Al_2O_3-CaF_2-CaO

Slag Atlas, 2nd Ed., Verlag Stahl-Eisen, Düsseldorf, 1995.

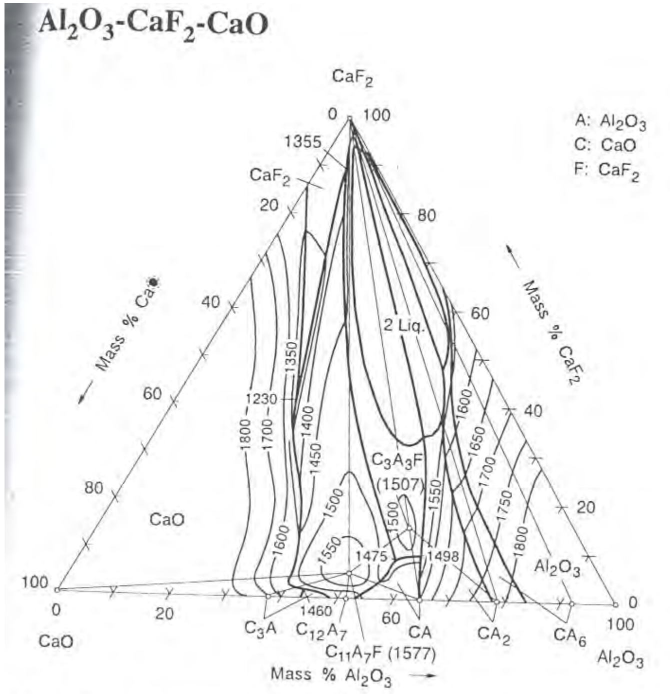
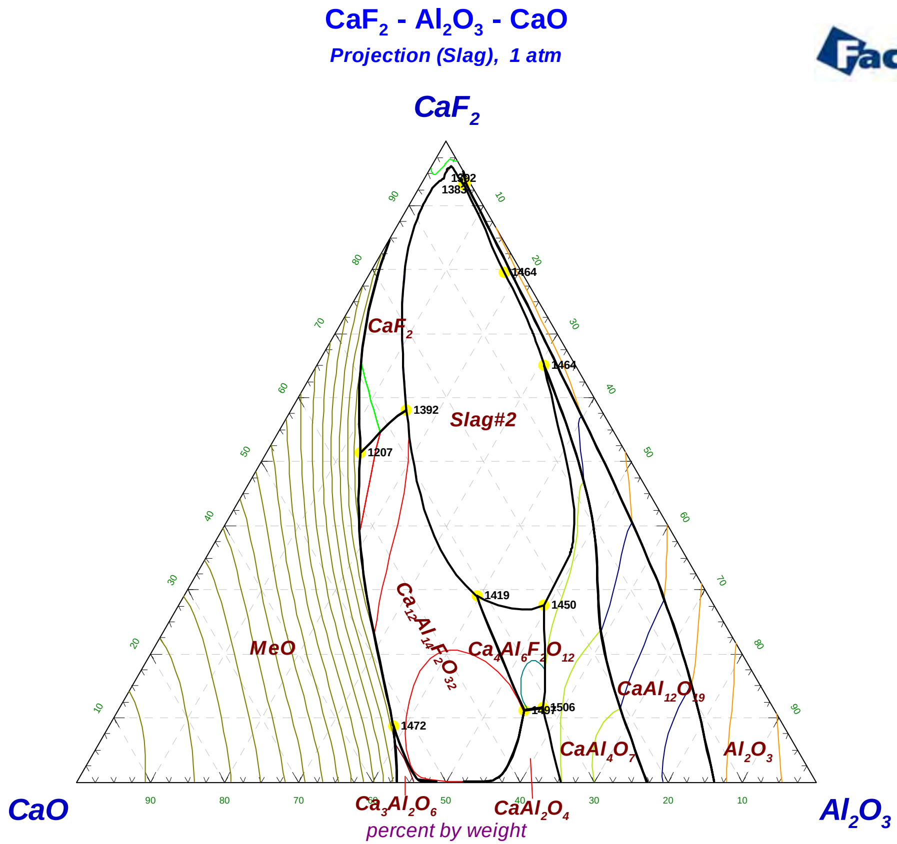
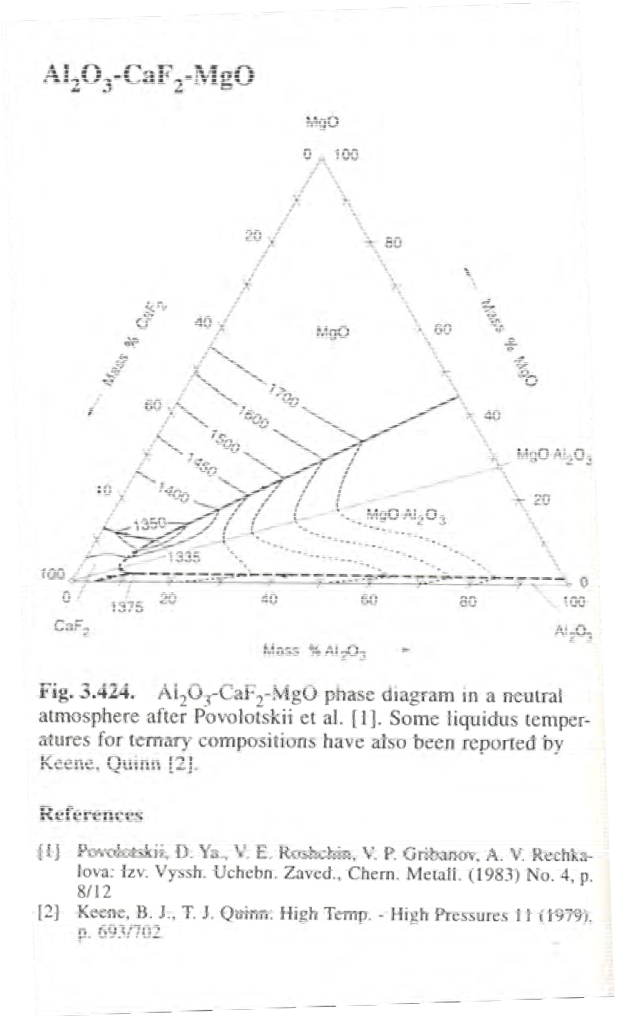


Fig. 3.420. Liquidus surface in the Al_2O_3-CaF_2-CaO system after Chatterjee, Zhmoidin [1] (sealed samples). For liquidus relations in numerous sub-systems, see also Chatterjee, Zhmoidin [2], Smirnov et al. [3] and Zhmoidin, Chatterjee [4].



Liquidus surface in Al_2O_3-CaF_2-MgO

Slag Atlas, 2nd Ed., Verlag Stahl-Eisen, Düsseldorf, 1995.



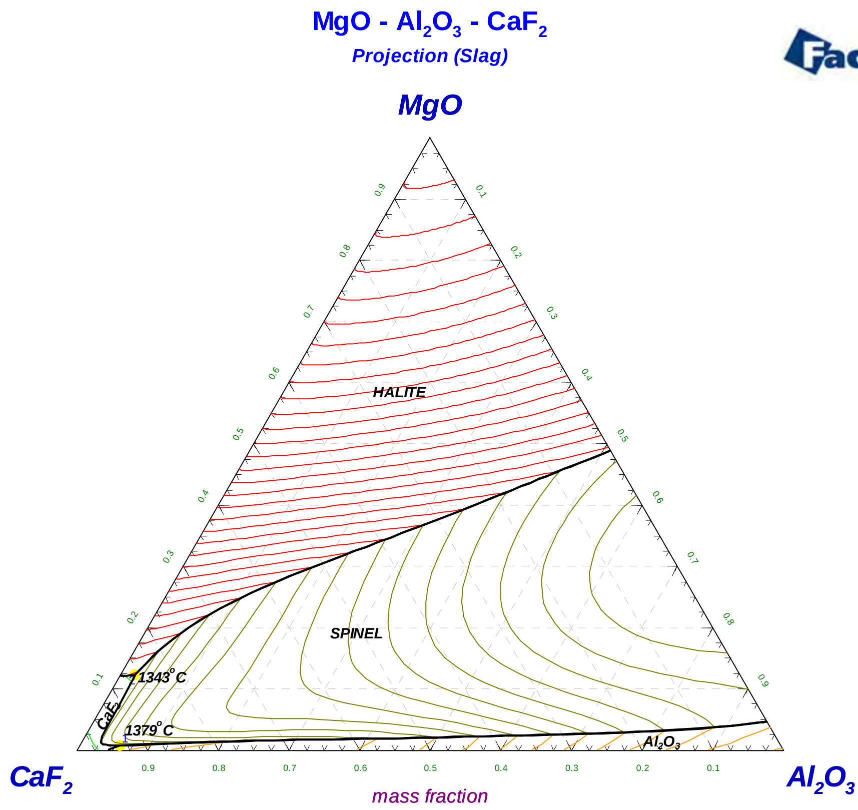
Al_2O_3-CaF_2-MgO

Fig. 3.424. Al_2O_3-CaF_2-MgO phase diagram in a neutral atmosphere after Povolotskii et al. [1]. Some liquidus temperatures for ternary compositions have also been reported by Keene, Quinn [2].

References

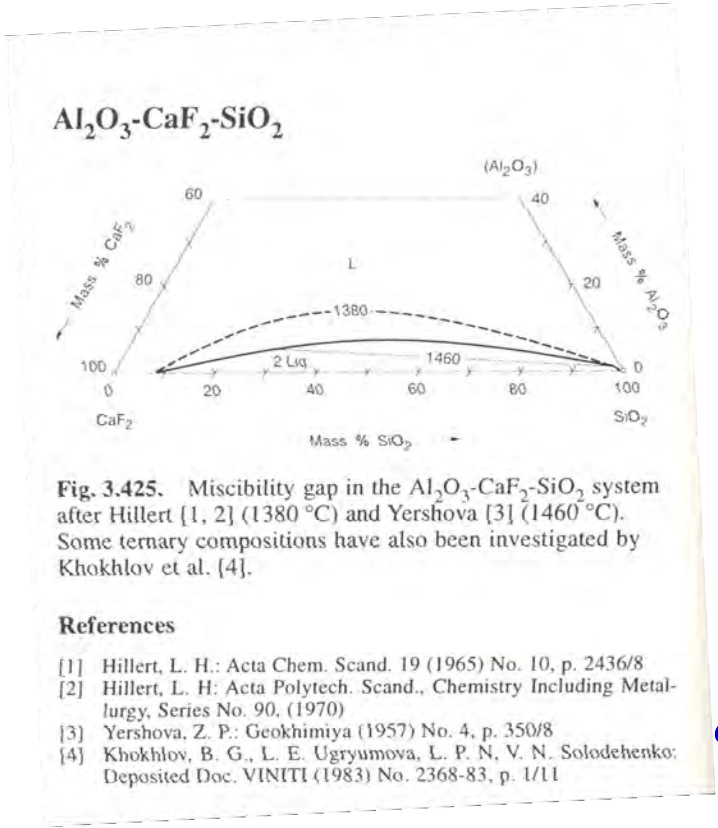
[1] Povolotskii, D. Ya., V. E. Roshchin, V. P. Gribanov, A. V. Rechkalova: Izv. Vyssh. Uchebn. Zaved., Chern. Metall. (1983) No. 4, p. 8/12

[2] Keene, B. J., T. J. Quinn: High Temp. - High Pressures 11 (1979), p. 693/702



Miscibility gaps in Al_2O_3-CaF_2-SiO_2

Slag Atlas, 2nd Ed., Verlag Stahl-Eisen, Düsseldorf, 1995.



Al_2O_3-CaF_2-SiO_2

Fig. 3.425. Miscibility gap in the Al_2O_3-CaF_2-SiO_2 system after Hillert [1, 2] (1380 °C) and Yershova [3] (1460 °C). Some ternary compositions have also been investigated by Khokhlov et al. [4].

References

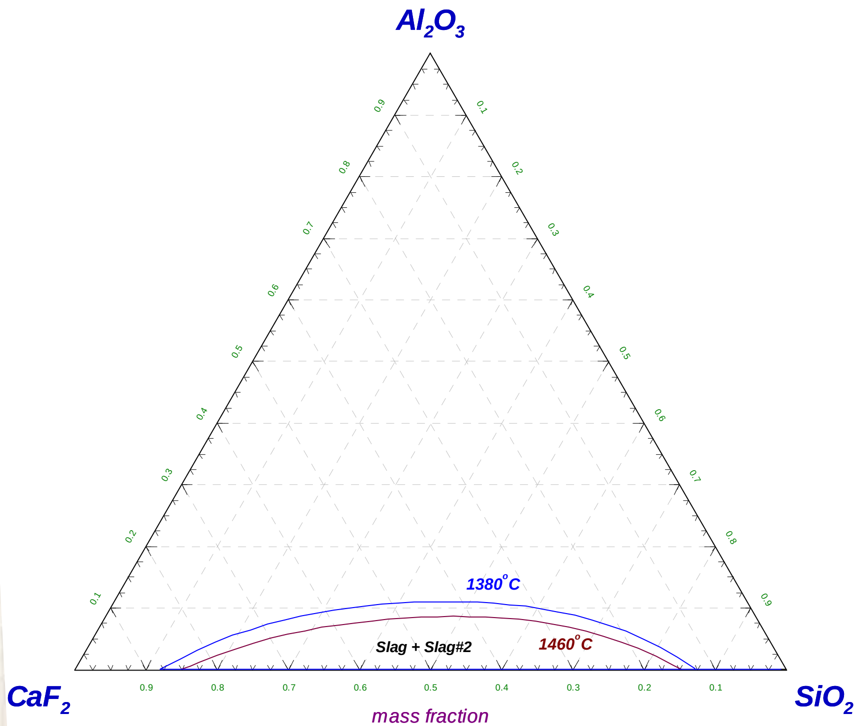
[1] Hillert, L. H.: Acta Chem. Scand. 19 (1965) No. 10, p. 2436/8

[2] Hillert, L. H: Acta Polytech. Scand., Chemistry Including Metallurgy, Series No. 90, (1970)

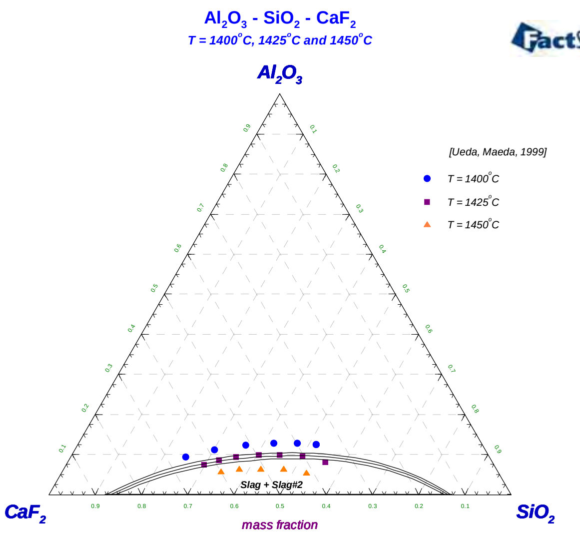
[3] Yershova, Z. P.: Geokhimiya (1957) No. 4, p. 350/8

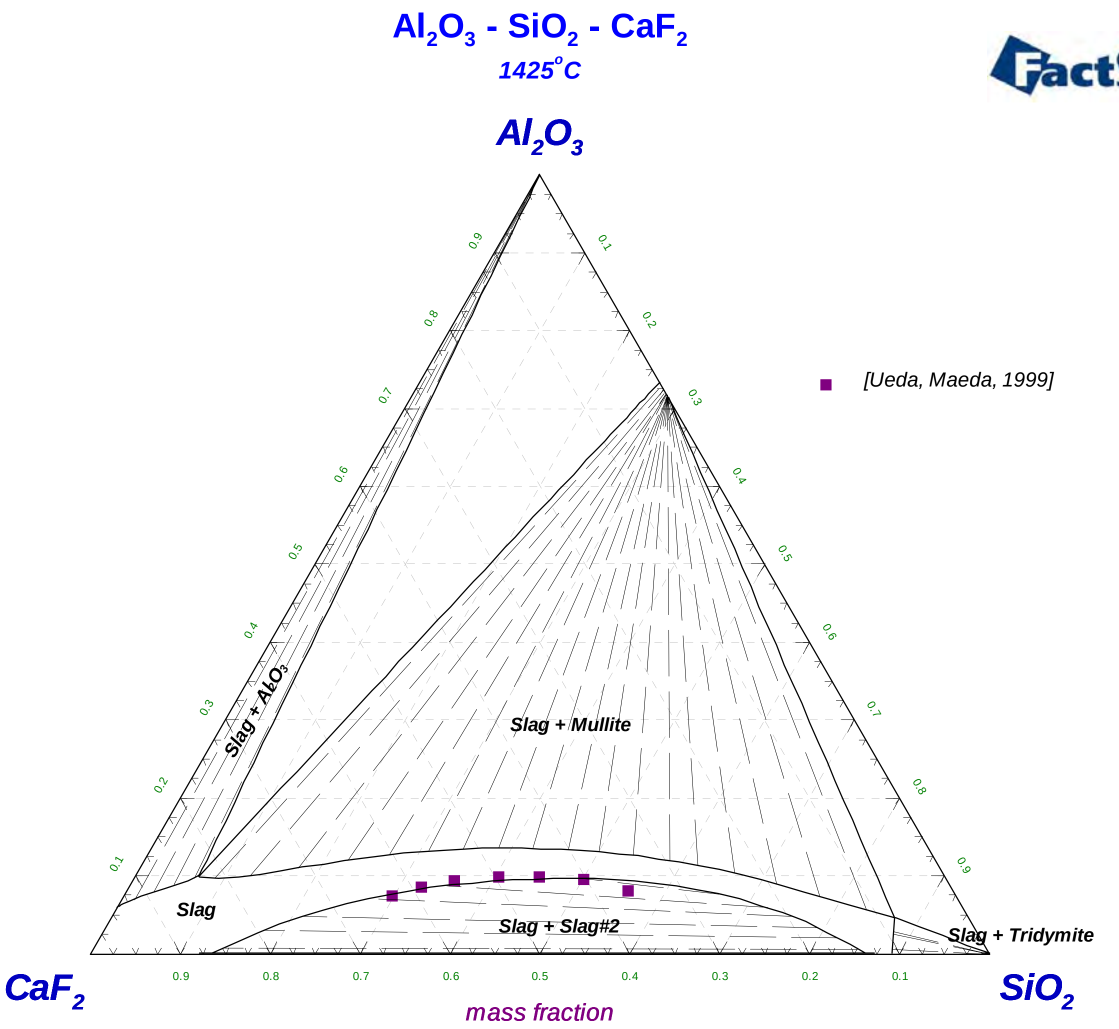
[4] Khokhlov, B. G., L. E. Ugryumova, L. P. N, V. N. Solodehenko: Deposited Doc. VINITI (1983) No. 2368-83, p. 1/11

Al_2O_3 - SiO_2 - CaF_2



Isothermal section at 1425°C in Al_2O_3-CaF_2-SiO_2





Predicted isothermal sections in Al_2O_3-CaF_2-SiO_2

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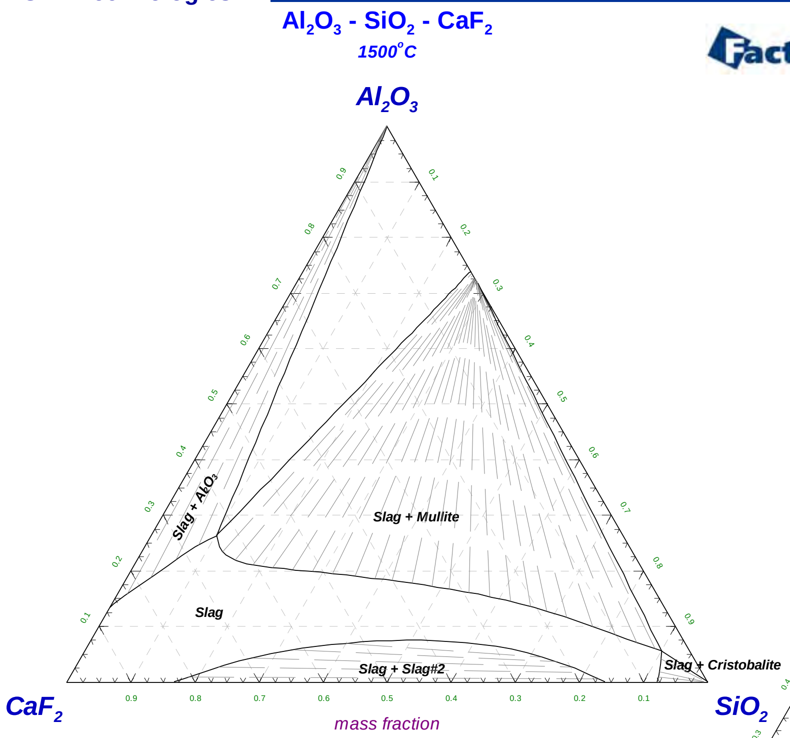
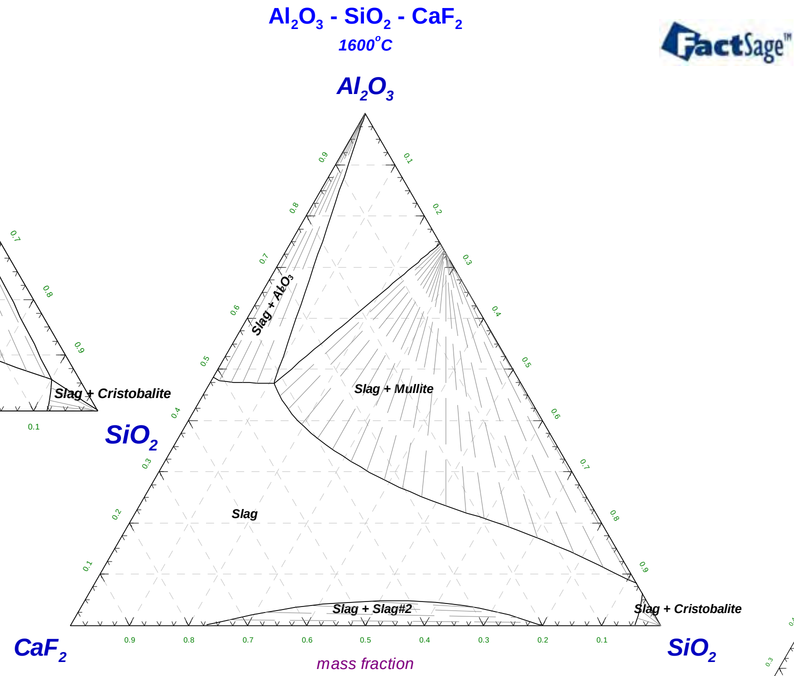
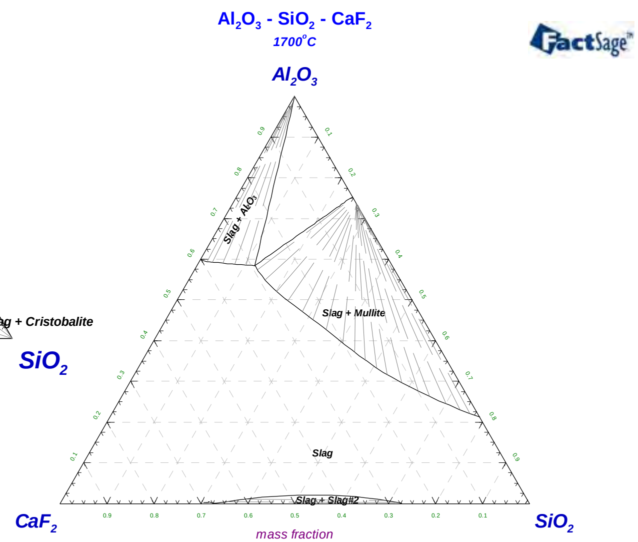






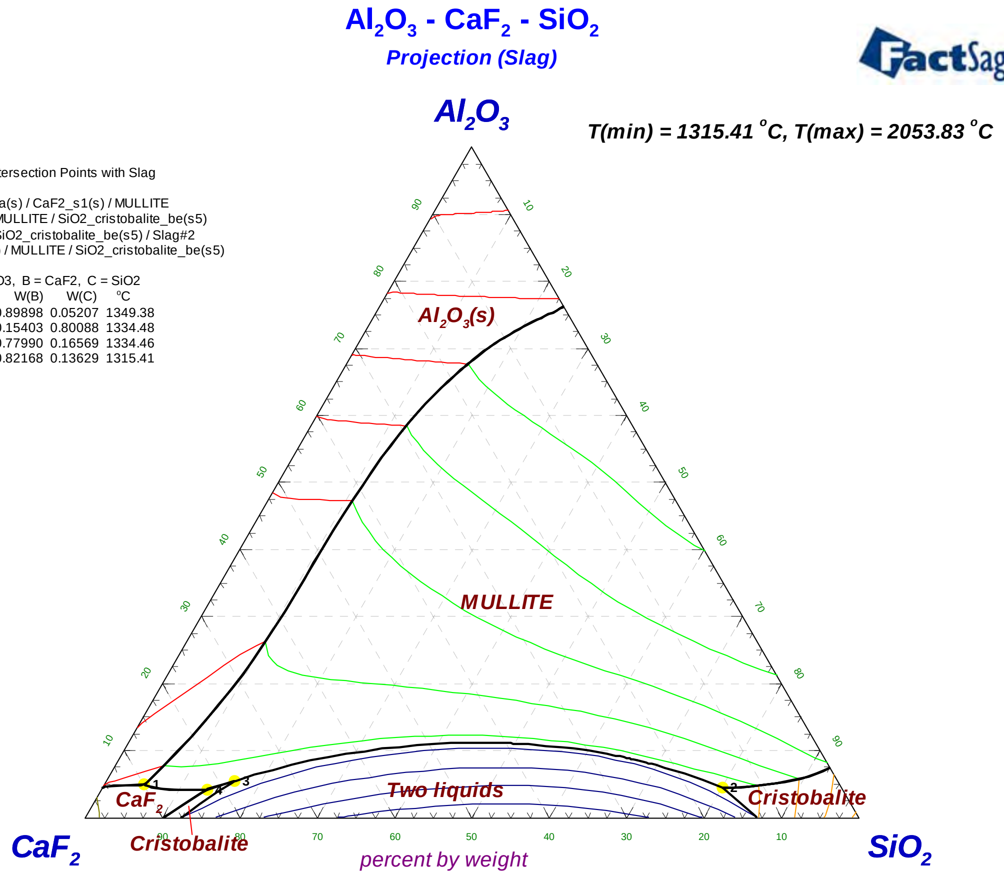
Predicted liquidus surface in Al_2O_3-CaF_2-SiO_2

Four-Phase Intersection Points with Slag

1: Al2O3_alpha(s) / CaF2_s1(s) / MULLITE
2: MULLITE / MULLITE / SiO2_cristobalite_be(s5)
3: MULLITE / SiO2_cristobalite_be(s5) / Slag#2
4: CaF2_s1(s) / MULLITE / SiO2_cristobalite_be(s5)

A = Al2O3, B = CaF2, C = SiO2

	W(A)	W(B)	W(C)	°C
1:	0.04896	0.89898	0.05207	1349.38
2:	0.04509	0.15403	0.80088	1334.48
3:	0.05441	0.77990	0.16569	1334.46
4:	0.04204	0.82168	0.13629	1315.41



Liquidus surface in CaF_2-CaO-MgO

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Slag Atlas, 2nd Ed., Verlag Stahl-Eisen, Düsseldorf, 1995.

CaF_2-CaO-MgO

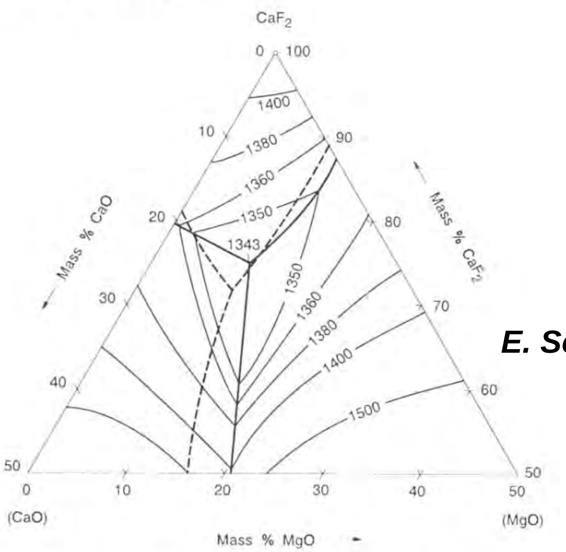
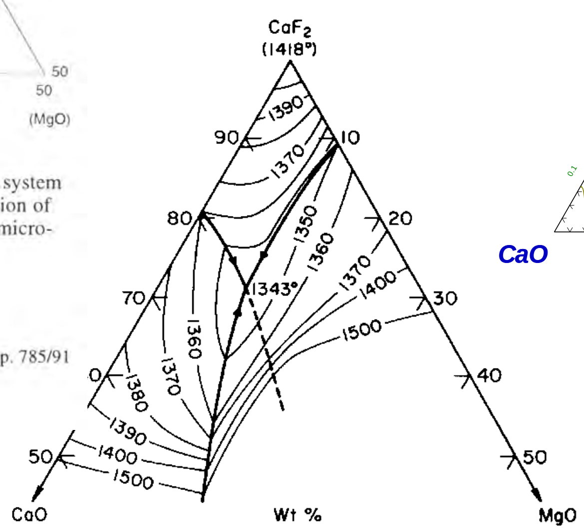


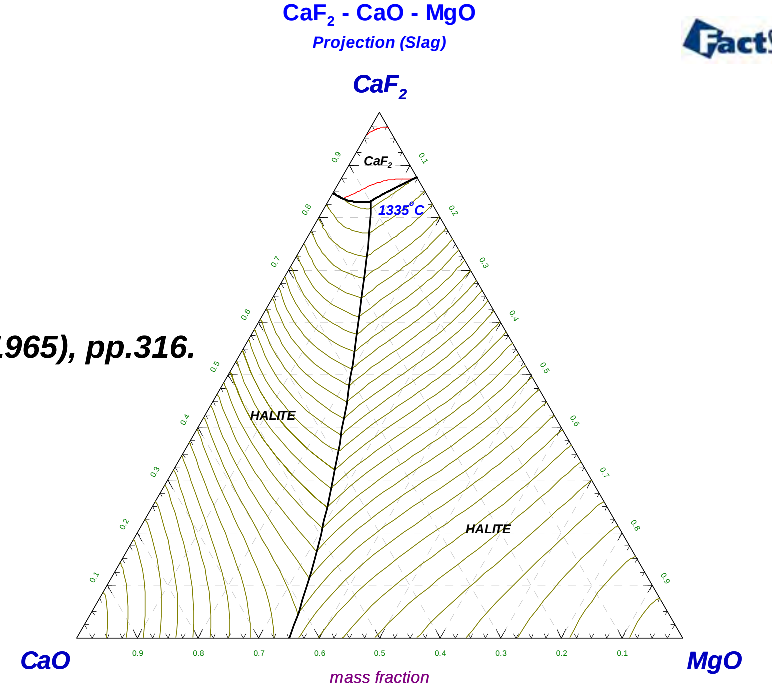
Fig. 3.429. Liquidus surface in the CaF_2-CaO-MgO system after Schlegel [1-3], as determined by DTA. The position of the ternary eutectic, determined by high-temperature microscopy [3], is shown in the figure with dashed lines.

References

[1] Schlegel, E.: Z. Chem. 5 (1965) No. 8, p. 316
[2] Schlegel, E.: Cercetari Metallurgice Bucureshi 9 (1967), p. 785/91
[3] Schlegel, E.: Silikattechnik 20 (1969) No. 3, p. 93/5

E. Schlegel, Z. Chem., 5 [8], (1965), pp.316.



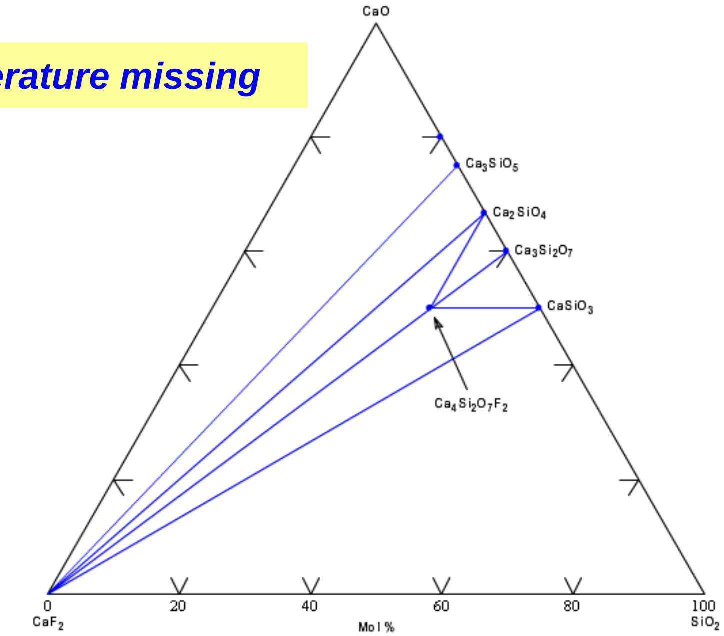


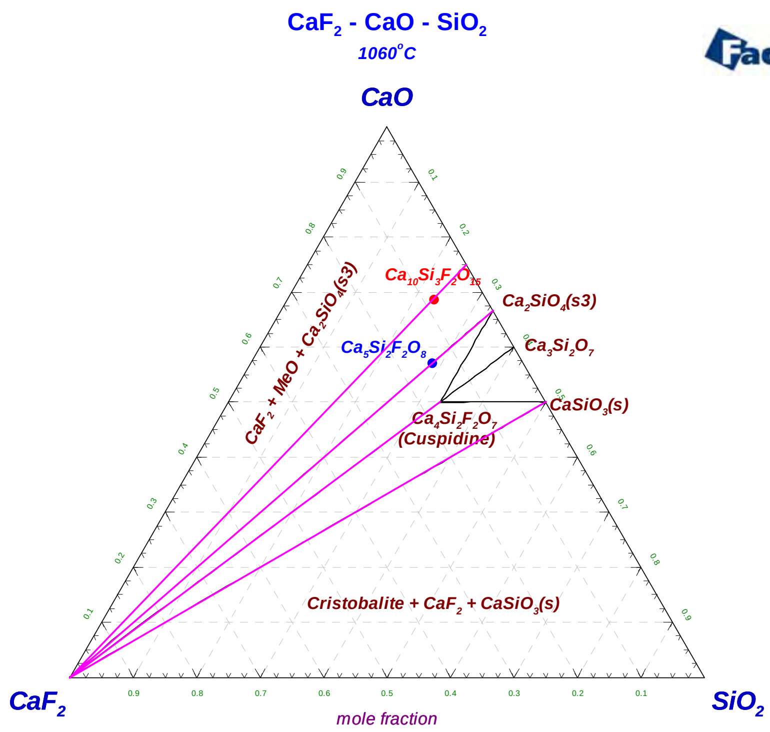
Isothermal section at 1000ºC in CaF_2-CaO-SiO_2

System CaF_2-CaO-SiO_2; compatibility triangles.

*C. Brisi, J. Am. Ceram. Soc., **40** [5] 174-178 (1957).*

Temperature missing

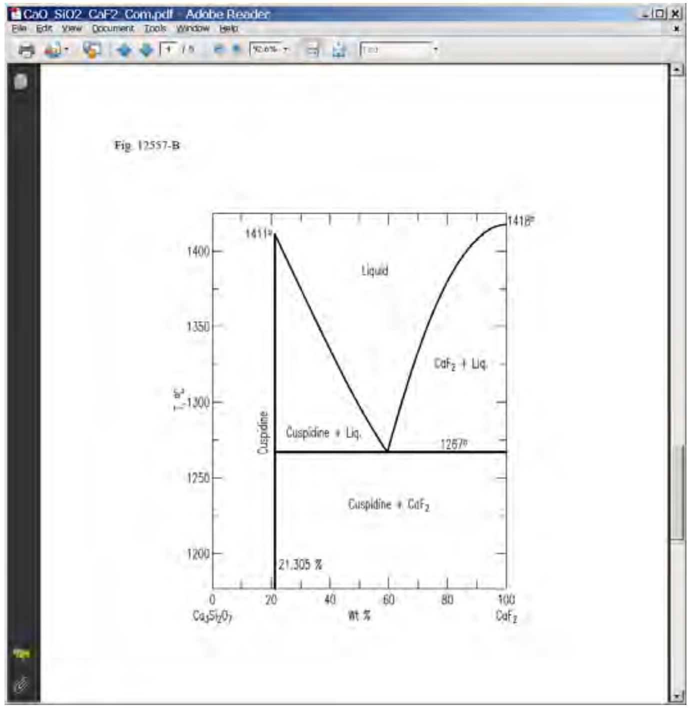


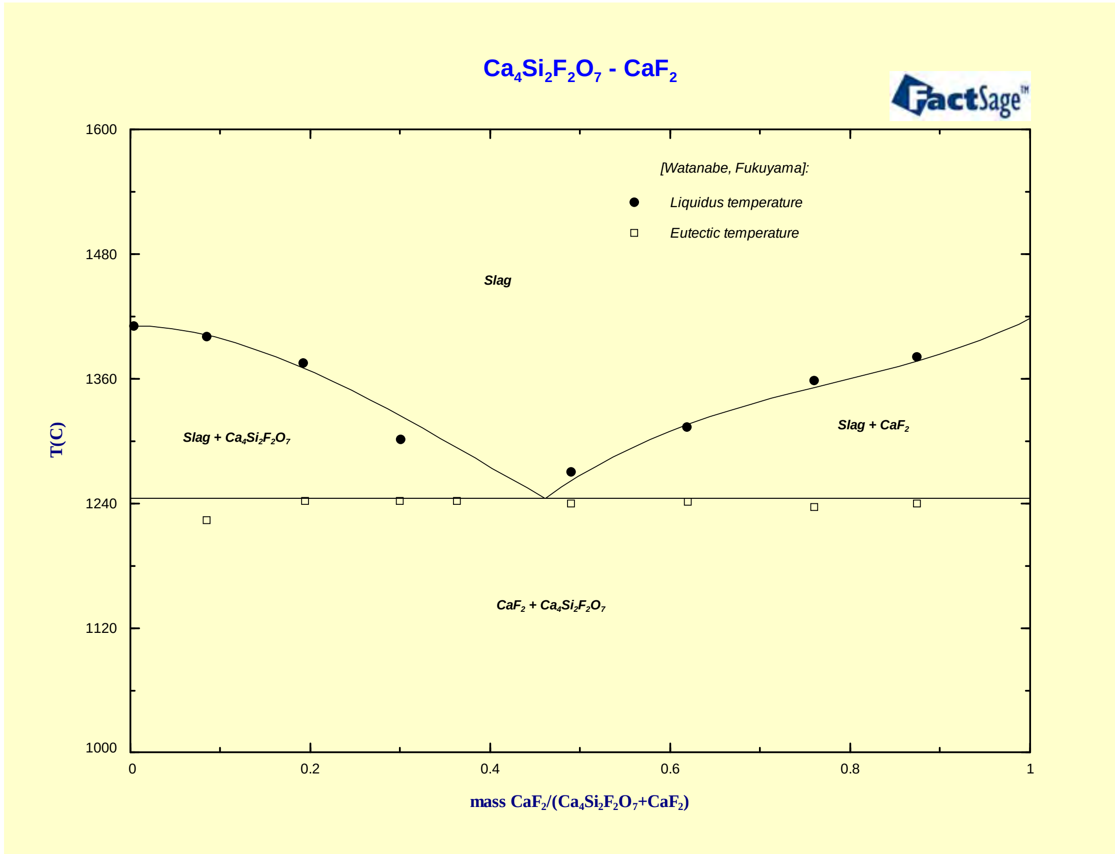


Cuspidine - from the Greek cuspis, for a spear, the characteristic shape of the twinned crystals.

Isopleth section Cuspidine – CaF_2

(B) *pseudobinary phase diagram for cuspidine-CaF2 system; Cuspidine = Ca4Si2O7F2*
H. Fukuyama, T. Watanabe, M. Susa, and K. Nagata, "Pseudo-binary Phase Diagram of the Cuspidine - CaF2 System - Relating to Mold Flux for Continuous Casting of Steel -"; pp. 61-75 in EPD Congr. 1999, Proc. Sess. Symp., TMS Annual Meeting, San Diego, California, February 28-March 4, 1999. Edited by B. Mishra, Minerals, Metals & Materials Society, Warrendale, Pennsylvania, 1999.



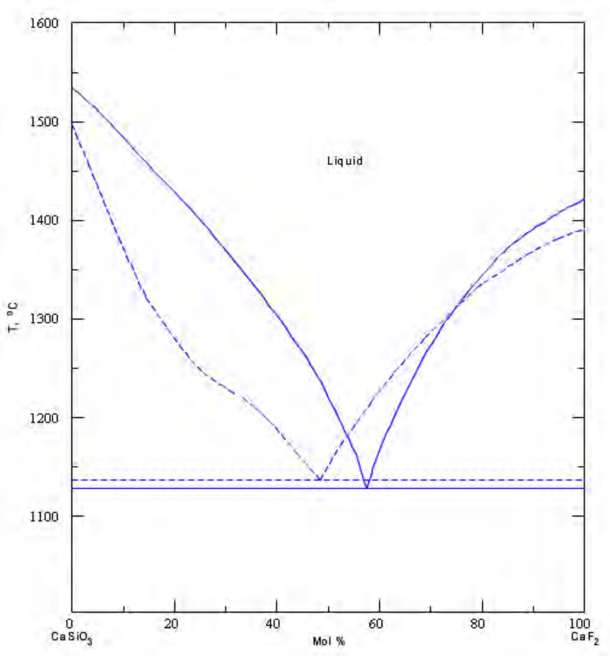


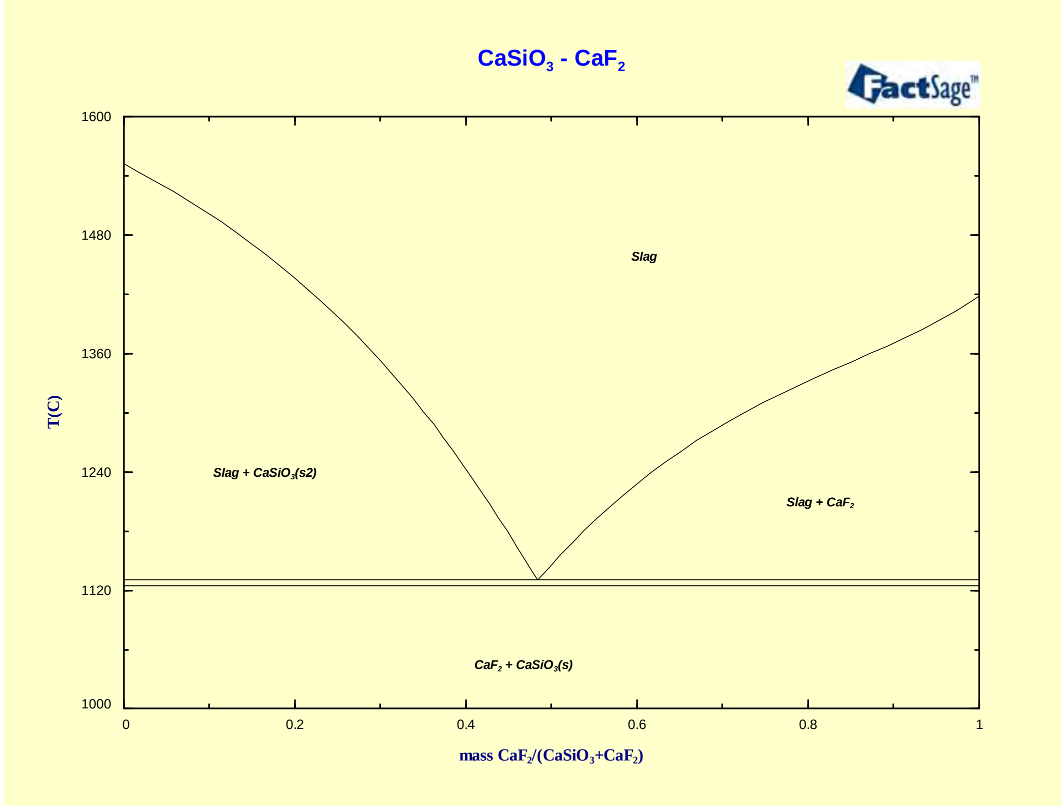
Isopleth section $CaSiO_3$ – CaF_2

GTT-Technologies

T. Baak and A. Oelander, Acta Chem. Scand., ***9*** *[8] 1350-1354 (1955).*

Dashed lines after Karandeef.
B. Karandeeff, Z. Anorg. Chem., ***68*** *[3] 188-197 (1910)*





Isopleth section Ca_2SiO_4 – CaF_2

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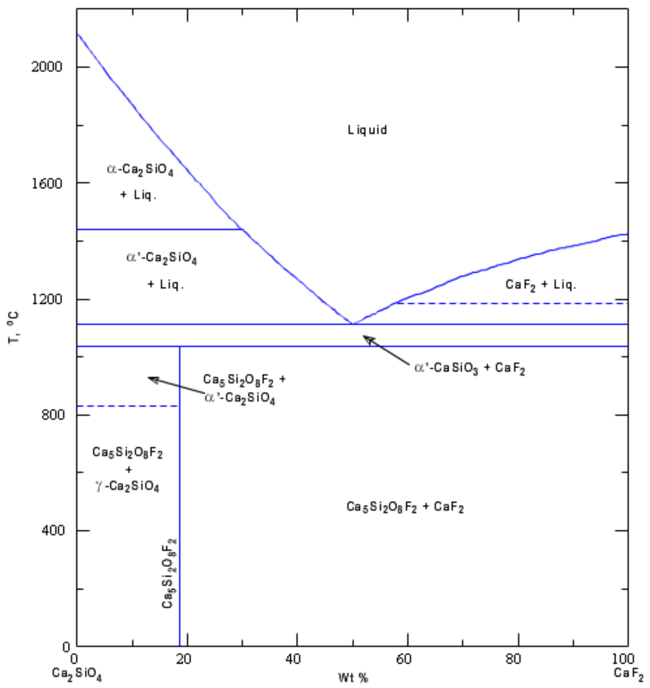
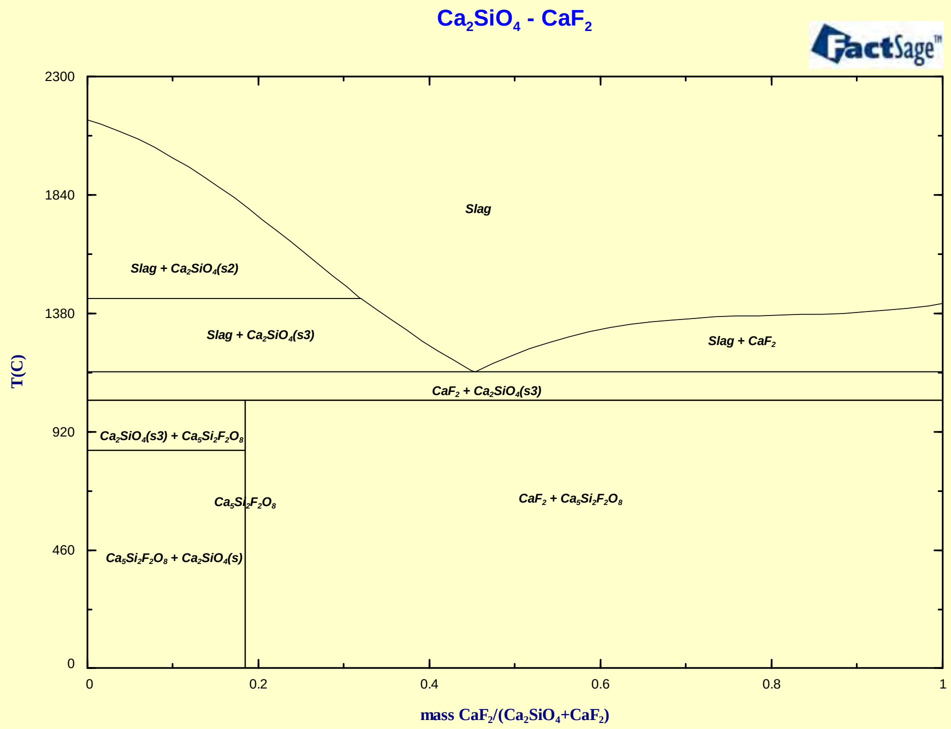
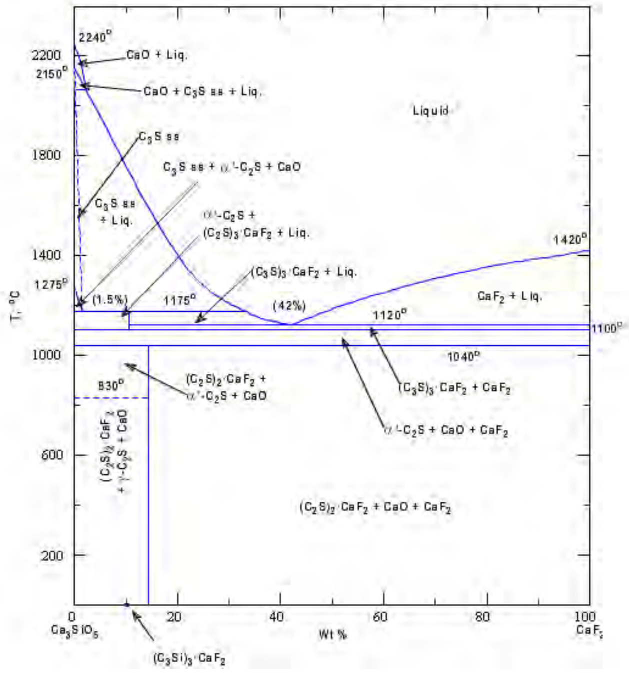
W. H. Gutt and G. J. Osborne, Trans. Br. Ceram. Soc., ***65*** *[9] 521-534 (1966).*

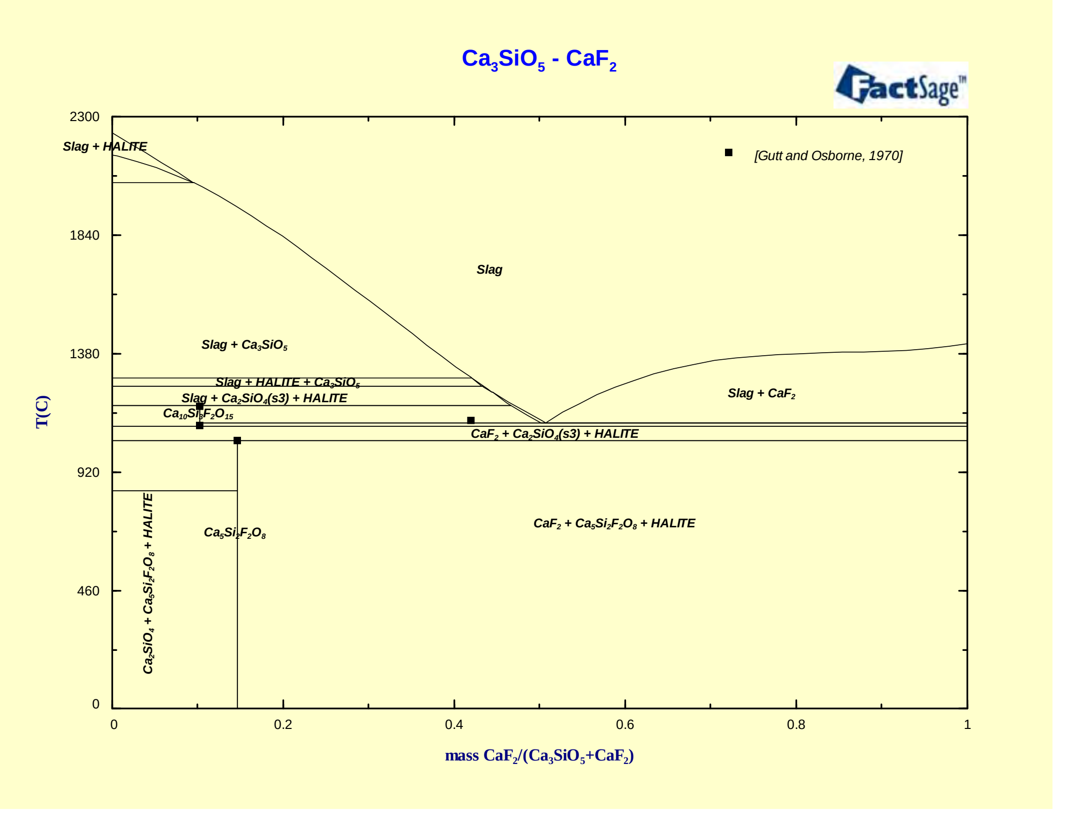




Isopleth section Ca_3SiO_5 – CaF_2

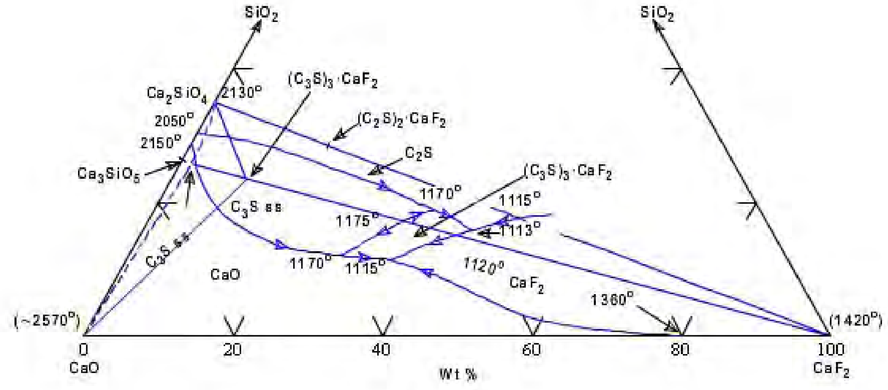
*W. H. Gutt and G. J. Osborne, Trans. Br. Ceram. Soc., **69** [3] 125-129 (1970).*

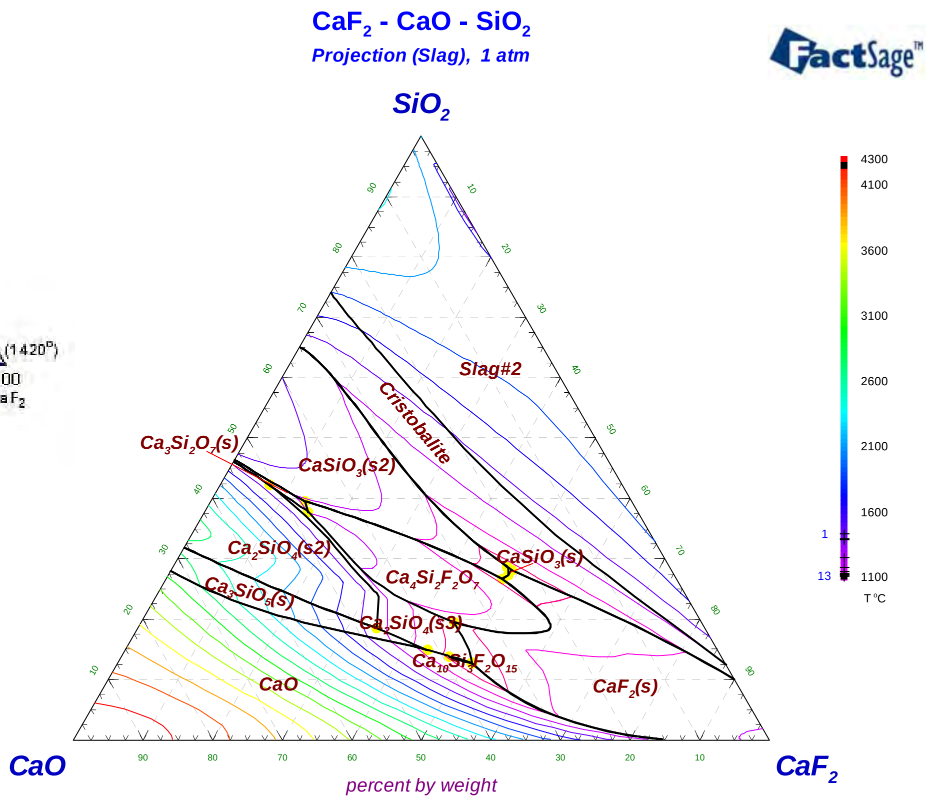




Liquidus surface in CaF_2-CaO-SiO_2

W. H. Gutt and G. J. Osborne, Trans. Br. Ceram. Soc., ***69*** *[3] 125-129 (1970).*





Addition of P_2O_5

- The Fe-P binary system
- The Al_2O_3-P_2O_5 system
- The CaO-P_2O_5 system
- The Cr_2O_3-P_2O_5 phase diagram in air
- The Fe_2O_3-P_2O_5 phase diagram in air
- The FeO–P_2O_5 phase diagram in equilibrium with Fe
- The MgO-P_2O_5 system

- The ternary FeO-Fe_2O_3-P_2O_5 system

Addition of P_2O_5

The associate species containing P were added in order to describe the liquid phase in the Al_2O_3-CaO-Cr_2O_3-FeO-Fe_2O_3-MgO system containing P_2O_5.

<table>
<tr><th>System</th><th>Associate species</th><th>Description
$MeO_x : P_2O_5$</th></tr>
<tr><td>Al_2O_3-P_2O_5</td><td>$AlPO_4$(SGPS)- Berlinite</td><td rowspan="3">1:1</td></tr>
<tr><td>$Cr_2O_3 – P_2O_5$</td><td>$CrPO_4$</td></tr>
<tr><td>$Fe_2O_3 – P_2O_5$</td><td>$FePO_4$(SGPS)</td></tr>
<tr><td>CaO - P_2O_5</td><td>$Ca_3P_2O_8$, $Ca_2P_2O_7$, CaP_2O_8 [Serena 2011]</td><td rowspan="3">3:1, 2:1, 1:1</td></tr>
<tr><td>FeO - P_2O_5</td><td>$Fe_3P_2O_8$, $Fe_2P_2O_7$, FeP_2O_6,</td></tr>
<tr><td>MgO - P_2O_5</td><td>$Mg_3P_2O_8$ (SGPS), $Mg_2P_2O_7$, MgP_2O_8</td></tr>
</table>

Modelling of binary P-containing phases

System	*Phase*	*Description*	*Used data*
Fe-P	fcc-A1 bcc-A2 FeP Fe2P Fe3P	(<u>Fe</u>, O, P)$_1$ (Va)$_1$ (<u>Fe</u>, O, P)$_1$ (Va)$_3$ stoichiometric stoichiometric stoichiometric	[99Lee] [99Lee] [99Lee] [99Lee] [99Lee]
Al_2O_3-P_2O_5	$3Al_2O_3 \cdot P_2O_5$ $AlPO_4$(s3) Berlinite $AlPO_4$(s2) $AlPO_4$(s1) $Al_2O_3 \cdot 3P_2O_5$	stoichiometric stoichiometric stoichiometric stoichiometric stoichiometric	- SGPS SGPS SGPS -
CaO-P_2O_5	$CaO \cdot 2P_2O_5$ $2CaO \cdot 3P_2O_5$ $CaO \cdot P_2O_5$ $2CaO \cdot P_2O_5$(s1,s2,s3) $3CaO \cdot P_2O_5$(s1,s2,s3) $4CaO \cdot P_2O_5$	stoichiometric stoichiometric stoichiometric stoichiometric stoichiometric stoichiometric	[Serena 2011] [Serena 2011] [Serena 2011] [Serena 2011] revised (T_{tr}) [Serena 2011] revised (T_{tr}) [Serena 2011]

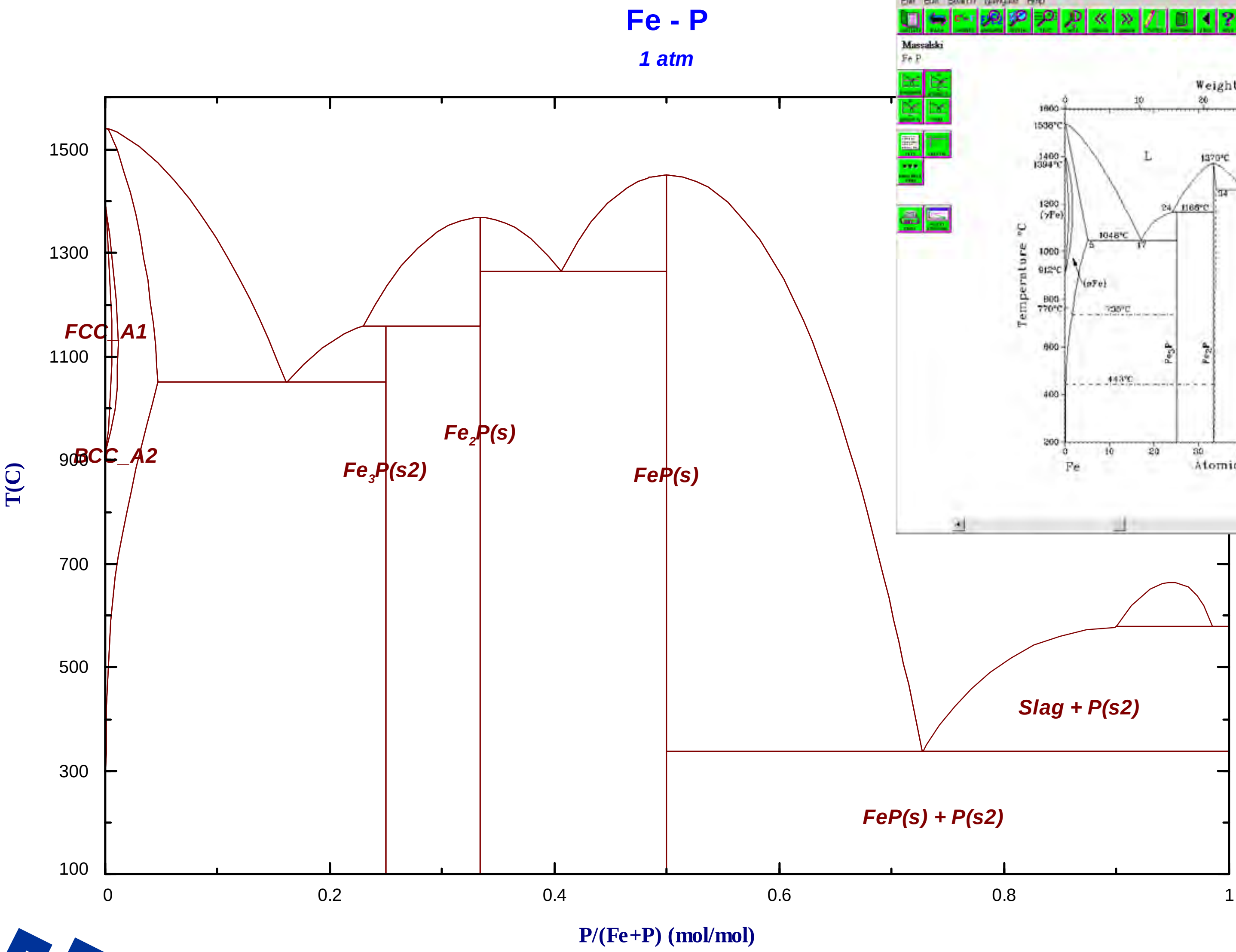
Modelling of binary P-containing phases

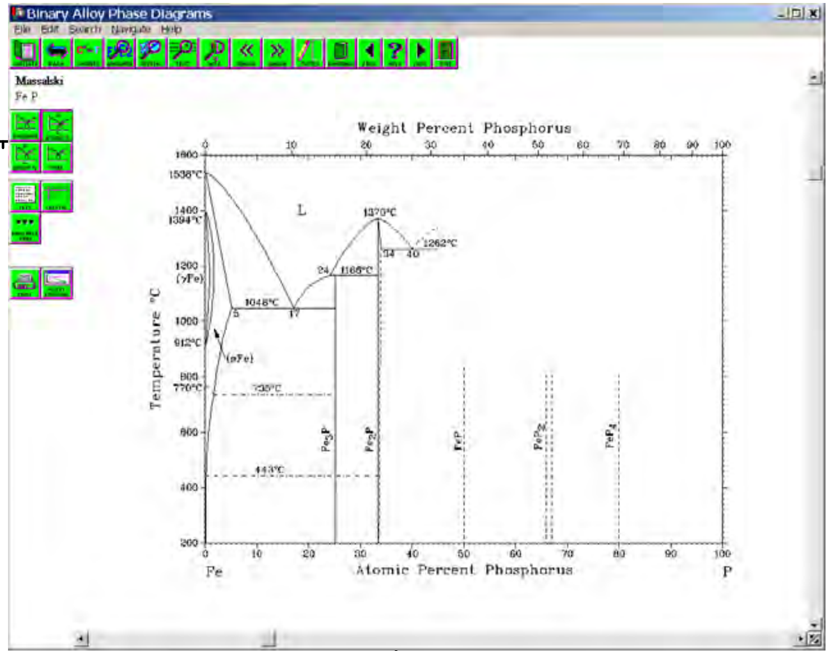
System	*Phase*	*Description*	*Used data*
Cr_2O_3-P_2O_5	$CrPO_4$ $5Cr_2O_3 \cdot P_2O_5$ $3Cr_2O_3 \cdot P_2O_5$	stoichiometric stoichiometric stoichiometric	
FeO-P_2O_5	$FeO \cdot P_2O_5$ $2FeO \cdot P_2O_5$ $3FeO \cdot P_2O_5$	stoichiometric stoichiometric stoichiometric	- - -
Fe_2O_3-P_2O_5	$Fe_2O_3 \cdot 3P_2O_5$ $2Fe_2O_3 \cdot 3P_2O_5$ $Fe_2O_3 \cdot P_2O_5$ $3Fe_2O_3 \cdot P_2O_5$	stoichiometric stoichiometric stoichiometric stoichiometric	- - SGPS changed -
MgO-P_2O_5	$MgO \cdot P_2O_5$ $2MgO \cdot P_2O_5$ $3MgO \cdot P_2O_5$	stoichiometric stoichiometric stoichiometric	- S_f, H_{fus} [Oetting, 1963] SGPS changed

Modelling of ternary P-containing phases

System	*Phase*	*Description*	*Used data*
FeO-Fe_2O_3-P_2O_5	$Fe_7P_6O_{24}$ $Fe_{18}P_2O_{24}$ $Fe_{10}P_6O_{26}$ $Fe_4P_2O_{10}$	$3FeO\cdot 2Fe_2O_3\cdot 3P_2O_5$ (stoichiometric) $16FeO\cdot Fe_2O_3\cdot P_2O_5$ (stoichiometric) $8FeO\cdot Fe_2O_3\cdot 3P_2O_5$ (stoichiometric) $2FeO\cdot Fe_2O_3\cdot P_2O_5$ (stoichiometric)	- - - -

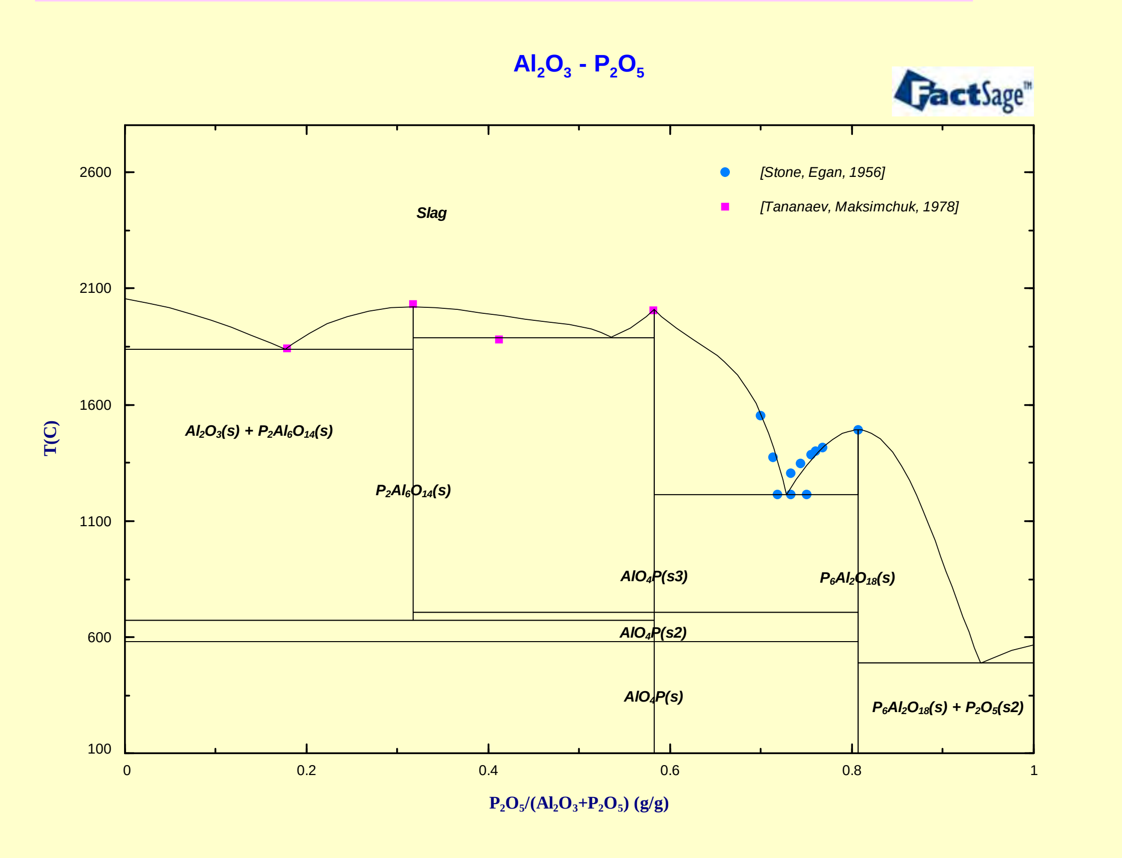
Binary Fe-P phase diagram [99Lee]

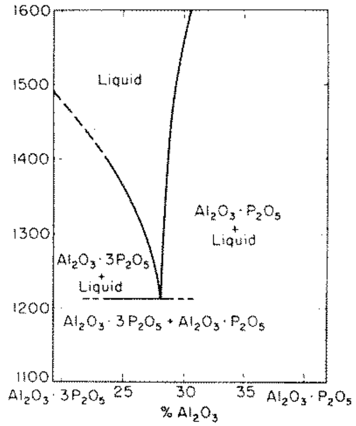




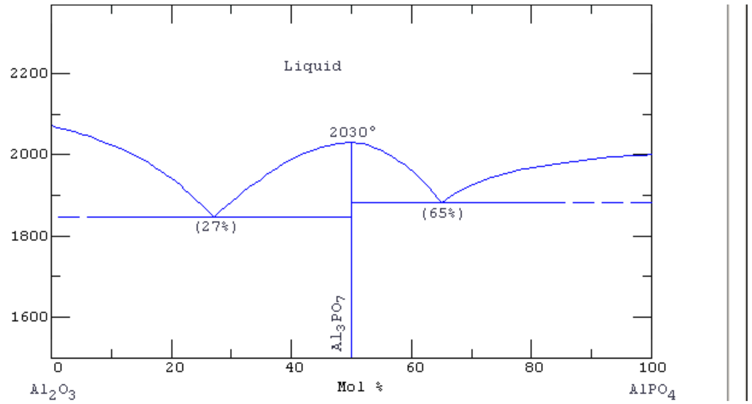
Binary Al_2O_3-P_2O_5 phase diagram

Berlinite ($AlPO_4$) - It was first described in 1868 for an occurrence in the Västana iron mine, Scania, Sweden and named for Nils Johan Berlin (1812–1891) of Lund University.





P.E. Stone, E.P. Egan, J.R. Lehr, J. Am, Ceram. Soc., 39 [3], (1956), pp.89-98.



I.V. Tananaev, E.V. Maksimchuk,Y. G. Bushuev, S.A. Shestov, Izv. Akad. Nauk SSSR, Neorg. Mater., 14 [4], (1978), pp.719-722.

CaO-P_2O_5 phase diagram

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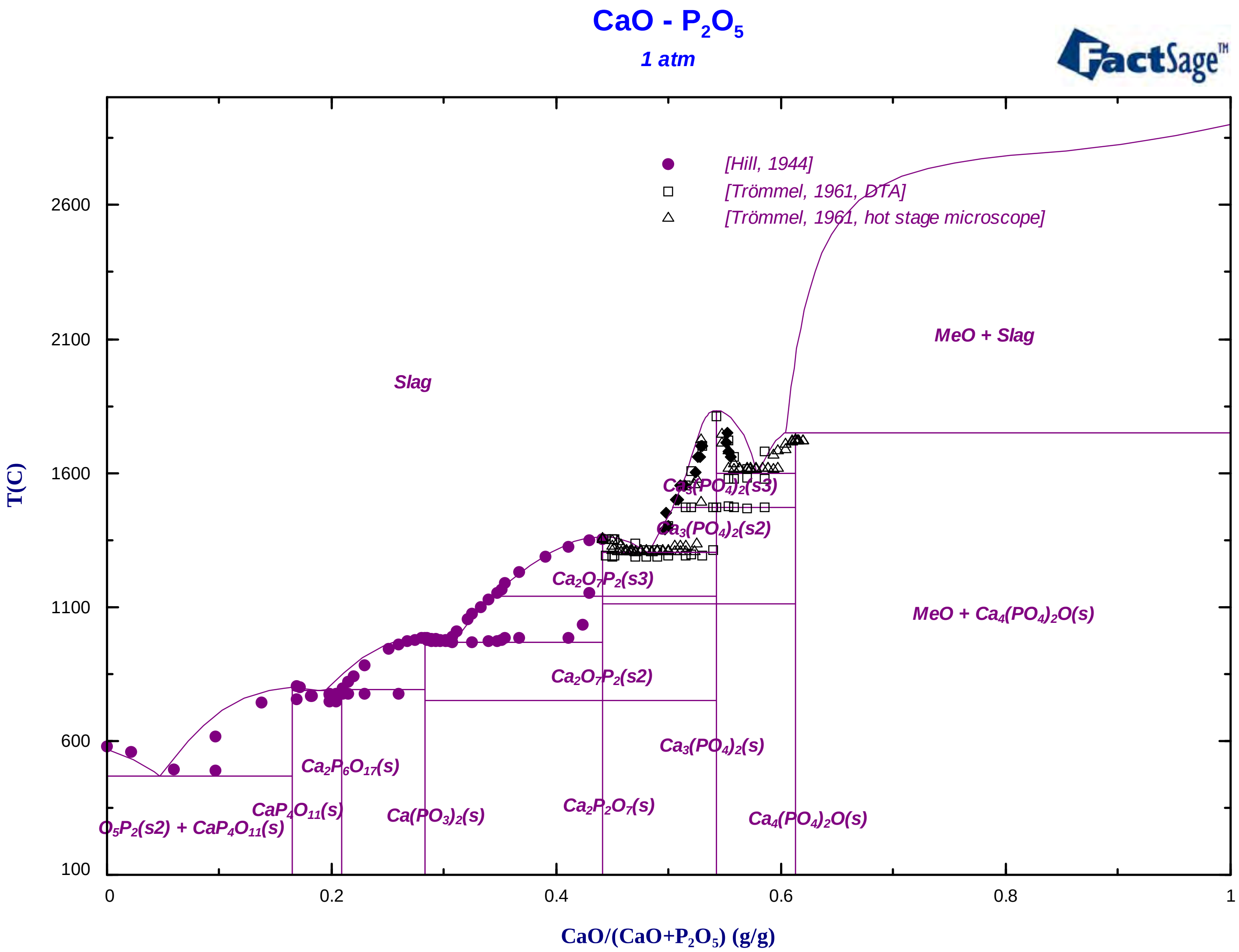
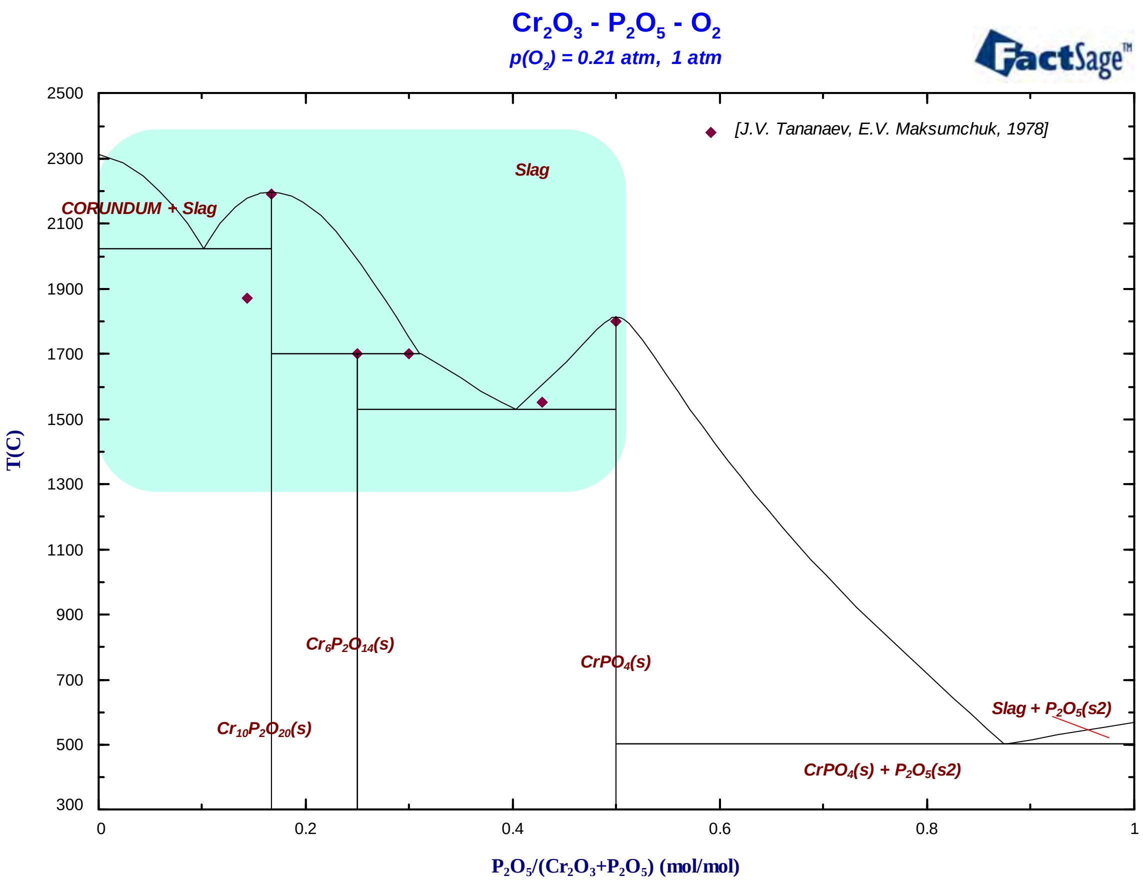
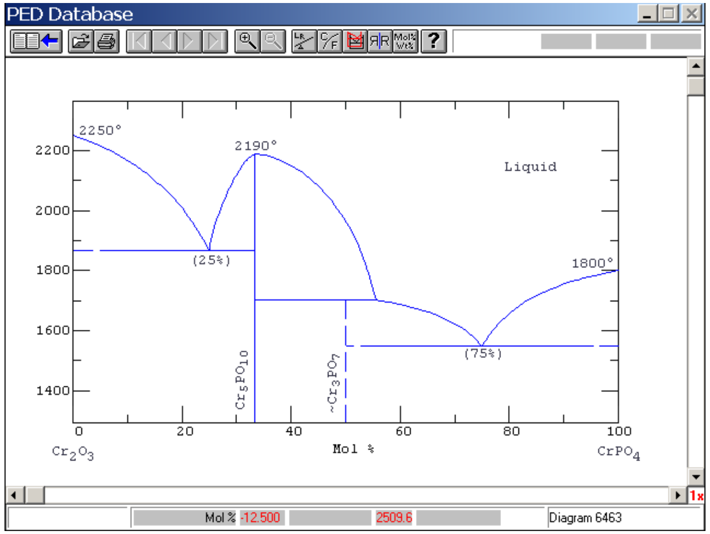


Cr_2O_3-P_2O_5 phase diagram in air





I.V. Tananaev, E.V. Maksimchuk, Y. G. Bushuev, S.A. Shestov, Izv. Akad. Nauk SSSR, Neorg. Mater., 14 [4], (1978), pp.719-722.

FeO-P_2O_5 phase diagram in equilibrium with Fe

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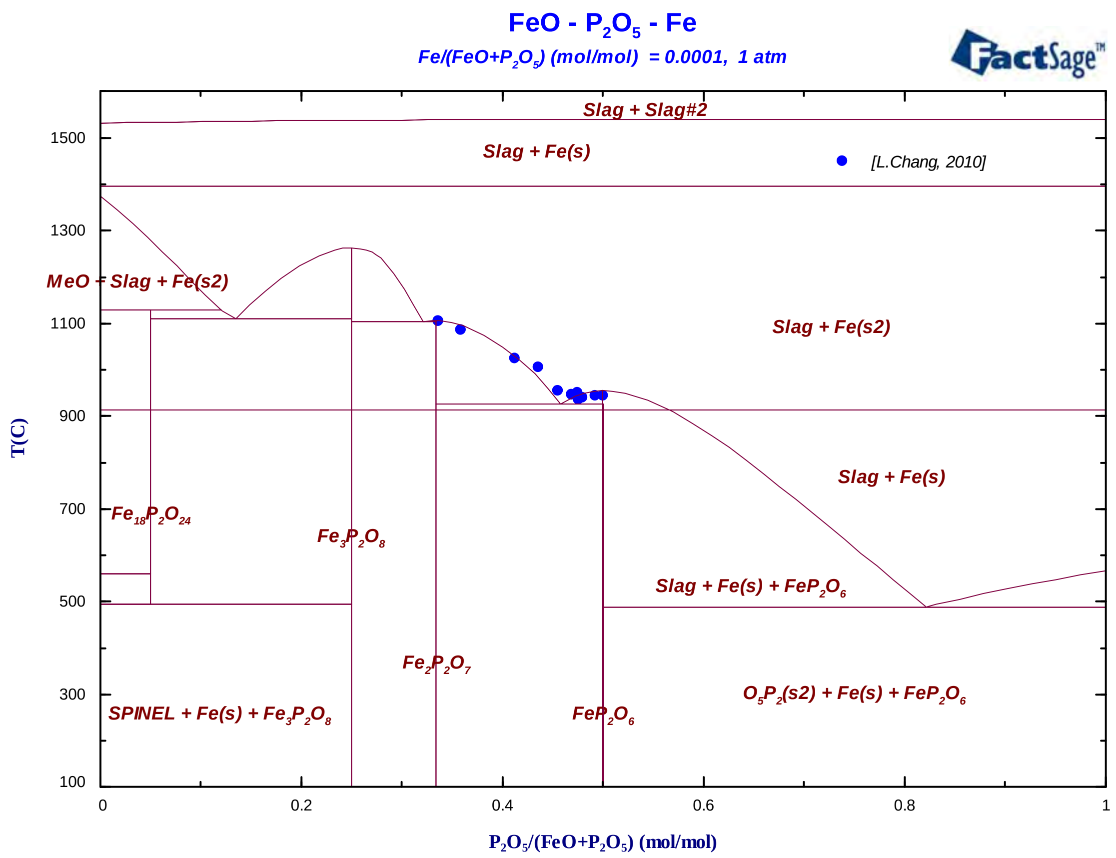
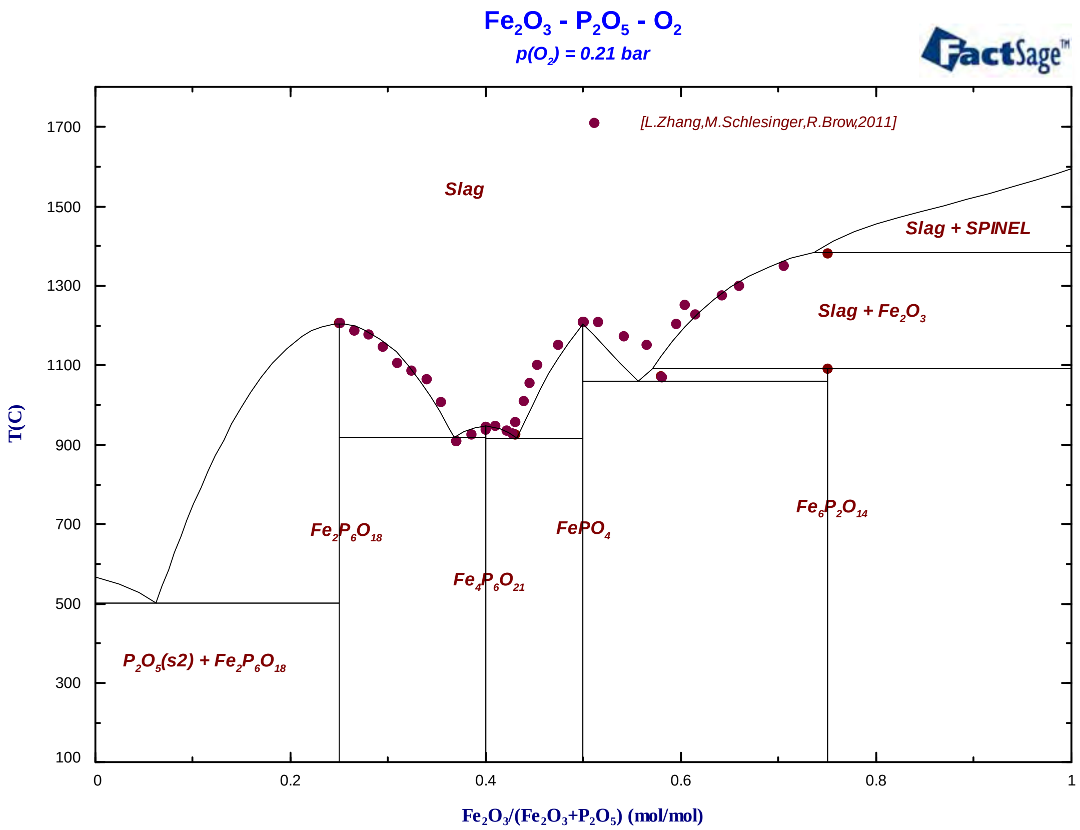
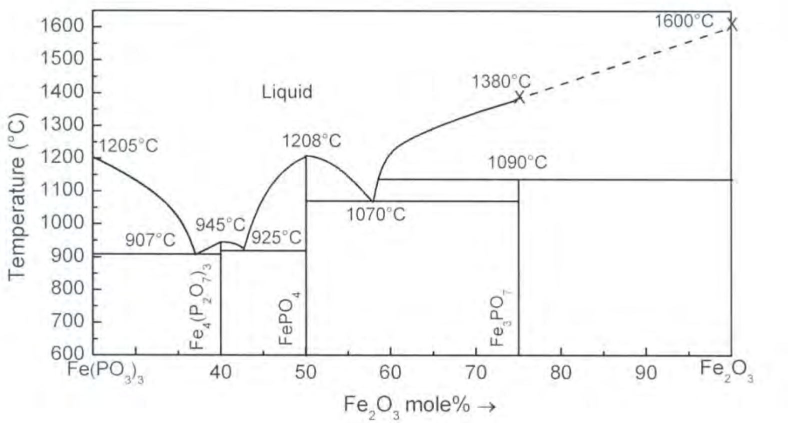
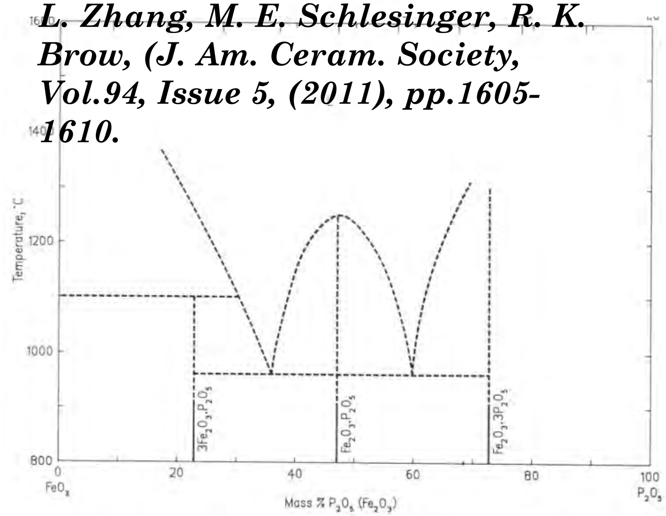


Fe_2O_3–P_2O_5 phase diagram in air



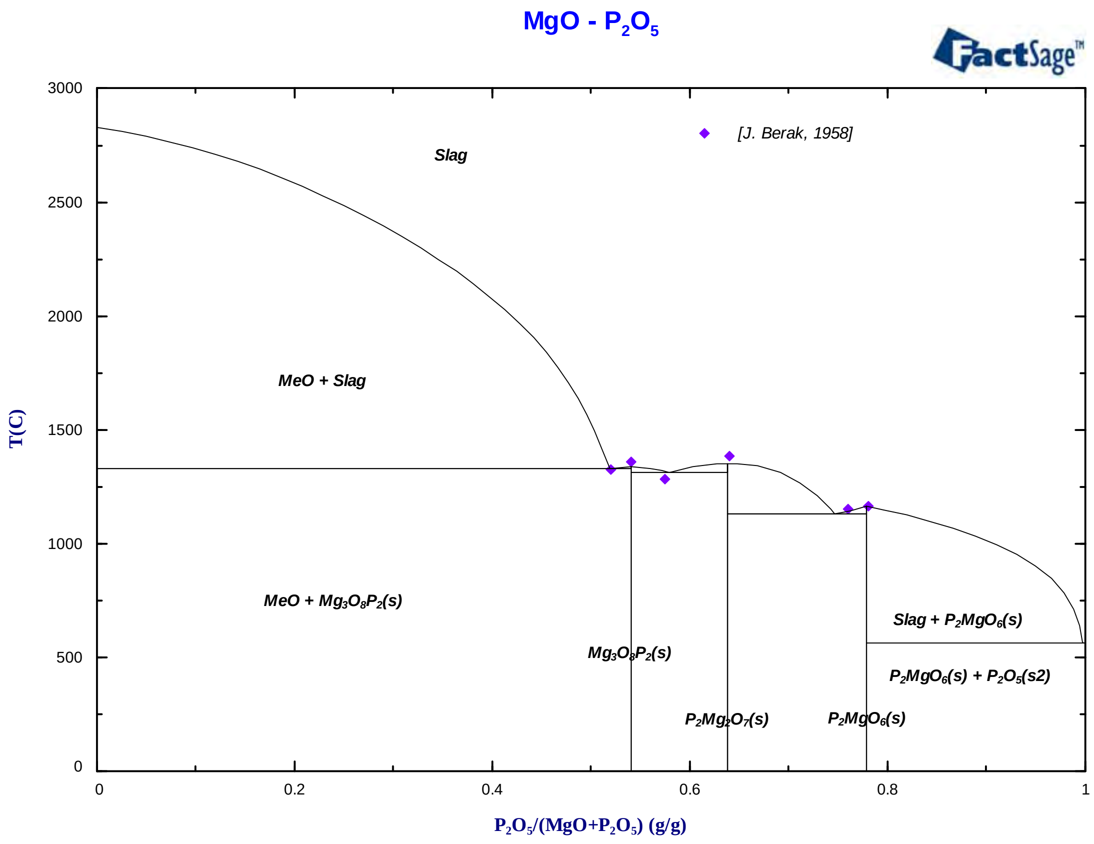


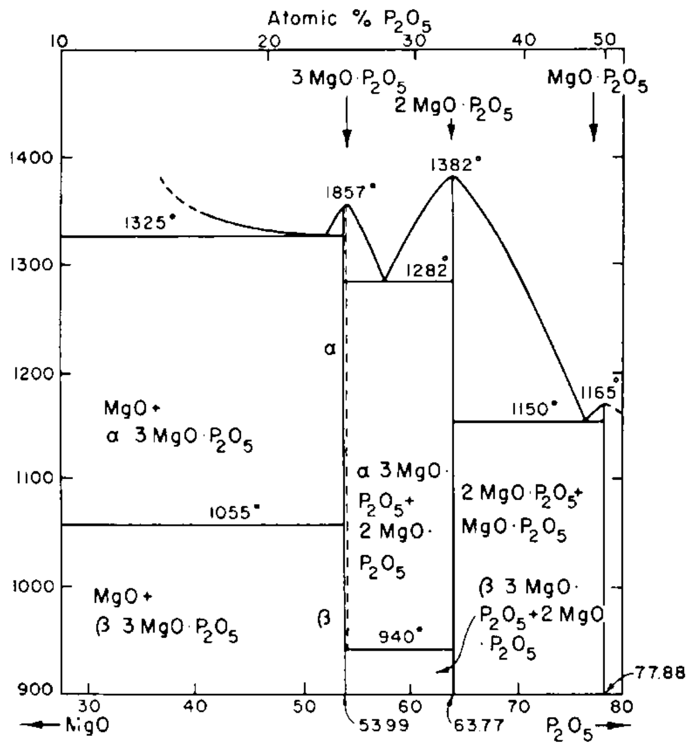
L. Zhang, M. E. Schlesinger, R. K. Brow, (J. Am. Ceram. Society, Vol.94, Issue 5, (2011), pp.1605-1610.



Slag Atlas, 2nd Ed., Verlag Stahl-Eisen, Düsseldorf, 1995., p.68.

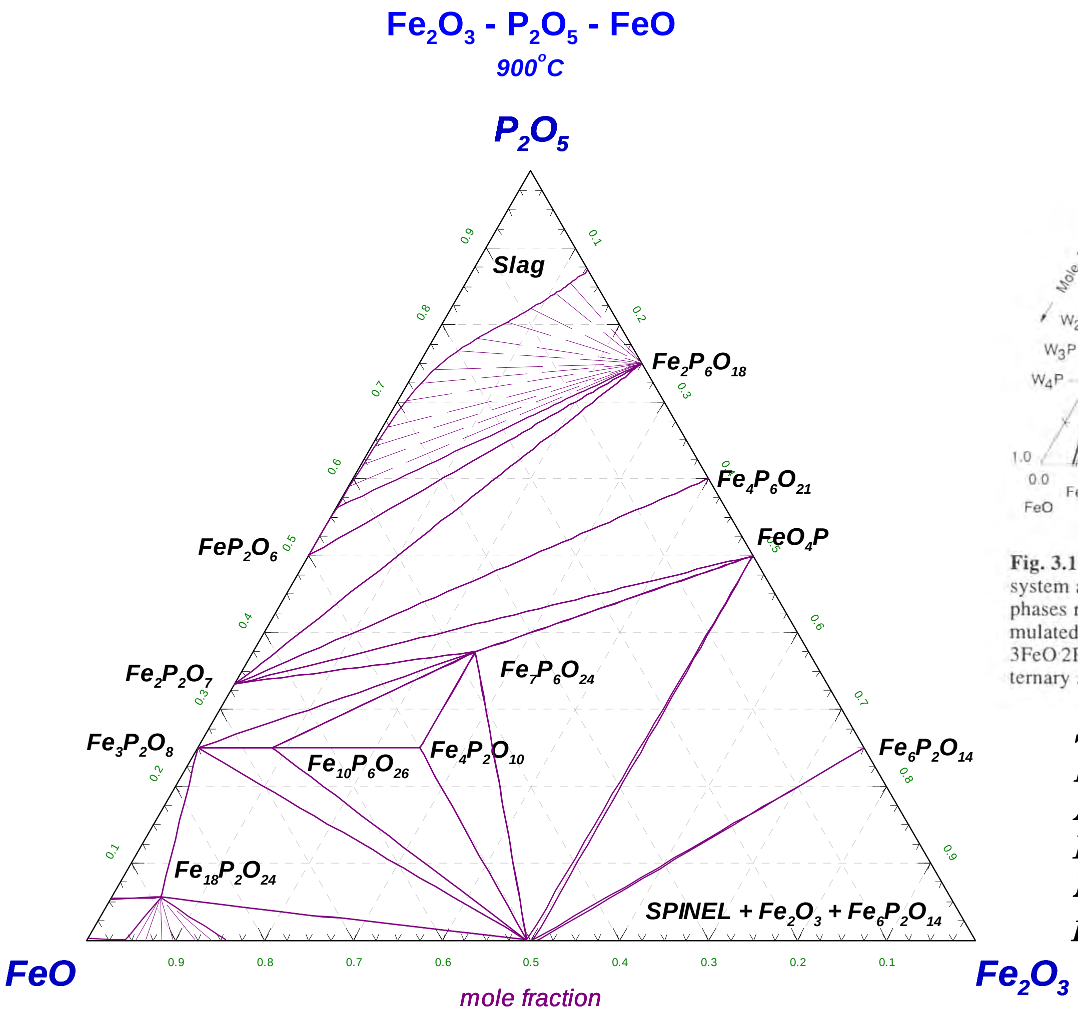
MgO-P_2O_5 phase diagram





J. Berak, Rocz. Chem., 32 [1], (1958), pp.17-22.

Isothermal section in $FeO-Fe_2O_3-P_2O_5$ at 900°C



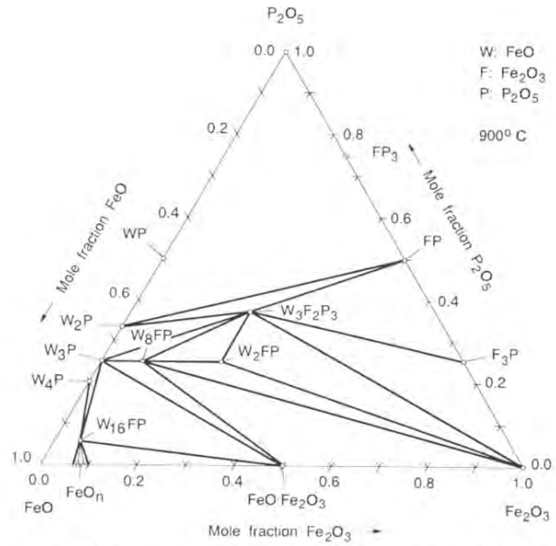


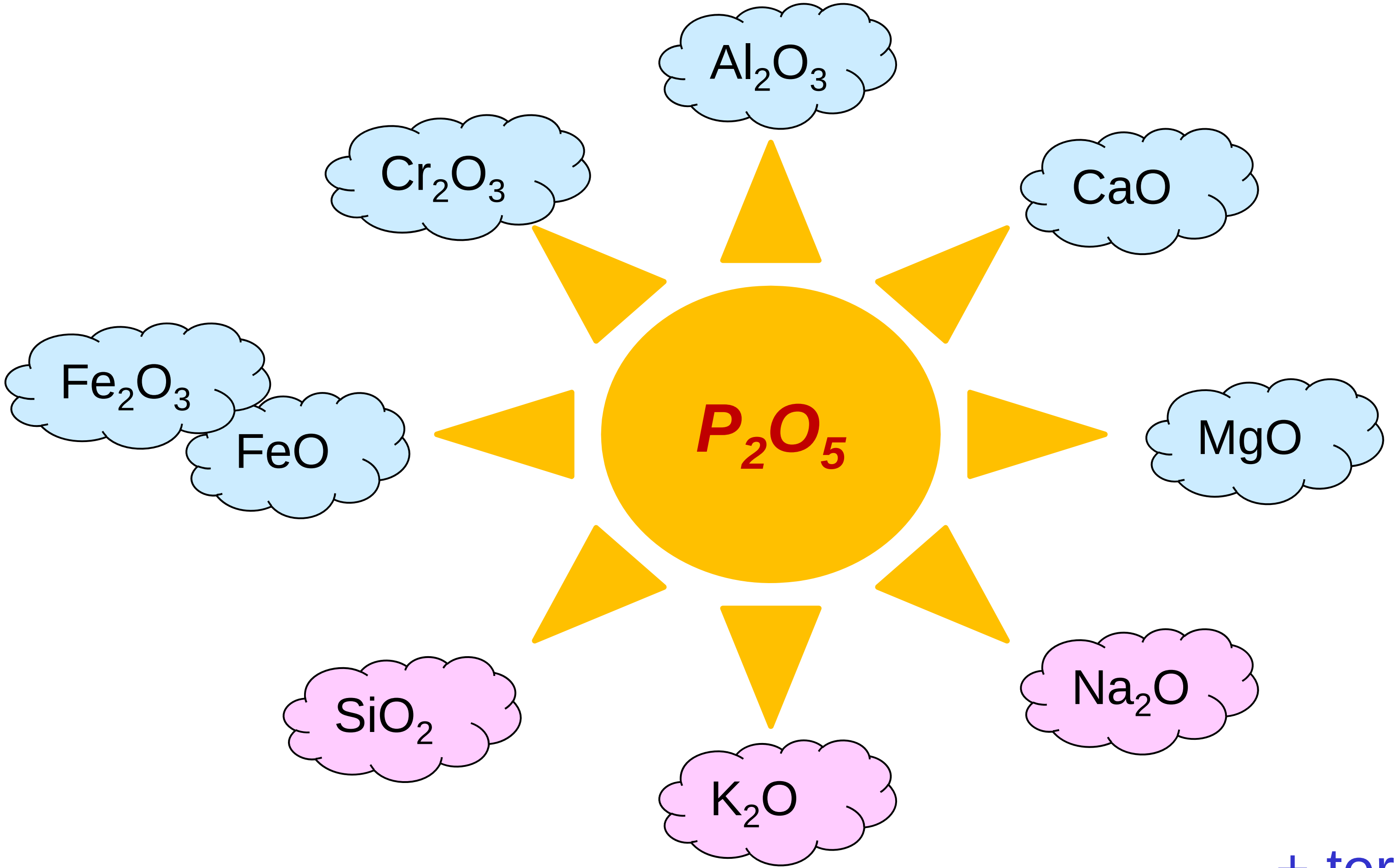
Fig. 3.105. Isothermal section through the $FeO-Fe_2O_3-P_2O_5$ system at 900 °C after Modaressi et al. [3]. Two ternary phases reported previously by Wentrup [1] have been reformulated. According to Gorgunov et al. [5], $3FeO \cdot 2Fe_2O_3 \cdot 3P_2O_5$ melts at 996 °C. For a discussion of the ternary system Fe-O-P, see Raghavan [6].

Slag Atlas, 2nd Ed., Verlag Stahl-Eisen, Düsseldorf, 1995., p.68.
A. Modaressi, J.C. Kaell, B.Malaman, R. Gerardi, C. Gleitze, Mat. Res. Bull. 18 (1983), No. 1, pp.101-109.

Conclusions

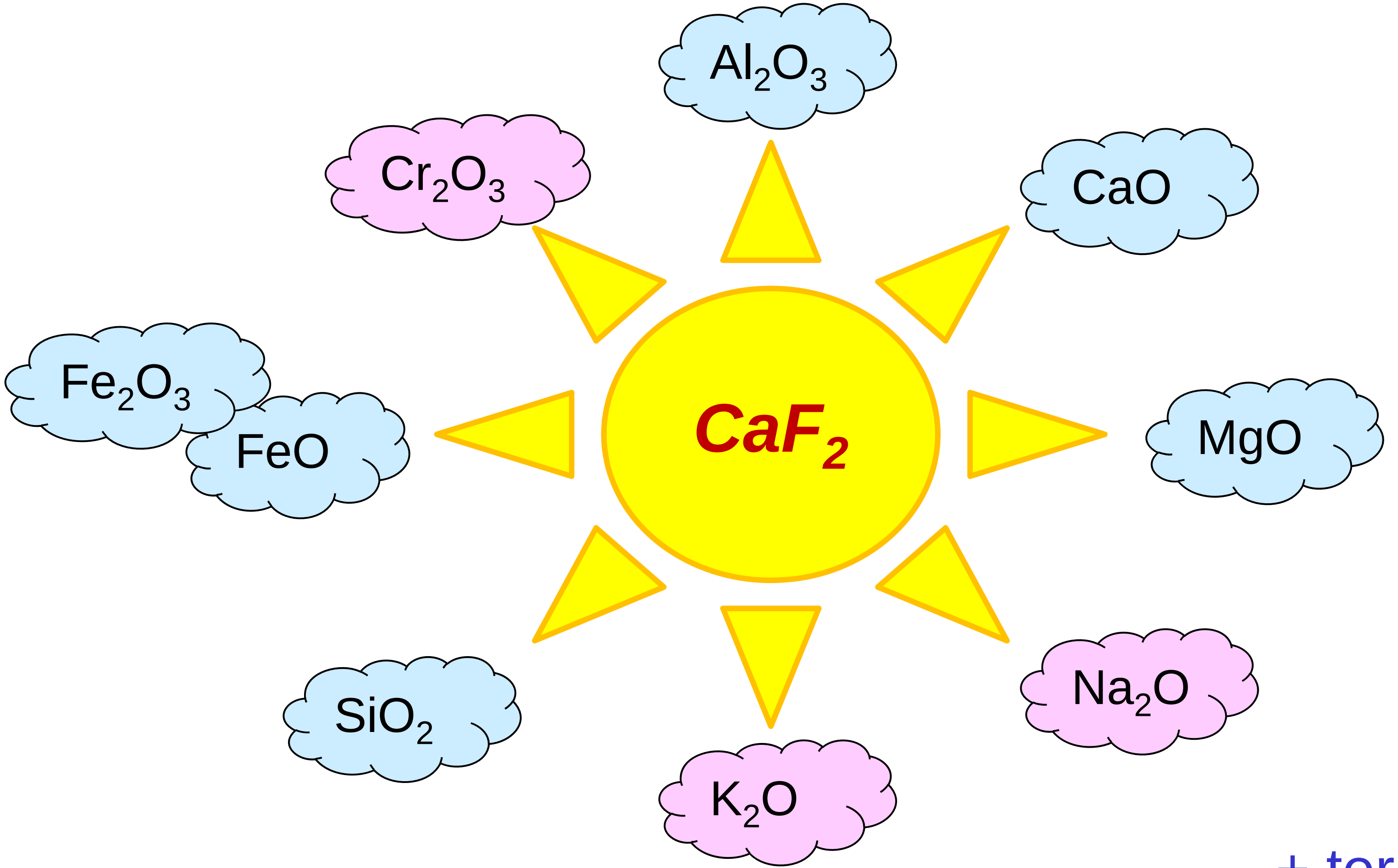
- All systems were assessed using experimental phase diagram information. Other experimental information, eg. enthalpies and activities, is scarce.
- The liquid phase in all subsystems was evaluated using associate species model (two cations per species).
- CaF_2 has so far been integrated into the reduced core system CaO-MgO-Al_2O_3-FeO-Fe_2O_3-SiO_2. All binary and 5 ternary systems were described.
- The stoichiometric phases $3CaO{\cdot}3Al_2O_3{\cdot}CaF_2$, $11CaO{\cdot}7Al_2O_3{\cdot}CaF_2$, $4CaO{\cdot}2SiO_2{\cdot}CaF_2$, $3CaO{\cdot}2SiO_2{\cdot}CaF_2$(Cuspidine), and $9CaO{\cdot}3SiO_2{\cdot}CaF_2$ were incorporated.
- The Al_2O_3-CaF_2-CaO system is critically evaluated according to the experimentally determined miscibility gap in the liquid phase.
- In the thermodynamic assessments of the binary systems Al_2O_3-P_2O_5, CaO-P_2O_5, Cr_2O_3-P_2O_5, FeO-P_2O_5, Fe_2O_3-P_2O_5, MgO-P_2O_5 as well as the ternary FeO-Fe_2O_3-P_2O_5 system 28 stoichiometric solid phases containing P were incorporated.

Future developments



+ ternaries

Future developments



+ ternaries?

Happy Birthday, Gunnar!