Characterization of Phase Equilibria and Thermodynamics with Integrated Experimental and Modelling Approach for Complex Lead Primary and Recycling Processing

E. Jak, M. Shevchenko, D. Shishin, T. Hidayat and P. C. Hayes

Abstract Substantial advances have been made in the characterization of phase equilibria and thermodynamic properties of complex multi-phase equilibria in gas—slag—matte—metal—speiss—solids systems directly relevant to primary and recycling lead smelting. The present review summarizes some of the outcomes of recent studies of the multi-component and multi-phase system containing PbO–ZnO–"Cu_2O"–FeO–Fe_2O_3–CaO–SiO_2–S as major components, Al_2O_3–MgO–Cr_2O_3 as minor slagging components, and As–Bi–Ni–Sb–Sn–Ag–Au–Co as other minor components. The research methodology that has been employed involves the integration of experimental measurement and thermodynamic database development. The target of these fundamental studies is to characterize phase equilibria and thermodynamics of this multi-component system and to develop a thermodynamic database as part of a computer package that can be used to assist in optimization of all stages of the value chain from sintering, smelting, and zinc fuming in primary production to complex chemistry processing in recycling. A summary and examples of research outcomes and applications are presented.

Keywords Phase equilibria · Thermodynamic modelling · Primary lead · Secondary lead · Matte formation · Minor elements · Lead refining · Speiss formation

E. Jak (✉) · M. Shevchenko · D. Shishin · T. Hidayat · P. C. Hayes
School of Chemical Engineering, PYROSEARCH, Pyrometallurgy Innovation Centre, The University of Queensland, Brisbane, QLD 4072, Australia
e-mail: e.jak@uq.edu.au

T. Hidayat
Department of Metallurgical Engineering, Bandung University of Science and Technology, Bandung, Indonesia

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Introduction

In the survey of global lead smelter operations reported in the Pb/Zn 2010 symposium [1], two major trends were noted: (i) the relatively rapid changes to process technologies and (ii) the increasingly diverse range of source materials, the use of mixtures of primary ores, complex process wastes and recycled materials by industry in their feed materials. These trends have continued at an accelerating pace. The task of optimizing process design and operation of modern metallurgical plants has become more complex. Accurate and reliable descriptions of phase equilibria and thermodynamics properties are required to describe these multi-component systems over wide ranges of bulk compositions and process conditions. In the previous review of phase chemistry of lead smelting systems [2], some of the advances that had been made in characterizing the PbO–ZnO–FeO–Fe_2O_3–CaO–SiO_2 system and the development of a thermodynamic database describing this base slag system were highlighted. The accuracy of the database at that time was reliant on experimental data available at two extremes in process conditions, in air and in equilibrium with metallic iron. Phase equilibrium data were available on the majority of PbO- and ZnO-containing low-order SiO_2-containing sub-systems, on selected multi-component systems in PbO–ZnO–"Fe_2O_3"–CaO–SiO_2 in air and ZnO–"FeO"–CaO–SiO_2 in equilibrium with metallic iron.

Since that time, with the financial support from major lead primary and recycling companies, this research program has further progressed and significant advances have been made to increase the range of chemical systems that can be experimentally characterized in these lead-containing systems. With the development of an integrated experimental and thermodynamic modelling research methodology, a program on development of the thermodynamic database for the complex multi-component and multi-phase gas—slag—matte—speiss—metal—solids PbO–ZnO–"Cu_2O"–FeO–Fe_2O_3–SiO_2–S–Al_2O_3–CaO–MgO–Cr_2O_3–(As–Bi–Ni–Sb–Sn–Ag–Au–Co) system is currently underway.

The strategic target of the program is to be able to produce sophisticated computer-based tools that can be used to predict the outcomes from industrial reactor systems, to support the introduction of feed-forward control systems, to increase the productivities and efficiencies of pyrometallurgical processes, and, ultimately, to develop and implement the use of computer-based virtual reactor models of these systems. These tools have the potential to deliver substantial economic and productivity benefits to industry as well as improve metal recycling and energy efficiency.

The present paper provides a brief outline of the following:

- The extensive experimental and thermodynamic database development research capability that has been established.

- Selected examples demonstrating recent research progress on phase equilibria in the development of the 19-component database, including equilibria in high-PbO-containing low-silica systems, equilibria with Pb metal; equilibria with Cu–Pb metal alloys; S-containing systems; minor elements distributions; refining conditions with speiss.
- Examples of industrial applications of the database to the analysis of complex multi-component systems.

A more detailed review of the information available in the literature of phase equilibria in high-temperature lead smelting and refining systems, and the corresponding references, will be given in a forthcoming journal version of the paper [3].

Outline of Research Techniques

An overview of the latest advances in the integrated experimental and modelling research approach to the thermodynamic database development for these pyrometallurgical systems has been provided in [4]. The unambiguous determination of the unique set of binary and ternary model parameters accurately describing a whole range of conditions from low- to high-order systems is critical for the accuracy and predictive power of the tool, and requires experimental data for all binary and ternary sub-systems, and for selected quaternary sub-systems. If there are no experimental data—there can be no unique reliable accurate parameters and therefore no reliable model.

Recently, significant advances in experimental techniques and capabilities have been achieved due to the development and availability of advanced microanalytical techniques including (i) electron probe X-ray microanalysis (EPMA) and (ii) laser ablation inductively coupled plasma mass spectrometry (LAICPMS). These microanalysis techniques provide breakthrough capabilities in phase equilibrium studies for pyrometallurgical applications greatly extending the range of systems that can be studied, and the accuracy of characterization with very few limitations on the range of bulk compositions and conditions that can be characterised. Details of the experimental technique based on the equilibration at fixed conditions, quenching, and direct measurement of phase compositions using microanalysis techniques EPMA and LAICPMS developed by the authors are outlined in the previous publications.

Thermodynamic modelling is performed with the FactSage computer package. The methodology used for the development of the database involves, first, developing a preliminary database using experimental data available in the literature data, the identification of key areas of disagreement or thermodynamic inconsistency, and the selection of critical experiments required to resolve these inconsistencies or omissions. The databases are developed so as to achieve a self-consistent set of parameters for the thermodynamic models, accurately describing phase equilibria and all other thermodynamic properties for all phases over the complete range of conditions in terms of temperature, composition, and atmospheric conditions. The

Table 1 Summary of phases and elements described in the current internal PYROSEARCH solution databases (UQPY-2019) important for Pb–Cu–Zn pyrometallurgical processing. Further detailed description is provided in [5]

Slag:(Cu^{1+}, Fe^{2+}, Fe^{3+}, Si^{4+}, Al^{3+}, Ca^{2+}, Mg^{2+}, Pb^{2+}, Zn^{2+}, Ni^{2+}, Sn^{2+}, Sb^{3+}, As^{3+}, Bi^{3+}, Ag^{1+}, Au^{1+})(O^{2-}, S^{2-}), modified quasichemical formalism in quadruplet approximation
Spinel: [Cu^{2+}, Fe^{2+}, Fe^{3+}, Ni^{2+}, Al^{3+}, Mg^{2+}, Zn^{2+}]^{tetr}[Cu^{2+}, Fe^{2+}, Fe^{3+}, Ni^{2+}, Al^{3+}, Ca^{2+}, Mg^{2+}, Zn^{2+}, $vacancy^{0}$]$_2^{oct}O_4$, compound energy formalism
Liquid matte/metal/speiss: (Cu^{I}, Cu^{II}, Fe^{II}, Fe^{III}, Pb^{II}, Zn^{II}, Ni^{II}, Sn^{II}, Sb^{III}, As^{III}, Bi^{III}, Ag^{I}, Au^{I}, O^{II+}, S^{II+}), modified quasichemical formalism in pair approximation
Monoxide: (CuO, FeO, $FeO_{1.5}$, NiO, $AlO_{1.5}$, CaO, MgO), Bragg-Williams formalism
Mullite: (Al^{3+}, Fe^{3+})$_2$(Al^{3+}, Si^{4+})(O^{2-}, $vacancy^{0}$)$_5$, compound energy formalism
Olivine: [Fe^{2+}, Ni^{2+}, Ca^{2+}, Mg^{2+}, Zn^{2+}]^{M2}[Fe^{2+}, Ni^{2+}, Ca^{2+}, Mg^{2+}, Zn^{2+}]$^{M1}SiO_4$, compound energy formalism
Melilite: [Ca^{2+}, Pb^{2+}]$_2$[Fe^{2+}, Fe^{3+}, Al^{3+}, Zn^{2+}][Fe^{3+}, Al^{3+}, Si^{4+}] $_2O_7$, compound energy formalism
Pyroxenes: [Fe^{2+}, Ca^{2+}, Mg^{2+}, Ni^{2+}]^{M2}[Fe^{2+}, Fe^{3+}, Mg^{2+}, Al^{3+}, Ni^{2+}]^{M1}[Fe^{3+}, Al^{3+}, Si^{4+}]$^{B}Si^{A}O_6$, compound energy formalism
Dicalcium silicates: (Ca_2SiO_4, Fe_2SiO_4, Mg_2SiO_4, Pb_2SiO_4, Zn_2SiO_4), Bragg-Williams formalism
Wollastonite: ($CaSiO_3$, $FeSiO_3$, $MgSiO_3$, $ZnSiO_3$), Bragg-Williams formalism
Willemite: [Zn^{2+}, Fe^{2+}, Mg^{2+}][Zn^{2+}, Fe^{2+}, Mg^{2+}]SiO_4
Corundum: ($FeO_{1.5}$, $AlO_{1.5}$, NiO), Bragg-Williams formalism
Fcc and bcc solids alloys: (Fe, Cu, Ni, O, S, Pb, Zn, As, Sb), Bragg-Williams formalism
Digenite-bornite: (Cu_2S, FeS, PbS, ZnS, Ni_2S, $vacancy_2S$), Bragg-Williams formalism
MeS cubic: (FeS, PbS, CaS, MgS, vacancyS), Bragg-Williams formalism
Solid stoichiometric compounds: (over 150 phases) including solid oxide, metal, sulfide, and sulfate solids
Ideal gas: >100 species including N_2, H_2O, CO, CO_2, SO_2, SO, As_2, AsS, AsO, Zn, ZnS, ZnO, AgS, Pb, PbS, PbO, Bi, Bi_2, BiO, BiS, Sn, SnO, SnS, Ag, AgO, AgS, Sb, Sb_2, SbO, SbS

overview of solution phases and thermodynamic models developed to date is given in Table 1.

Overview of Recent Results

The following section presents selected examples demonstrating recent research progress on the characterization of phase equilibria in the 19-component system of importance for Pb and Zn pyrometallurgy.

Phase Equilibria in High-PbO Slag Systems in Air, with Pb Metal and with Pb–Cu Metal Alloys

There are major experimental difficulties in measuring phase equilibria in high-PbO slag systems in oxidizing conditions and particularly in equilibrium with Pb and Pb–Cu metal alloys. The slags and liquid metal alloys are chemically aggressive, dissolving conventional refractory and metal containment materials resulting in contamination of the system with unwanted chemical components, difficulties in retaining the original liquid phase as a uniform amorphous phase on quenching due to low viscosity, fast crystallization, and high vapor pressures leading to selective vaporization of metal and metal oxide species. These difficulties have been overcome in recent research [6], which has resulted in important advances in experimental technique. These improved techniques include the use of primary phase substrates and closed system experiments in which samples are sealed in evacuated silica ampoules. Using these approaches, many PbO-containing systems have been characterized; some examples are listed below:

In air, the liquidus and phase equilibria for the systems, the PbO–"$FeO_{1.5}$"–CaO [6] and PbO–"$FeO_{1.5}$"–SiO_2 [7].
In equilibrium with metallic Pb, the liquidus and phase equilibria for the system PbO–"FeO"–SiO_2 [8].
In equilibrium with Cu–Pb metal alloys, new information on the liquidus surface for the PbO–"Cu_2O"–SiO_2 system in equilibrium with Pb–Cu metal and the distribution of copper between slag and bullion [9]. Note that the Pb–Cu metal alloy present in this system is in the solid state for a range of Pb/Cu ratios.

In summary, with the development of improved experimental techniques, many low-order oxide and oxide-metal sub-systems of high importance for Pb and Zn complex pyrometallurgy have been accurately characterized over the last decade. These include many Pb- and Zn-containing oxide sub-systems. Using the same methodology and similar research techniques, a great deal of new fundamental data on phase equilibria in Cu-containing oxide-metal sub-systems has also recently become available.

Slag-Matte Equilibria

The distribution of elements between slag and matte is one of the key factors in lead smelting and refining. No systematic studies were found in the open literature describing the equilibria between the lead slag, matte, and metal. The problems associated with characterizing phase equilibria in lead matte systems are even more challenging than those with metal due to the high vapor pressures of PbS gas species. With the recent advances in experimental capability, it is now possible to obtain accurate measurements of metal distributions in multi-phase systems containing sulfur.

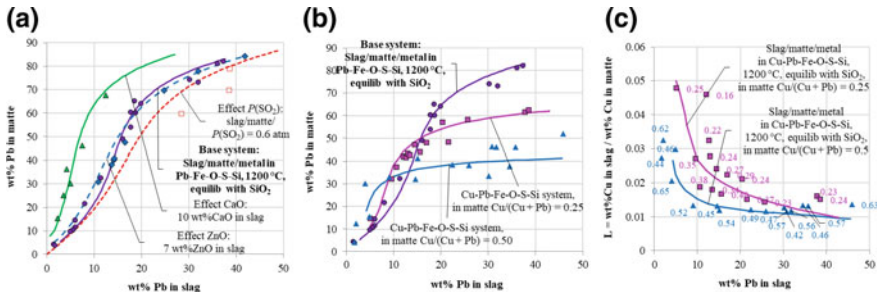

Fig. 1 Slag-matte-metal-SiO_2 equilibria in the Pb–Fe–O–S–Si–(Ca, Zn, Cu) system at 1200 °C showing **a** effects of CaO [4], ZnO, $P(SO_2)$, and Cu, including, **b** %Pb in slag and in, **c** the distribution coefficient of Cu between PbO-containing slag and matte. The lines represent FactSage calculations with current internal PYROSEARCH database. The symbols represent experimental data from PYROSEARCH (2016–2019). For $P(SO_2) = 0.6$ atm, the metal phase is not present

At PYROSEARCH, investigation of the equilibria between lead slag, matte, and metal in the Pb–Fe–O–S–Si system has started; the effects of temperature, $P(SO_2)$, CaO, Al_2O_3, ZnO, and Cu_2O, on the three phase equilibria have been measured. The distribution of lead and copper between matte and slag phases in the Pb–Fe–O–S–Si system with addition of CaO, ZnO, and Cu_2O over a range of conditions is shown in Fig. 1.

Slag-matte systems are important in processing of complex Cu–Pb–Zn materials. Most of the previous studies have been focused on Cu and Cu–Ni slag-matte systems. Almost no data in laboratory-controlled conditions were found in the literature for the Pb–Cu–Zn slag-matte systems. The new experimental techniques developed in PYROSEARCH enable experimental investigations in these systems to be undertaken. The availability of these new data greatly improves the accuracy and reliability of the thermodynamic database for predictions relevant to Cu–Pb–Zn smelting.

Minor Element Distributions

By combining improved experimental techniques, electron probe X-ray microanalysis (EPMA) and laser ablation inductively coupled plasma mass spectrometer (LAICPMS) microanalysis techniques and experiments in closed system in ampoules, minor element distributions between phases can now be accurately measured. The effective partial pressures of $P(O_2)$ and $P(S_2)$ in these systems can be derived from the microanalytical measurement of trace metal concentrations in the condensed phases, e.g. "Cu_2O" concentrations in ZnO–"FeO"–SiO_2 slags. The experimental data available to date as well as new experimental data obtained recently at PYROSEARCH have been used to develop the thermodynamic database describing these systems. The predictions presented in Fig. 2a–d provide a summary and

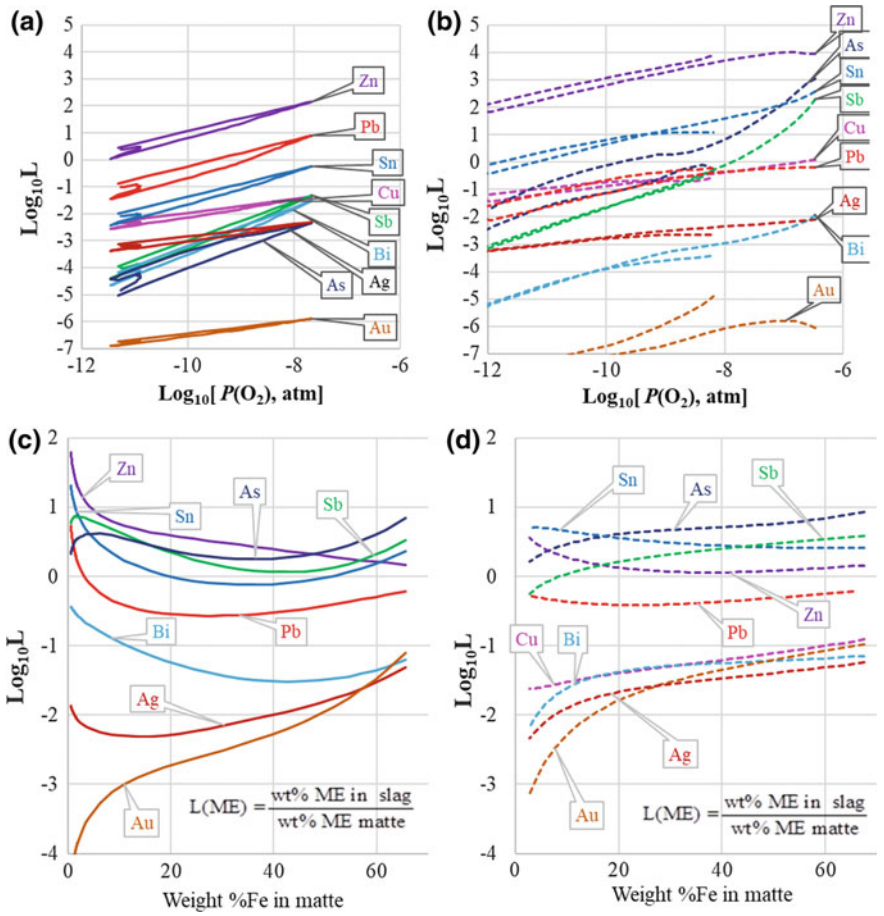

Fig. 2 Summary of distribution coefficients of minor elements (ME) between slag and liquid metal in equilibrium with solid SiO_2 at 1200 °C in the following systems **a** Cu–Fe–O–Si–(ME), slag/liquid copper, **b** Pb–Fe–O–Si–(ME), slag/liquid lead, and between slag and matte in equilibrium with solid SiO_2, at $P(SO_2) = 0.25$ atm and 1200 °C in the following systems, **c** Cu–Fe–O–S–Si–(ME), slag/Cu_2S–FeS matte, and **d** Pb–Fe–O–S–Si–(ME), slag/PbS–FeS matte. The lines represent FactSage calculations with current internal PYROSEARCH database

comparison of slag/metal and slag/matte distribution coefficients of a number of minor elements in copper and lead systems.

Most of the previous studies on minor element distributions reported in the literature on non-ferrous systems are focused on Cu– or Cu–Ni–systems. Very limited data are available for the Pb– and Pb–Cu–systems. Examples of recent experimental measurements of distributions of As, Sb, Cu, and Ag between PbO–FeO–Fe_2O_3–CaO–SiO_2 slag and liquid Pb as a function of $P(O_2)$ and slag composition are given in [10]. The distributions of Ag, Au, Bi, and Zn between Pb metal and Pb–Fe–O–Si slag are reported in [11]. The distribution coefficients between slag/liquid lead and

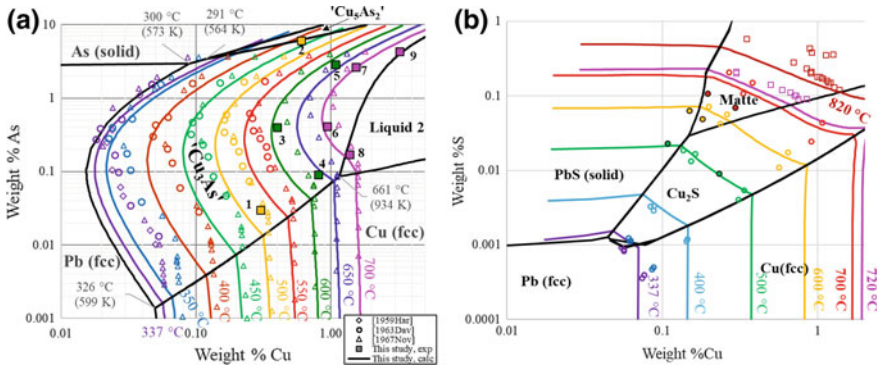

Fig. 3 The Pb-corner of the liquidus surface in the **a** Cu–Pb–As and **b** Cu–Pb–S systems. The lines represent FactSage calculations with current internal PYROSEARCH database. Symbols represent experimental data

slag/PbS–FeS matte are measured and also integrated into the database describing copper smelting systems.

Metal-Speiss Equilibria

During the refining stages of lead processing copper–arsenic and sulfur-containing phases are formed. Figure 3 demonstrates experimental data and thermodynamic database predictions for the key refining systems of Pb–Cu–As and Pb–Cu–S.

The availability of accurate data for the binary systems is critical for the development of accurate thermodynamic databases for systems in which the speiss phase is formed. Critical assessments are available for the Cu–Sb, Cu–Sn, and Cu–As systems. The solubilities of the elements in liquid lead at low temperatures have been given in the following Pb–Cu–As, Pb–Cu–S, Pb–Cu–Sb, and Pb–Cu–Sn systems. Even fewer results are available for the multi-component systems, where solid solutions may form between solid As- and Sb-containing compounds. Some kinetic aspects of copper removal in the presence of speiss-forming elements were reported.

Overview of Industrial Applications

Systematic Analysis of Pb–Cu–Fe Matte/Slag Equilibria

Having obtained new experimental data and incorporated these into the optimized thermodynamic database, it is possible to use these powerful predictive tools to systematically analyze the trends in phase formation and chemical compositions of

these phases in complex chemical systems. The availability of the thermodynamic database enables the systems to be presented and viewed in different ways depending on the needs of the user.

The example presented in Fig. 4a shows a projection of the matte compositions in the Pb–Cu–Fe–O–S–Si slag-matte-metal system in equilibrium with SiO_2 on to the Cu–Fe–Pb composition triangle. Figure 4b contains information on the %PbO in slag corresponding to these matte compositions. The lines drawn on the figure correspond to the data provided previously in Fig. 1a for selected compositions of Cu/(Cu + Pb) in matte. By constructing information in this way, the trends in chemical behavior can be visualized and more efficiently analyzed. This type of calculation and representation can be undertaken for any set of process conditions and bulk chemical compositions to assess the potential outcomes of these multi-phase equilibrium reactions. For instance, the formation of the matte phase can be a problem during the bullion tapping in the Kivcet process. Thermodynamic calculations can provide better understanding of desulfurization reactions in the shaft.

Multi-component Fluxing Diagrams

The thermodynamic database can also be used to construct diagrams that can be used to assist in the selection of optimum flux additions to slags. Examples of the application of these diagrams to the optimization of flux additions to complex lead smelting slags are given in Fig. 5a–d. The effect of the CaO/SiO_2 ratio on the liquidus temperatures of Pb–Zn–Fe–Al–Ca–Mg–Si–O slags for a given PbO concentration and Zn/Fe ratio in the slag is given in Fig. 5a. Here, it can be clearly seen that the slag is fully liquid below 1200 °C for the CaO/SiO_2 weight ratios between 0.1 and 0.6. The liquidus temperature at low flux additions in the spinel primary phase field corresponds to a Fe/SiO_2 ratio of approximately 0.33–0.37 for the above range of CaO/SiO_2 ratios. If the flux addition results in sub-liquidus phase assemblage at these operating conditions, the % solids in the slag can be estimated from Fig. 5c. Excessive % solids in the slag can lead a practical operating issues, such as difficulties in tapping due to increased slag viscosity and the loss of operating volume due to the build-up of solids on the reactor lining. The effect of variable Zn/Fe ratio in slag can be assessed through the use of a pseudo-ternary section ZnO–"FeO"–($CaO + SiO_2$), an example of which is shown in Fig. 5b. Here, the liquidus temperature is shown to vary with Zn/Fe ratio with the minimum liquidus temperatures occurring at the join between the zincite, (Zn, Fe) O, and spinel, $ZnO \cdot Fe_2O_3$, primary phase fields.

Process Modelling

Important principles of using thermodynamics in process modelling were outlined in recent publications. Reliable model predications are important for navigating the

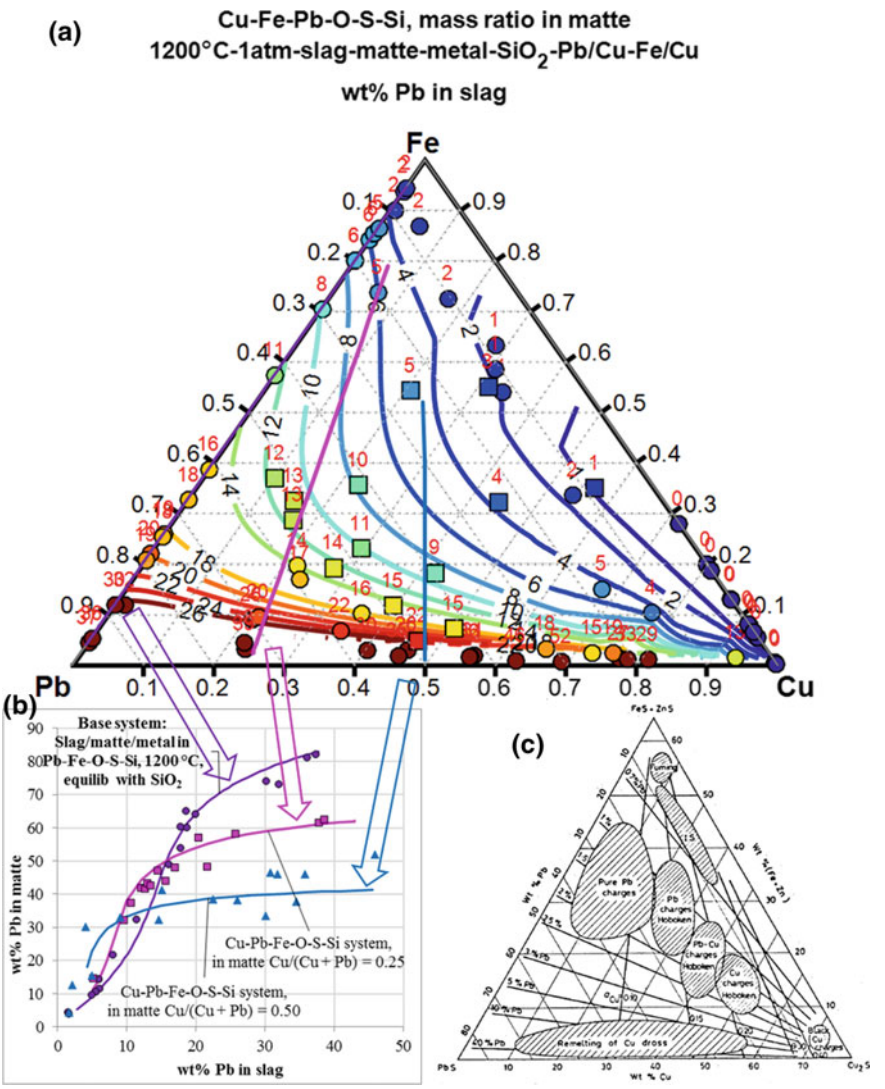

Fig. 4 Slag-matte-metal-SiO_2 equilibria in the Pb–Fe–O–S–Si–(Cu) system at 1200 °C. **a** Projection of the matte compositions on to the Cu–Fe–Pb composition triangle (mass fractions). Iso-lines and labels next to experimental points show %Pb in slag as a function of matte composition. **b** Three sections through the triangle showing the effect of Cu/(Cu + Pb) ratio in matte on the distribution of lead between slag and matte, and **c** the diagram from Fontainas et al. [12], which inspired the systematic study of slag-matte-metal equilibria. Lines in (**a**) and (**b**) represent FactSage calculations with current internal PYROSEARCH database. Symbols represent experimental data

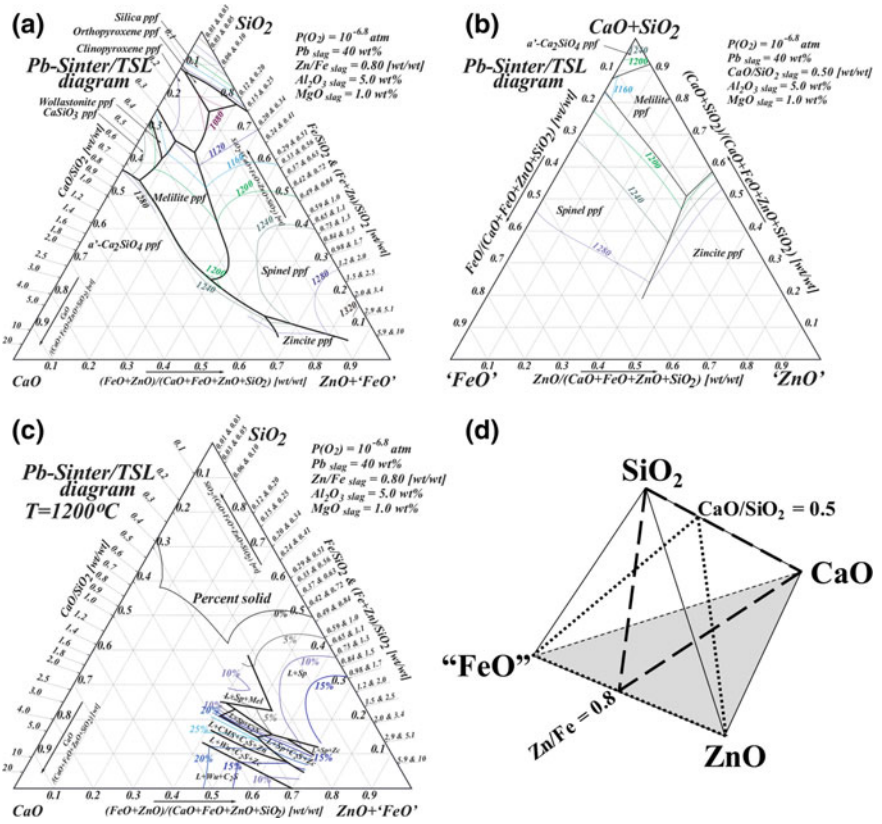

Fig. 5 Fluxing diagrams for multi-component lead slag smelting systems at fixed %PbO in slag. **a** The pseudo-ternary section (ZnO + "FeO")–CaO–SiO_2 showing the effect of CaO/SiO_2 on liquidus temperature for a given Zn/Fe ratio in feed, **b** pseudo-ternary section ZnO–"FeO"–(CaO + SiO_2) showing the variation of liquidus temperature with CaO/SiO_2 ratio in slag for a fixed Zn/Fe ratio, **c** % solids at a 1200 °C for the process conditions given in (**a**), and **d** pseudo-ternary sections of the ZnO–"FeO"–CaO–SiO_2 quaternary

complex reality of modern pyrometallurgical processes. Thermodynamics and phase equilibria are the foundation of sophisticated models. Earlier thermodynamic simulations of lead processes are using the simplified thermodynamic database available for copper drossing and the lead blast furnace.

The thermodynamic database developed by PYROSEARCH can be used together with software products, such as FactSage, ChemApp, ChemSheet, and SimuSage. Several examples of the use of thermodynamic modeling in copper systems are reported. Again full details of references will be provided in [3].

Concluding Statements

Advances in the research methodologies used in high-temperature phase equilibrium studies, improved experimental measurement techniques, and the integration of experimental and thermodynamic database studies have resulted in:

- significant increases in the ranges of process conditions temperature, oxygen and sulfur partial pressures, that can be characterized,
- considerable extension of the number and complexity of chemical systems that can be characterized, and
- improvements in the accuracy of the thermodynamic databases relevant to lead smelting and refining systems.

These fundamental studies have led to the development of sophisticated thermodynamic databases that can be used to predict the outcomes of high-temperature reactions. These databases are particularly important for the optimization of complex process flowsheets that contain multi-element, multi-phase separation systems. The development of these databases is particularly relevant to lead smelting and refining technologies, which are increasingly used as a means of recovering and recycling metals form the end of life of electrical and electronic devices. In these processes, elemental separation can be achieved through the controlled partitioning between gas—slag—matte—metal—speiss—solid phases.

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