



Evidence of sulfide melting and melt fractionation during amphibolite facies metamorphism of the Rajpura–Dariba polymetallic sulfide ores



Kamal Lochan Pruseth^a, Biswajit Mishra^{a,*}, Neshat Jehan^a, Bijay Kumar^b

^a Indian Institute of Technology, Kharagpur 721302, India

^b Geological Survey of India, Bangalore 560078, India

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ABSTRACT

Partial melting of sulfide ores during prograde metamorphism could have been more prevalent than generally accepted. However, identification of such melting is difficult as sulfide melts do not form glasses and the textures generated on quenching are obliterated due to the tendency of sulfides for ready recrystallization. The polymetallic base metal sulfide deposit at Rajpura–Dariba, Rajasthan, India is a typical stratiform ore metamorphosed to the middle amphibolite facies. The peak metamorphic temperature of 600 °C should have been sufficient to initiate sulfide melting as evident from experimental studies in the ZnS–PbS–Cu₂S–FeS₂–S system. Further, syn-metamorphic melting of the original SEDEX ore was abetted by the high f_{S_2} condition that prevailed as a consequence of barite dissolution. A Zn–Fe–S melt containing minor Pb, Sb and Cu but no Ag fractionated from an initial melt in the above system resulting in a residual immiscible sulfosalt-bearing PbS melt. The final metallic melts, represented by formation of dyscrasite (Ag₃Sb) from the sulfosalt-bearing melt and breithauptite (NiSb) or ullmannite (NiSbS) from the sulfosalt-absent melt, were a product of independent fractional crystallization of the immiscible sulfide and PbS–sulfosalt melts.

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1. Introduction

Sulfide anatexis in metamorphosed sulfide ore deposits is difficult to recognize as sulfide magmas do not quench to a glass and even if they do so, relatively faster solid state diffusion in sulfides at even room temperature may erase all signatures of melting. Indirect signatures of sulfide partial melting have been recorded at Broken Hill, Australia (Mavrogenes et al., 2001; Frost et al., 2002) and Hemlo, Canada (Tomkins et al., 2004), and its role as a potential ore genesis process has been recognized. Frost et al. (2002) and Tomkins et al. (2004) compiled a number of phase relations that point towards the possibility of sulfide melting at even middle amphibolite facies of metamorphism. Many sulfosalt phases are capable of melting in isolation at such temperatures (see the compilation of Tomkins et al., 2007), but the volume of sulfide melt produced by them will be significantly small. Eutectic compositions in assemblages containing the major sulfide phases such as pyrite, pyrrhotite, sphalerite, galena and chalcopyrite may have melting temperatures lower than that of the individual minerals in the assemblages. Generally, the presence of low melting chalcophile elements (LMCEs) such as As, Te, Sb, Bi and Ag helps further depress melting temperatures. For example, the eutectic temperature of 800 °C in the PbS–ZnS–FeS system was depressed by more than 28 °C at 1 atm by the addition of 1 wt.% of Ag₂S (Mavrogenes et al., 2001).

However, total pressure increases melting temperature at a rate of 6 °C/kbar. Wykes and Mavrogenes (2005) have experimentally shown that H₂O at 1.5 GPa can depress the PbS–FeS–ZnS eutectic to 865 °C from 900 °C and can be dissolved substantially in sulfide melts. However, at lower P, the effect of H₂O addition is not that spectacular. In the absence of textural evidence of melting it is convenient to work back from the observed sulfide bulk composition and check its possible state at the temperature and pressure of peak metamorphism inferred from silicate or sulfide thermobarometry. Bulk compositions of isolated pockets of sulfide minerals seem to represent sulfide melts in which the LMCEs are preferentially concentrated (Frost et al., 2002).

From observed mineral assemblages and reported P–T conditions of metamorphism from deposits all over the world, Frost et al. (2002) postulated sulfide partial melting at about 26 ore deposits, including that of Rajpura–Dariba. The polymetallic base metal sulfides of Rajpura–Dariba are hosted in a deformed volcano-sedimentary package of Proterozoic age, which has been regionally metamorphosed to the amphibolite facies. Two distinct parageneses of ore minerals represented by a stratiform ‘banded’ facies and a vein facies have been identified by Mishra and Mookherjee (1986, 1991) on the basis of field and textural observations. These two parageneses are interpreted as due to different mineralization events separated in time and space and so to be unrelated to each other. There is a strong possibility that the vein ore facies was remobilized from the banded facies by partial melting during peak metamorphism. However, stress-induced mechanical and fluid-assisted hydrothermal remobilization (cf., Tomkins, 2007) of the ore

* Corresponding author.

E-mail address: bmgg@iitkgp.ac.in (B. Mishra).

would have preceded or accompanied remobilization by partial melting. During prograde metamorphism, pyrite is a major source of free S as a consequence of desulfidation forming pyrrhotite. The resulting increase in f_{S_2} can trigger sulfide anatexis as discussed by Tomkins et al. (2007). Alternatively, dissolution of barite may also supply free S and cause melting of sulfides (Tomkins et al., 2004, 2006). Experimental studies at sulfur fugacities favoring the existence of pyrrhotite as a stable phase demonstrate that minimum temperature at which melting can occur is 800 °C in the FeS–PbS–ZnS system (Mavrogenes et al., 2001) and below 730 °C, if additionally chalcopyrite (CuFeS₂) is incorporated (Stevens et al., 2005). From the experimental study of sulfide melting and subsequent fractionation, Mavrogenes et al. (2013) identified three types of immiscible sulfide melts. These are: (i) true sulfide melts, which crystallize to form common sulfide phases (pyrite, pyrrhotite, chalcopyrite, galena, and sphalerite); (ii) sulfosalt melts containing significant amounts of Sb and As, apart from sulfur and crystallize to form sulfosalts or a mixture of sulfosalts and sulfides; and (iii) metallic melts that are devoid of sulfur and are dominated by low-melting metals such as Sb, As, Bi and Te, and crystallize as alloys. Pruseth et al. (2014) obtained sulfide partial melts at 600 °C in the system FeS–PbS–ZnS by incorporating Cu, under high f_{S_2} -conditions that stabilize pyrite instead of pyrrhotite. On the basis of available experimental studies and our new melting experiments, we have interpreted the different mineral assemblages encountered in the vein ore of Rajpura–Dariba to represent various stages during fractionation of a sulfide melt that evolved into two immiscible melts, which independently fractionated to produce metallic melts of two diverse compositions in the end.

2. Geological setting and background information

Poddar and Mathur (1963) and Raja Rao et al. (1972) were the first to describe the geology and mineralization in and around Rajpura–Dariba. According to Raja Rao (1976), the country rocks in the N–S to NE–SW trending Dariba–Sindesar Khurd–Bethumni belt occur as disconnected enclaves within the Banded Gneissic Complex (BGC) and are a part of the Bhilwara Group, which is thought to constitute the basement for the Aravalli sedimentation. Naha et al. (1987) furnished a geological map of the Rajpura–Dariba deposit and identified three stages of deformational structures. Deb et al. (1989) determined a model Pb-age of 1.8 Ga, which most likely indicates the depositional age of the stratiform ore. Recently, Hazarika et al. (2013) obtained two age clusters, i.e., at ca. 1.86 Ga and ca. 1.62 Ga by EPMA dating of monazites from the metapelites of Rajpura–Dariba; the latter having been assigned to the age of metamorphism of the host rocks along with the associated syn-sedimentary ores. Formation of the vein ores by mobilization of partial melts derived from the stratiform ores could have happened during this metamorphic event. On a local scale, calcareous biotite schist, calc-silicate rocks and garnetiferous mica schist containing staurolite or kyanite occur from the footwall to the hanging wall in the Rajpura–Dariba mine area (Deb and Pal, 2004; Mishra et al., 2006). Deb et al. (1978), on the basis of well preserved primary sedimentary structures, and Nair and Agarwal (1976), on the basis of a number of secondary deformational features in the ores, proposed a syn-sedimentary origin for the deposit that was later modified during metamorphism. Basu (1981) advocated that the ores were syngenetically formed due to emission of metal rich brines in ‘third order’ basins within an epicontinental sea and were later metamorphosed. A sulfosalt-rich vein ore metallization occurred towards the waning phases of metamorphism. The deposit is an example of biphase mineralization consisting of the major stratiform ore, which was metamorphosed and a minor sulfosalt-rich unmetamorphosed vein ore (Mishra and Mookherjee, 1986, 1991; Mishra, 2000). Mishra et al. (2006) studied the host and associated rocks surrounding the deposit and suggested a peak metamorphic P–T of 5.5 to 6.1 kbar and 580 to 600 °C, along a clockwise P–T path.

3. Experimental methodology and natural samples

All experiments were carried out using the evacuated quartz tube method (Scott, 1974) for which quartz tubes of 6 mm internal diameter and 1 mm wall thickness were used. In general, the quartz capsules had a length of 3 to 4 cm, with the charge confined to around 0.5 cm at the bottom and the top was occupied by a quartz rod of 6 mm diameter. High purity elemental Cu, Pb, Fe, Zn and S were utilized for synthesis of the starting sulfides Cu₂S, PbS, FeS and ZnS. For synthesizing galena small pieces of Pb metal were cut from a Pb foil following careful removal of the thin Pb-oxide coating with a stainless steel knife and washing with acetone. Fine powders of Zn and Cu were obtained by abrading zinc ingots and copper rods with the help of a jeweler's file. Copper filings were washed with dilute HCl and distilled water. Powders of Fe and Zn were reduced in hydrogen flow at 700 °C for 2 to 3 h. LENTON furnaces equipped with EURO THERM temperature controllers were used. The temperature in the hot zone of the furnace was maintained within ± 1 °C of the set temperature as monitored by an external Pt–Pt₉₀Rh₁₀ thermocouple. Melting experiments were performed at 600 °C and 760 °C, respectively for 15 and 8 days. The total quantity of the charge was limited to about 0.25 g of very finely powdered and well mixed starting compounds. The lack of evidence for textural disequilibrium, homogeneous compositions of different solid phases and the reproducibility of phase assemblages in the products of runs of different durations and different starting materials have been accepted to suggest attainment of equilibrium.

Samples of ores and host rocks were collected from the underground mining zones and their vicinities at different levels. Presently, active underground mining at Rajpura–Dariba is going on in parts of all the East-, North- and South-lodes. Mounted polished sections and polished thin sections were prepared for microscopic examination as well as for electron probe microanalysis.

4. Analytical conditions

Both natural ore/rock samples and experimental run products were analyzed by a CAMECA SX-100 electron probe microanalyzer (EPMA) and BSE imaging was performed with the help of a JEOL JSM6490 scanning electron microscope (SEM). Both the instruments are housed in the Department of Geology and Geophysics, IIT Kharagpur. The typical EPMA analytical conditions for silicates were 15 kV acceleration voltage, 20 nA probe current and ~ 1 μ m beam size. However, for sulfides, sulfosalts and other ore minerals an accelerating voltage of 20 kV was used. The following standards were used: Cu, Ni, Ag metals, pyrite for Fe and S, galena for Pb, sphalerite for Zn, stibnite for Sb, and GaAs for As. The raw EPMA data were matrix corrected with the PAP correction program by Pouchou and Pichoir (1984).

5. Evidence of sulfide melting

As seen from the compilation of Tomkins et al. (2007), there are ample possibilities of generating an anatectic sulfide melt at ≤ 600 °C, the peak metamorphic temperature attained at Rajpura–Dariba. Dilution by trace amounts of other elements in the major sulfide phases or phase systems may depress the melting temperatures of the individual phases or the eutectic compositions for the latter. However, an increase in sulfur fugacity, which can be brought about by various ways, seems to be the dominant cause of the decrease of melting points (Tomkins et al., 2007). Pyrite being the most abundant sulfide phase in the Earth's crust is the obvious candidate for the source of sulfur during prograde metamorphic heating, resulting in the formation of pyrrhotite, by the reaction $2\text{FeS}_2 = 2\text{FeS} + \text{S}_2$.

Barite is one of the possible phases that might have caused an elevated f_{S_2} condition during metamorphism; but till date, its presence at Rajpura–Dariba has only been speculated from the presence of high Ba in one of the bulk analyses given by Deb and Pal (2004). We document for the

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