Projeto de Pesquisa Científica e Tecnológica
CRA-175-02
RELATÓRIO TÉCNICO FINAL

Aplicação da radiação NIV (near infrared) no estudo genético (microscópico e microtermométrico) de minérios opacos de valor econômico e minerais opacos de valor gemológico de Minas Gerais

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Local onde foi desenvolvido o Projeto:
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Palavras chave: radiação NIV (near infrared); microscopia de infravermelho (MIV); gênese de minérios de ferro.

Sentença chave: Trata-se da utilização (para metalogênese e prospecção de minério de ferro) de uma metodologia de trabalho inédita no âmbito das Geociências, tanto no Brasil quanto na América do Sul.

INTRODUÇÃO.

Os estudos genéticos (petrográficos e inclusões fluidas) de minerais que constituem o minério da jazidas, são considerados de grande importância para o desenvolvimento de quaisquer setor mineral. Através desses estudos é possível determinar a origem do minério, caracteriza-lo e, finalmente, propor modelos de prospecção.

Entretanto, a boa transparência dos minerais constitui um dos grandes limites da petrografia convencional: minérios opacos (a exemplo do Fe e Au) não podem ser estudados utilizando a microscopia de luz visível pois comportam-se como minerais escuros sob luz visível (0,35-0,75μm), o que impossibilita seu estudo.

Nos últimos 15 anos, apenas cinco laboratórios do hemisfério norte conseguiram estudar, com sucesso, feições internas e inclusões fluidas em minerais notadamente escosos
(opacos) utilizando radiação NIV (microscópios e câmeras de infravermelho que permitem operar na faixa de comprimento de onda entre 0.75 e 2.2 μm). (Campbell *et al.* 1984 e 1987, Richards & Kerrich 1993, Lüders 1996, Lüders e Ziemann 1999, Bailly *et al.* 2000). Quando submetidos à incidência da radiação infravermelha, esses minerais opacos tornam-se transparentes sendo possível observar (através de monitores de alta definição) zoneamentos, linhas de crescimento e inclusões sólidas e fluidas.

Os estudos petrográficos e microtermométricos nesses minerais opacos são de grande importância em estudos genéticos de jazidas minerais já que, geralmente, constituem o próprio minério dos depósitos. Os primeiros testes de microscopia de infravermelho (MIV), utilizando radiação NIV no Brasil foram desenvolvidos com sucesso no Laboratório de Incluções Fluidas e Metalogênese (LIFM) da CNEN-CDTN-BH entre 1999 e 2001 (Ríos *et al.*, 2000).

O projeto FAPEMIG CRA-175 forneceu dois equipamentos (câmera de infravermelho e objetiva de longa distância) que foram adquiridos com o intuito de aprimorar os resultados obtidos em vários minerais opacos. Inicialmente foram estudadas diversas gerações de hematitas das formações ferríferas do Quadrilátero Ferrífero -MG, turmalinas e granadas escuras; e, posteriormente, nióbio-tantalatos, esfaleritas ferrosas, etc.

A continuidade no aprimoramento dessa metodologia de trabalho permitirá, a curto prazo, estender as pesquisas para minérios opacos radioativos, a exemplo dos existentes no setor de Lagoa Real (BA).

**CONTEXTO GEOLOGÍCO**

As amostras de hematita estudadas correspondem ao Grupo Itabira do Quadrilátero Ferrífero de MG. De acordo com Dorr (1969), o Grupo Itabira está dividido nas Formações Cauê (base, com formações ferríferas - BIF) e Gandarela (topo, com dolomitos e mármore). Mudanças químicas e mineralógicas durante a deposição (relacionadas a mudanças de fácies na bacia) originaram variações nos tipos de BIF

**OBJETIVOS e RESULTADOS OBTIDOS**

Através desse projeto:

a) foi desenvolvido, e colocado em rotina, o segundo estágio do sistema de microscopia de infravermelho do CDTN (Belo Horizonte). Para isto foram adquiridos, com os fundos do Projeto CRA-175, uma câmera de infravermelho Modelo Eletrophysics e uma objetiva de longa distância 50X da Leica.

b) foram estudados os fluidos responsáveis pela formação das hematitas e os corpos de minérios de ferro de alto teor associados as formações ferríferas bandadas do Grupo Itabira e a real extensão de sua influência na geração dos diferentes tipos de minérios de alto teor, formados sob condições distintas de metamorfismo e deformação.

Para isso, foram feitos estudos de inclusões fluidas comparativos entre diversas gerações de hematitas provenientes de Mina da Conceição (Itabira), buscando a comparação entre os fluidos que as originaram.

c) foram feitos testes de microscopia de infravermelho em amostras da Mina do Pico (QF, MG) visando sugerir propostas para o problema da crepitação do minério de ferro (hematitas). Em forma preliminar, foi possível diferenciar as fases que crepitam das não crepitantes, através da resposta que os minerais oferecem sob luz infravermelha.
d) foram elaborados e publicados os seguintes resumos expandidos e abstracts em congressos Nacionais e Internacionais:

**Simpósio Brasileiro de Exploração Mineral, Ouro Preto, 2004**

Resultado preliminares do estudo metalogenético do minério hematítico de alto teor da Mina de Ferro N5-Carajás, utilizando microscopia e microtermometria de infravermelho

**Timing of hydrothermal iron mineralization, Carajás Province**

**32 International Geological Congress, Florence, Italy, 2004**

*The origin of hematite in high grade iron ores based on infrared microscopy and fluid inclusion studies*

XII Simpósio de Geologia de Minas Gerais

**Geocongress 2002 & 11th Quadrennial IAGOD Symposium, Namíbia, 2002**

Deformation controlled high grade iron ores

**Infrared microscopy and fluid inclusions analysis in hematite from high grade iron ore. Genetic considerations**

XII Simpósio Geologia de Minas Gerais, Ouro Preto, 2003

*The origin of hematite in high grade iron ores based on infrared microscopy and fluid inclusion studies*

e) foi elaborado e publicado o seguinte artigo

**Revista Economic Geology**

The origin of hematite in high grade iron ores based on infrared microscopy and fluid inclusion studies: The example of the Conceição Mine, MG, Brazil

f) Foi orientada e introduzia na metodologia de Microscopia de Infravermelho a aluna de Iniciação Científica Wherineia Cotta Dias
DISCRIMINAÇÃO DOS RESULTADOS OBTIDOS

A) SISTEMA DE MICROSCOPIA E MICROTERMOMETRIA DE INFRARVERMELHO

ABSORÇÃO IR EM MINERAIS OPACOS Os minerais escuros (sob luz transmitida) comportam-se como opacos em decorrência da absorção de fotons durante as transições eletrônicas nos orbitais moleculares. Assim, a opacidade de vários sulfetos, arseniatos, etc, com relação a luz visível, é devida a transições band-gap (Richards e Kerrich 1993). O band gap, num mineral semiconductor, é a energia mínima necessária para excitar um elétron desde o estado (atômico) ligado na rede (a banda de valência) até um estado livre (deslocado), no qual o elétron torna-se livre para se mover (Campbell et al. 1984). Entretanto, quando a energia dos fotons é menor do que a mínima requerida para essa transição eletrônica a luz não é absorvida e o mineral comporta-se como transparente nesse comprimento de onda (Richards e Kerrich 1993).

O valor mínimo de energia para absorção (band gap energy) é atingido, para a maior parte dos sulfetos e sulfosais, na faixa do infravermelho (Burns, in Campbell et al. 1984), sendo que a radiação NIV possui maior comprimento de onda, e menor energia, do que a luz visível (Mancano e Campbell 1995).

Portanto, é necessário utilizar uma energia menor que a do gap (no caso luz NIV) para que o mineral opaco se comporte como transparente. Entretanto, a substituição dentro da estrutura cristalina por impurezas de valência contrastante muda a configuração eletrônica do mineral, diminuindo a energia do band gap, resultando num degrau da transparência (Richards e Kerrich 1993).

Como conseqüência da não disponibilidade no Brasil de um microscópio petrográfico de infravermelho foi montado no LIF do CDTN (adaptando equipamentos do laboratório e com apoio da FAPEMIG nProjeto CEX-175-2002) um sistema que permite operar no início do espetro do infravermelho (0,75-1,2μm). Para isso, foi necessário construir uma fonte emissor, para ser acoplada num microscópio Leica DMRXP. O sistema consiste num resistor, acoplados a fontes de IR, que opera com corrente variável e possui alta
sensibilidade. Como fontes de iluminação foram testados dois tipos diferentes de laser leds (convencionais e da Laser Monitoring (λ entre 0.7 e 1.8 μm). A resposta dos leds é boa para wolframita e bastante limitada para hematitas.

Tentou-se, então, com lâmpadas de infravermelho extraídas de espectrômetros IR de última geração. Trata-se da fonte modelo Hosobuchi, de 50W (12V), que abrange boa parte do espectro de infravermelho. Foi a melhor opção encontrada para estudos microtermométricos, onde é necessária maior potência de iluminação. Também foram testados *bulbs* halogênicos de 100W (12V), que forneceram bons resultados no início do espectro de infravermelho. Foram feitas, ainda, tentativas com *elementos IR-cerâmicos", da Oriel INC, que atingem temperaturas superiores de 700°C, fato que constituiu um sério problema durante os experimentos. Não obtivemos resultados com essa fonte, portanto não foi utilizada nos testes subsequentes.

Foto 01. Emissor de IR construído no CDTN. Na foto é possível ver dois tipos de fontes de infravermelho utilizadas: o elemento cerâmico de alta temperatura (protegido com uma cápsula vermelha) e a fonte Hosobuchi.
Foto 2. Fonte IR Hosobuchi sendo testada

Foto 3. Elemento de IR Cerâmico da Oriel, sendo testado
Foto 4. Laser Leds 1,8 (da Laser Monitoring) sendo testado

Foto 5. Fonte de infravermelho acoplada no setor inferior (abertura para luz transmitida) de microscópio Leica DRMXP (Projeto FAPEMIG 787/92)
Para visualizar as imagens de IV foram utilizadas três tipos de câmeras.
Inicialmente, foi testada uma câmera de alta resolução modelo CC-IR da Computar-Japan, que opera na faixa de λ que abrange desde a luz visível até 1,2 μm.
Posteriormente, foi testada uma câmera Exwavehad SONY, que opera na faixa near infrared e forneceu excelentes resultados com minério de ferro (hematitas).
Finalmente, foi testada uma câmera ultrasensível Eletrophysics 7290A, adquirida com o Projeto FAPEMIG CRA175-02. Trata-se duma câmera analógica. É relativamente lenta, se comparada com a Exwavehad. Entretanto, é bem mais sensível para operar em frequências maiores de 1,5nm. O tubo fotosensível é muito delicado e não permite estudar amostras onde há mistura de minerais transparentes. A transmissão de luz normal através de minerais transparentes pode danificar o tubo.

Foto 6. Câmera Eletrophysics acoplada num microscópio Leica DRMXP.
Foto 7 Exemplo de amostras estudadas (lâmina polida de hematita) e objetiva 50X longa distância adquirida com o Projeto FAPEMIG 787/92

As imagens de infravermelho são observadas através dum monitor acoplado a video printer da Sony Trinitron (Projeto FAPEMIG 787/92).

Foto 8. Vista geral do sistema de microscopia de infravermelho adaptado num microscópio LEICA
Para os estudos microtermométricos com luz infravermelha, foi utilizada uma platina US Fluid Inc. Foram feitos testes com uma platina Chaixmeca, porém, devido a sua particular constituição, a mesma não permitiu desenvolver estudos utilizando a metodologia MIV.

Dessa forma foi possível estudar, pela primeira vez no Brasil e na América Latina, feições e inclusões fluidas em minerais opacos tais como hematita, wolframita e variedades opacas de esfalerita. A viabilidade do sistema foi confirmada por especialistas do US Geological Survey, Japan Geological Survey e University of Western Australia (dentre outros).

B) ESTUDOS DE MIV NA MINA DE FERRO DE CONCEIÇÃO (QF, MG)
Foi desenvolvido um estudo aprofundado sobre a metalogênesse do minério de ferro na Mina de Conceição (MG). Os resultados foram publicados na Revista Economic Geology.

A seguir, o texto completo do artigo
THE ORIGIN OF HEMATITE IN HIGH-GRADE IRON ORES BASED ON INFRARED MICROSCOPY AND FLUID INCLUSION STUDIES: THE EXAMPLE OF THE CONCEIÇÃO MINE, QUADRILÁTERO FERRÍFERO, BRAZIL

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Abstract

Petrographic and textural analysis combined with fluid inclusion studies by infrared microscopy of high-grade (>65% Fe) hematite ore samples from the Conceição deposit, in the northeastern part of the Quadrilátero Ferrífero, Brazil, indicate a complex process of oxidation and mineralization during two orogenic events, each developed under different conditions and involving distinct fluids. The earliest mineralization formed massive magnetite-rich orebodies under relatively reducing conditions in the early stages of the Transamazonian orogeny. Magnetite was oxidized (martitized) with the development of porous hematite crystals (hematite I). Possibly during this stage, new hematite crystals were also formed from low-temperature, low- to medium-salinity fluids, as indicated by two-phase fluid inclusions. The origin of these fluids is still uncertain but tentatively interpreted as being modified surface water. The fluids were transported along normal faults and fractures during post-tectonic collapse following the Transamazonian orogeny (2.1–2.0 Ga) and creation of the dome-and-keel structural pattern of the Quadrilátero Ferrífero. These solutions were also likely responsible for the initial oxidation of the iron formations and the development of hematite I. Subsequent uplifted hot basement rocks or post-tectonic plutons were probable heat sources for the regional metamorphism and development of a granoblastic fabric of hematite II grains in the iron formations and high-grade orebodies. However, the ore was only partially recrystallized; as several relics of the early magnetite, martite, and hematite are still preserved in the granular hematite II crystals. During the Brasiliano-Pan-African orogeny (0.8–0.6 Ga), high-salinity fluids, with temperatures varying from ~120°C to a maximum of approximately 350°C, penetrated the iron formations along shear zones, crystallizing initially tabular and thereafter play hematite crystals (hematite III and specularite) forming schistose orebodies. Quartz veins that cut across the ore and envelop specularite plates and ore fragments formed from late-stage, high-temperature, and low-salinity fluids containing CO₂. These later fluids did not alter the ore.

Each of these stages of mineralization produced orebodies with distinct features. Recurrent hydrothermal mineralization is thought to have been responsible for the development of giant, high-grade iron ore deposits in structurally favorable sites. Fold hinges with enhanced permeability and deep faults able to conduct the fluids to the surface, repeatedly over time, should be important targets for exploration of new resources.

Introduction

The economically important, giant iron ore deposits of the Quadrilátero Ferrífero district, Minas Gerais, southeastern Brazil, are host to metamorphosed iron formations, referred to as limestones, of the Paleoproterozoic Itabira Group. Concordant and discordant, hard, massive orebodies consisting of magnetite martite and hematite occur together with structurally controlled, schistose, high-grade bodies and with friable weathered ores. The range of different types of iron orebodies has led to considerable debate about their origins. Two end-member genetic models have been proposed: supergene versus hydrothermal enrichment of a banded iron formation protore. In the case of the Hamersley and Animikie basins, several authors have suggested that hypogene enrichment by metasomatic replacement was responsible for mineralization (Gruener, 1924, 1930, 1937; Morey, 1999; Powell et al., 1999). Guild (1953, 1957) and Doré (1965, 1969) postulated a similar origin for the Brazilian ores from the Quadrilátero Ferrífero. Taylor et al. (2001) provided an alternative interpretation for the role of hydrothermal fluids, whereby residual enrichment through SiO₂ leaching was the principal mechanism for upgrading the ore. The importance of deformation in the concentration of hematite was also emphasized by Taylor et al. (2001) who established that the deposits from the Hamersley province are associated with medium-scale, normal faults that act as channelways for the mineralizing fluids. Morey (1999) and Powell et al. (1999) proposed a compressional setting for the mineralization in the Hamersley province and Mesabi Range with synorogenic fluids driven by a regional tectonic gradient. Guild (1933, 1957) and Cuba (1982) considered that the high-grade orebodies of Minas Gerais formed by iron remobilization, controlled by shear zones and hinges of meter-scale folds. Leith (1933), Van Hise and Leith (1911), Leith et al. (1935), and Taylor et al. (2001) all considered that supergene enrichment by descending meteoric solutions was an important mineralizing process for the high-grade bodies in each of these districts.

It is likely that these different processes represent steps in a complex succession leading to the present shape and appearance of the different orebodies, as proposed by Taylor et al. (2001). In a first attempt to establish the nature and composition of the fluids involved and the conditions that
prevalled during oxidation and mineralization of the banded iron formation proteros in the Quadrilátero Ferrifero, textural analysis together with microthermometric studies of different generations of hematite and specularite were conducted on samples from the Conceição deposit, located in the northeasternmost part of the district. Previous detailed petrographic and microstructural analyses, leading to the identification of several distinct hematite and specularite generations, are used as a basis for this study (Rosière, 1981; Rosière and Chemnitz, 1991; Rosière et al., 2001).

The Quadrilátero Ferrifero District

The Quadrilátero Ferrifero is located at the southern border of the São Francisco craton, a geotectonic unit of Brasiliano age (0.8–0.6 Ga), surrounded by the converging Arãçu, Brasilia, Rio Fretó, Riacho do Pontal, and Sergipano orogenic belts (Dorr, 1969; Almeida, 1977; Schobbenhaus et al., 1984). The structure of the Quadrilátero Ferrifero (Fig. 1a) is defined by a roughly rectangular arrangement of synclines, with Paleo-Proterozoic metasedimentary rocks of the Minas Supergroup separated by antiformal structures dominated by Archean greenstones of the Rio das Velhas Supergroup and domes of Archean and Proterozoic crystalline rocks (Machado and Cardoso, 1992; Machado et al., 1992; Noce, 1995). The youngest ages for basement rocks are 2612 ± 5 Ma (Noce, 1995) and 2593 ± 10 Ma (Romano, 1989), for the Sato do Parapeba and Florya metavolcanic rocks, respectively. The Minas Supergroup comprises, from bottom to top, the Caraca, Itabira, Piracica, and Sabará Groups (Dorr, 1969). The thickest sequence of iron formations together with enclosing high-grade iron orebodies belong to the Itabira Group, comprising itabirite, dolomite, and subordinate metapelitic units. Carbonate rocks of the upper Itabira Group, which contain algal remnants, have been dated by Babinski et al. (1995) at 2419 ± 13 Ma (Pb-Pb isochron data). Carbonate rocks of the Piracica Group, in contrast, yielded Pb-Pb ages of 2050 ± 230 Ma, which are interpreted to be metamorphic ages (Babinski et al., 1995). The Sabará Group comprises a 3.0- to 3.5 km-thick sequence of metavolcaniclastic rocks, turbidites, and conglomerates, separated by an unconformity from the underlying Piracica Group. Based on U-Pb dating of detrital zircons, Machado et al. (1989, 1992) postulated a depositional age of 2125 Ma.

The regional structure is the result of the superposition of two main deformation events (Chemnitz, Jr. et al., 1994). The first produced the nucleation of regional synclines in the supracrustal sequence, upfolding of the gneissic domes during the Transamazonian orogeny (2.1–2.0 Ga), and the regional metamorphism. The second was related to a west-verging thrust belt of Brasiliano/Pan-African age (0.8–0.6 Ga); tectonic transport arrows, Fig. 1a). The latter event, which was more dramatic in the eastern half of the Quadrilátero Ferrifero, deformed the earlier structures and was responsible for the deformation gradient present in the area and possibly for the metamorphic gradient determined by Pierce (1993). Two main structural domains (Rosière et al., 2001) can be delimited regionally (Fig. 1). The eastern high-strain domain includes regional thrust systems and shear zones that may be several hundred meter wide, whereas the western low-strain domain displays well-preserved megasynclines that are discontinuously cut by discrete shear zones and faults. The iron formation of the Itabira Group delineates the main structures of the entire Quadrilátero Ferrifero as a regional marker (Fig. 1b), and high-grade iron deposits occur along the limbs of the megasynclines as well as in shear zones in the eastern high-strain domain.

The main regional thermal metamorphic event in the Quadrilátero Ferrifero is related to the late extensional phase of the Transamazonian event and predates the pervasive foliation developed during the Brasiliano orogenesis and the formation of specularite (Chemnitz, Jr. et al., 1994; Alkmim and Maranhão, 1998; Rosière et al., 2001). Regional metamorphic zoning is defined by the presence of chlorite, biotite, and staurolite (Herz, 1978), with metamorphic grade increasing eastward, and the zoning roughly follows the deformation gradient. Pierce (1993) redefined the zones of gneissette (CZ), cummingtonite (CZ), actinolite (AZ), and tremolite-actinolite (TZA) (Fig. 1b) based on the metamorphic mineralogy of the iron formations of the Itabira Group. Thermal aureoles surrounding the domes, however, interfere with this zoning and produce a local higher grade metamorphic overprint (Herz, 1978; Maranhão et al., 1992).

Itabirites and Iron Ores of the Itabira Group

The Itabira Group is a sequence of predominantly chemical, sedimentary rocks deposited in a platform marine environment and is composed of two formations (Dorr, 1969): the Caçu Formation, which comprises a thick sequence (ca. 250–300 m) of iron formations (itabirites and hematitic bodies) intercalated with hematitic phyllites, dolomitic phyllites and marbles; and the Gandarela Formation, which conformably overlies the Caçu Formation and is composed of carbonate rocks (calcitic and dolomitic marbles) with subordinate phyllites and banded iron formation.

The name itabirite is used to describe metamorphosed, oxidized, and heterogeneously deformed banded iron formations (Dorr, 1969). There are several distinct mineralogical and textural types due to variation in the original composition of the sediments, intensity of deformation, and degree of metamorphism and hydrothermal alteration.

Hard, high-grade iron ore occurs both as massive and as schistose bodies within the itabirite. The shape of the massive orebodies is totally or partially controlled by the bedding of the banded iron formation protore, and the granoblastic fabric commonly mimics the original structure. Irregular pockets of high-grade iron ore with a breciated fabric may also occur but are not as common. Schistose orebodies occupy shear zones that crosscut the banded iron formation.

Mineralogy of the Iron Oxides in the Quadrilátero Ferrifero

Microcrystalline hematite (primary hematite) and other minerals, typical of very low grade metamorphic banded iron formations such as in the Hamersley Range (Trendall and Blackley, 1970) or in the Transvaal/Griqualand regions (Beukes, 1984) have not been observed in the itabirites of the Quadrilátero Ferrifero. Magnetite both in the itabirite and high-grade orebodies is typically and typically kummelnorthemite (Kullerud et al., 1969; Morris, 1980; Rosière, 1981), an Fe²⁺-deficient variety, which is found ubiquitously in low-strain portions of the
or, mainly in the western domain of the Quadrilátero Ferrífero, and is interpreted to be the oldest iron oxide of the assemblage. Magnetite normally occurs as relics in mafite (hematite pseudomorphous after magnetite) and in hematite aggregates or even by itself in some bodies. The high abundance of magnetite relics indicates that it was the main iron oxide of iron formation and high grade ores in the Itabira Group, prior to the oxidation that affected the entire Caué Formation and resulted in rocks containing predominantly hematite (Rosière, 1981).
In the western low-strain domain of the Quadrilátero Ferrífero, hematite occurs mainly as subhedral to euhedral granular crystals (0.01–0.2 mm) and, together with anhedral grains, has an overall granoblastic fabric. This fabric is considered to represent post-tectonic partial recrystallization (Rosière 1981; Rosière et al., 2001).

In shear zones, which are more pervasive in the eastern high-strain domain, hematite commonly occurs as euhedral, tabular-shaped or very elongated specularite platelets, which commonly overgrow relics of granoblastic aggregates (similar to those found in the western domain) or occur in strain shadow microdomains. Specularite platelets have a preferred orientation, and define a schistosity of variable intensity and penetration with the development of a wide variety of deformational and metamorphic fabrics (Rosière et al., 2001).

The Conceição Iron Ore Deposit

The Conceição deposit is the southernmost of the Itabira Complex in the northeastern part of the Quadrilátero Ferrífero (Fig. 1a). The mine is operated as an open pit by the Companhia Vale do Rio Doce (CVRD) and is located in the re-folded hinge zone of the Conceição syncline, one of the folds that compose the Itabir or synclinorium, which is the main regional structure in the area (Fig. 1a). The rocks in the northeastern extremity of the Quadrilátero Ferrífero experienced high strain during the Brasiliano orogeny (Chennault, Jr. et al., 1994) with the development of shear zones, tight folds, and partial transposition of the primary bedding of the Itabirite. In the Conceição syncline two main schistosities developed. These are S1, which developed during the first-generation folding F1, and S2, which is axial planar to the second-generation folds F2 (Chennault et al., 1997, Rosière et al., 1997). The latter schistosity is related to northeast-trending transpression, during which thrust sheets interacted with the 1,670 ± 32 Ma Borachados granite intrusive body (Chennault et al., 1994; Chennault et al., 1997). In the Conceição deposit, F1 and F2 folds are coaxial, plunging 40 to N 60 E, and the macroscopic structure represents a classical type 3 interference pattern of Ramsay (1962; Fig. 2).

**Fig. 2.** (a), Simplified geologic map of the Conceição iron ore deposit, showing the location of CO260 diamond drill hole, based on data furnished by the Companhia Vale do Rio Doce as well our own observations and mapping. (b), Schematic representation of cross section SVCE21, showing the location of analyzed samples in the diamond drill hole CO260. Map and section are referenced to mine internal coordinate system. (c), Schematic drawing of the analyzed core sample.
Metamorphic mineral assemblages in the rocks of the Minas Supergroup surrounding the Conceição deposit indicate intermediate-grade metamorphic conditions between upper greenschist and lower amphibolite facies, with temperatures of about 450°C (Dorr and Barbosa, 1965; Schoroscher, 1975; Herz, 1978; Chemale, 1987; Pires, 1993). Schoroscher (1975) placed the staurolite isograd at 4 km northeast of the Itabira district, whereas Pires (1995) located it just west of the Conceição deposit (Fig. 1b). Oxygen isotope studies of the iron ores and itabirites in the area (e.g., Hoefs et al., 1982; Müller et al., 1986) indicate even higher metamorphic temperatures with sharp local variations (408°-660°C).

**Analyzed Samples and Petrographic Characteristics under Reflected Light**

In order to study the origin of the fluids related to the oxidation and enrichment processes in itabirites and the high-grade orebodies, infrared microscopic analysis and fluid inclusion studies were carried out on doubly polished thin sections. These were prepared from samples of hard ore and quartz veins collected from the diamond drill core CO260 located at the southern part of the Conceição deposit (hole CO260, Fig. 2). The sampled section extends from the lower contact zone between the moderately deformed high-grade massive orebody and itabirite protore and is cut across by discrete microshear zones.

The samples were grouped into three types: samples of massive ore, samples from a shear zone cutting the massive ore, and samples from a quartz vein.

The massive high-grade ore consists of hematite grains with variable characteristics (Table 1) and martite aggregates with magnetite relics. These samples exhibit magnetite-martite textural relationships similar to the ores from other Brazilian deposits located in the western low-strain domain (Hacksparser, 1979; Rosière, 1981; Rosière and Chemale, 1991), as well as in some Australian ores described by Morris (1980, 1983). Martite is developed along the (111) planes of magnetite or irregularly from the grain boundaries inward, resulting in a complex patchwork of anhedral porous hematite with magnetite relics and tiny inclusions with lower reflectivity under normal reflected light (hematite I, Fig. 3a). Most hematite grains, however, occur as nearly isometric granular crystals, usually <200 μm (hematite II, Fig. 3a-b, Table I). They have curved to straight grain boundaries and are intergrown with hematite I, which appears as inclusion-bearing porous microdomains.

In the shear zone that cuts the massive ore two types of hematite occur: euhedral to subhedral tabular hematite crystals (hematite III, Fig. 3c-d, Table I), which are dominant in microscopic dilational zones or occupying interstices between hematite II aggregates; and thin specularite platelets (Fig. 3e, Table I) up to 500 μm long, oriented parallel or subparallel to the sharp boundaries of the shear.

The quartz vein is comprised of saccharoidal crystals (1-2 mm diam) that form a mosaic fabric enveloping ore fragments and distorted single crystals or aggregates of specularite. The vein has irregular borders and partially invades the ore along its banding and in the hinge zones of microfolds (Fig. 3f-h).

**Analytical Procedures and Experimental Conditions**

The use of infrared microscopy in the study of fluid inclusions in opaque minerals was first described by Campbell et al. (1984) and Campbell and Robinson Cook (1987). The methodology is described in Campbell et al. (1984), Richards and Kerrich (1993), Mancano and Campbell (1995), and Lüders and Ziemann (1999). The first application of infrared microscopy and microthermometry to the study of hematite was documented by Lüders et al. (1999).

Fluid inclusions and textural analyses in this study were conducted at the Fluid Inclusion Laboratory (IFLM) of the

<table>
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<tr>
<th>Mineral</th>
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<th>Fluid inclusions</th>
<th>Fabric</th>
<th>Distribution in sample CO200</th>
</tr>
</thead>
<tbody>
<tr>
<td>Magnetite</td>
<td>Irregularly shaped relics in hematite aggregates; intense martilization</td>
<td>Not transparent under infrared radiation</td>
<td>Originally as single idiomorphic or as massive aggregates</td>
<td>Irregularly dispersed</td>
</tr>
<tr>
<td>Hematite I (Hm I)</td>
<td>Porous anhedral crystals; lobate to serrated grain boundaries</td>
<td>Numerous tiny (&lt;1 μm), indistinct inclusions (solid inclusions or irregularities?)</td>
<td>Irregular to amoeboid, granoblastic fabric</td>
<td>Patches in the massive ore</td>
</tr>
<tr>
<td>Hematite II (Hm II)</td>
<td>Subhedral, nearly isometric, granular</td>
<td>(FI-1) generally spherical to elongated, two-phase; (FI-2) surrounded to tear drop-shaped, single phase with probable neck-down features</td>
<td>Polyneph, granoblastic fabric</td>
<td>Main component of the massive ore</td>
</tr>
<tr>
<td>Hematite II-III (Hm II-III)</td>
<td>Subhedral hybrid shapes (granular-tabular)</td>
<td>Two-phase, regular, partially elongated, restricted to the granular domain</td>
<td>Rare; together with Hm III</td>
<td>Interstitial in dilational zones</td>
</tr>
<tr>
<td>Hematite III (Hm III)</td>
<td>Euhedral to subhedral, tabular</td>
<td>Small, two-phase, aligned on the basal plane</td>
<td>Interstitial in the granoblastic fabric</td>
<td>Interstitial in dilational zones</td>
</tr>
<tr>
<td>Specularite</td>
<td>Plate</td>
<td>Two, three, and multiphase inclusions, negative crystal shape</td>
<td>Strong preferred orientation</td>
<td>Shear zone-hosted</td>
</tr>
</tbody>
</table>

**Table 1. Main Characteristics of the Iron Oxide Generations from the High Grade Ore**
Fig. 3. Microscopic features of the analyzed ore types. (a). Porous microfacies of massive ore comprising crystals of anhedral porous hematite I (Hm I) and granular hematite II (Hm II) surrounding and enclosing marlite with magnetite relic (Mt) (reflected light, partially crossed polarizers). (b). Same as (a) under infrared radiation. The original shape of incomplete martitized magnetite is recognized as the opaque mineral (Mt). Complete developed hematite crystals are transparent. (c). Aggregate of hematite II-III and hematite III. Hematite-I-III is characterized by a tabular syntactic overgrowth on granular hematite. Reticles of low-temperature and low-salinity fluid inclusions are depicted in the inset, enlarged in Figure 4d (infrared radiation). (d). Tubular crystal of hematite III (Hm III) with fluid inclusions (FI) aligned on the basal plane (001) (infrared radiation). (e). Microshear zone with oriented specularite crystals cutting aggregate of hematite II grains (Hm II). Inset depicts microfolded domain enlarged in Figure 3f (reflected light, partially crossed polarizers). (f). Enlargement of inset from (e), showing twinning lamellae in specularite plates and quartz (qtz) in the hinge zone of the microfold (infrared radiation). (g-1). Detail of microfold hinge zone. (g-2). Enlargement of inset from g-1, showing specularite plates enveloped by vein quartz (qtz) (plane reflected light). (h). Quartz (qtz) from vein domain envelops single plates and specularite aggregates (1a and 1b in plane reflected light; 2a and 2b in cross-polarized transmitted light).
Centro de Desenvolvimento de Tecnologia Nuclear (CDTN), Belo Horizonte, Brazil, using a Fluid Inc. heating/freezing system stage adapted to a Leica DMR-XP microscope. For calibration and precision tests commercially available synthetic standards were used. The microscope is equipped with a 100-W (12-V) transmitted light illuminator, using halogen light bulbs as a source of visible and very near infrared radiation. The doubly polished sections studied were about 80 to 100 μm thick. The hematite crystals generally exhibit a good transparency in the very near infrared spectrum.

A Sony ExwaveHad black and white, very near-infrared camera and an Electrophysics monochrome model 7290-A IR camera, both coupled to a high-resolution monitor with a video graphic printer, provided the infrared images (Rios et al., 2000).

For the observation of thicker hematite sections, when the transparency under the regular halogen lamp was unsatisfactory, an infrared emission regulator with a 50-W 12-V Hosobuchi iodine-tungsten lamp was used. The infrared beam between the lamp bulb and the sample covered an overall distance of 40 cm and a maximum voltage of 5 V was applied during the measurements in order to avoid overheating of the sample.

During infrared analysis under room temperature (25°C) condition, no temperature variation was registered in the sample, even in time intervals longer than 1 hr between measurements, inferring that the heat from the infrared source did not influence the thermometric determinations. At temperatures higher than 300°C, a quick decline in the transparency of the hematite crystals could be observed.

Infrared microscopy of different hematite types

Infrared microscopy permits the observation of several remarkable features usually unseen under normal light. Under near-infrared light, martite appears entirely opaque, and idiomorphic black magnetite ghosts (Mt, Fig. 3b) can be observed surrounded by hematite aggregates. This striking effect enables the recognition of the original shape and size of the magnetite crystals, despite oxidation that has almost totally obliterated the early fabric as seen under normal reflected light.

Under infrared light, it was also possible to recognize that some of the tabular hematite III crystals overgrow hematite II relics. The latter preserve two-phase fluid inclusions of the FI-I type as described in the next section. This tabular hematite was classified separately as hematite II-III (Hm II-III, Fig. 3c). Under infrared light, oriented specularite crystals display twinning lamellae (growth lamellae?) crisscrossing the plates (Fig. 3f). These features were not visible under polarized reflected light.

Fluid Inclusion Petrography and Microthermometry in Hematite Crystals

Martite and hematite I

Martite is opaque under infrared radiation and no fluid inclusions could be observed, although martite has a remarkable porosity under normal reflected light. Likewise, in the intergrown porous anhedral hematite I, it was not possible to identify fluid inclusions.

Hematite II

Although not all granular hematite II grains contain fluid inclusions, they are quite common and occur very noticeably as isolated inclusions with variable dimensions (5-50 μm). Two distinct groups can be distinguished (Table 2).

The first group (FI-I) comprises primary, two-phase inclusions, (L + V, fill ratio = 0.9–0.95). They are evenly

| Table 2. Microthermometric Data from Primary Fluid Inclusions of Hematite and Quartz Crystals |
|---------------------------------------------------|---|---|---|---|---|---|---|---|
| Mineral phase | Primary fluid inclusions | Fill ratio† | Dmin (μm) | Tmel (°C) | Tcal CO2 (°C) | Tevap (°C) | Tcond CO2 (°C) | Observations |
| Hematite II (Hm II) | Two-phase (FI-I) | 0.9–0.95 | 5–50 | 2–6.7 (18) | 115–145 (18) | – | – | Decrystallized fluid inclusions (7) |
| | Single-phase (FI-I) | 1 | <4–50 | NR | NR | – | – | |
| Hematite II-III (Hm II-III) | Two-phase | 0.9 | 5–50 | 2–2.5 (14) | 120–145 (15) | – | – | Similar to (FI-I) |
| Hematite III (Hm III) | Two-phase | 0.9–0.95 | <8 | 2–2.3 (4) | 120–140 (5) | – | – | Solid phases are absent |
| Specularite | Two, three and multiphase | 0.8–0.9 | 5 to 40 | –10.0 to –24.5 (24) | 140–205 (19) | – | – | Possible total dissolution of daughter crystals at 350–400°C |
| Quartz | Two-phase | –0.8 | <20 | 280–351 (24) | –57.0 (12) | 5.0 to 6.0 | 20.0 to 28.0 | Late fluids in relationship to specularite |

Notes: Tmel = melting temperature of ice, Tcal = homogenization temperature, Tcal CO2 = melting temperature of CO2, Tcond CO2 = homogenization temperature of CO2, number in parentheses = number of measurements; NR = not recorded, + = no data

† Fill ratio = volume fraction of liquid over total inclusions volume

By reading the text naturally, it appears to be a scientific paper discussing the use of infrared microscopy to study hematite and martite, highlighting the differences in their appearance and the presence of fluid inclusions. The table provides microthermometric data from primary fluid inclusions of hematite and quartz crystals.
distributed through the entire grain, almost always in smaller crystals (on avg 80 μm) with sharp, straight boundaries, and near hexagonal sections, free of porous domains and magnetic relics. They are usually large (5–50 μm, Fig. 4a–b), nearly spherical or elongated, and lack any solid phases or daughter crystals. Freezing of the inclusions to −120°C caused no phase change and no clathrate formation, which suggests the lack of a CO₂ phase. Determination of the eutectic temperature (Tₑ) was not possible due to difficulties in the microscopic observation, but ice melting temperatures (Tₑ(cal)) were determined between −2.8° and −8.7°C, (salinities from 4.6 and 10.2 wt % NaCl equiv; Bodnar and Vityk, 1994). However, most of the measurements (14) varied from −2.8° and −3.0°C (Fig. 5a), corresponding to salinities from 4.6 to 4.9 wt percent NaCl equiv (Bodnar and Vityk, 1994). During heating runs, two-phase fluid inclusions homogenized (Tₑ) into liquid at temperatures of less than 145°C (Fig. 5b).

The second group (F1-2) is the most common and consists of single-phase inclusions that occur alone or in groups concentrated in the core of larger crystals (usually >100 μm), generally surrounded by a clear, inclusion-free rim (Fig. 4c). They have subrounded to teardrop shapes with sizes from 4 to 50 μm and strong relief. Some of the larger inclusions are partially constricted (probably evidence of necking down) and surrounded by smaller inclusions (Fig. 4c–e). All of the analyzed inclusions had apparently leaked and were probably empty.

**Hematite II-III**

In the tabular-granular hematite II-III crystals, primary fluid inclusions are present only in the granular domain (Fig. 4d, Table 2). They are two-phase inclusions (L + V), with variable sizes (5–50 μm). Solid phases are absent, and the fill ratio is constant (0–9). Microthermometric determinations indicate a behavior similar to the F1-1 inclusions observed in hematite II crystals. The Tₑ(cal) is −2.0° to −2.5°C (corresponding to salinities from 3.39–4.18 wt % NaCl equiv; Bodnar and Vityk, 1994), and Tₑ is the liquid phase varies from 120° to 145°C. With continued heating all inclusions decomposed at 345° to 350°C. This thought to be due to weakening of the crystal structure during subsequent shearing (see below). Determination of the eutectic temperature (Tₑ) was not possible due to optical difficulties in the microscopic observation.

**Hematite III**

Idiomorphic tabular crystals of hematite III contain minute (<8 μm), two-phase (L + V) fluid inclusions of primary origin, some of them as negative crystals (Fig. 4e, Table 2). Most of the inclusions are oriented along growth planes, possibly parallel to the basal pinacoid. During freezing and heating Tₑ(cal) was determined in the range −22° to −23°C, and Tₑ was between 120° and 140°C. Determination of the eutectic temperature (Tₑ) was not possible due to optical difficulties in the microscopic observation.

**Specularite**

Fluid inclusions in specularite are primary, usually with negative crystal shape, smaller than 40 μm, and composed of two, three, and rarely four phases (L + V + S + S, fill ratio=0.8–0.9; Fig. 4f, Table 2). Solid phases are ubiquitous but always very small (<10%) of the total volume of the fluid inclusions and never exhibit a definitive geometric shape. Fluid inclusions are concentrated along the basal planes, and in slightly deformed crystals they locally have a sigmoidal shape (Fig. 4g). The eutectic temperatures (Tₑ) in some inclusions range between −60° and −50°C, suggesting the presence of Ca²⁺, Fe²⁺, or Mg²⁺ in the solution. The Tₑ(cal) values varied between −10.0° and −24.5°C (Fig. 5a), and most of the values concentrated in the narrow range of −19.0° and −24.5°C. In freezing and heating runs, no development of clathrates or melting or homogenization of CO₂ was observed, suggesting that CO₂ is not present in these inclusions. Homogenization temperatures (Tₑ) in the liquid phase ranged from 140° to 205°C (Fig. 5b). At 300°C the solid phase would commonly begin to dissolve, and above 300° to 350°C optical definition rapidly decreased and no detailed observation (final melting temperature) could be made. The total dissolution of the solids at a temperature higher than 350°C together with the determined Tₑ(cal) suggest a high salinity, above 30 wt percent NaCl equiv (Bodnar and Vityk, 1994).

**Quartz**

Quartz contains two main types of fluid inclusions. The first is represented by primary and small (<20 μm), three-phase, aqueous carbonic inclusions (t₁CO₂ + t₂CO₂ + t₃CO₂) at 25°C, with polygonal shapes (Fig. 4h). During freezing runs, Tₑ was determined at −57°C, suggesting the presence of other components in the carbonic phase. Raman spectra measurements in these fluid inclusions indicated the possible presence of traces of N₂ (Manduca et al., 2001). The clathrate melting temperature (Tₑ(clathrate)) varied between 5° and 6°C, indicating salinities close to 8 wt percent NaCl equiv (Collins, 1979). CO₂ homogenization to the liquid phase occurred between 26° and 28°C. The temperature of total homogenization into the liquid varied between 280° and 351°C, with modal values at 330° to 340°C (Fig. 5c).

The second type of fluid inclusion is less common and paragenetically late (secondary fluid inclusions). They occur aligned along fracture planes, usually close or crossing the grain borders. They are aqueous two-phase inclusions (L + V) without carbonic phases (no development of clathrates or melting or homogenization of CO₂ was observed in freezing and heating runs), and they have salinities between 6 and 7.8 wt percent NaCl equiv. During heating they homogenized in the liquid phase between 169° and 224°C (Fig. 5c).

**Discussion**

Evidence from petrographic analysis and fluid inclusions studies indicate that martitization and formation of hematite I, and crystallization of hematite II, hematite III, and specularite occurred during, at least, three distinct stages involving different hydrothermal fluids, metamorphism and deformation processes (Figs. 6-7) as discussed below.

An initial low-salinity fluid was possibly responsible for the development of an originally magnetite-rich ore, in a way similar to that proposed by Taylor et al. (2001), followed by oxidation to hematite I and upgrading during a subsequent hydrothermal event. Post-tectonic recrystallization contributed to grain growth and to the development of a granoblastic
Fig. 4. Fluid inclusions in hematite, all under infrared radiation in quartz, under normal light. (a). Large, two-phase aqueous fluid inclusions (FI-1) in granular hematite II. Hematite II crystal exhibits birefringence under infrared light with the formation of a double-image in some FI-1 fluid inclusions. (b). FI-1—FI-2 fluid inclusions in neighboring hematite II crystals that compose the granoblastic fabric. (c-1). Single-phase fluid inclusions (FI-2) in core domain of granular hematite (Hm II). Borders are clear and fluid inclusion free. (c-2). Larger balloon-shaped single-phase fluid inclusion surrounded by smaller inclusions (FI-2) in Hm II crystal. Inset shows enlargement of the balloon-shaped fluid inclusions and evidence of probable necking down. (d). Enlargement of inset from Figure 3(c), showing primary two-phase fluid inclusions typical of Hm II crystals, enclosed in an Hm II-III grain. Some of the inclusions are elongated parallel to the basal plane and decrepitated at 345° to 350°C. (e). Two-phase inclusions oriented parallel to the basal plane in hematite III (Hm III) crystal. (f). Primary fluid inclusion in specularite crystal. The sigmoidal shape is interpreted to be due to gliding along the basal plane. (g). Large fluid inclusions with hexagonal shape in specularite. The fluid inclusions in the left-hand side contain a small solid saturation phase. Insets g1 and g2 are enlargements showing solid inclusions that formed after heating. In g1, two solid phases formed after heating to 400°C and subsequent cooling. In g2 a single solid phase formed after heating and cooling. (h). Primary aqueous carbonic fluid inclusions in quartz at 25°C, $T_{\text{min}} = 16.8°C$ and $T_{\text{max}} = 149°C$. 

[^1]: Adapted from [1] with permission.
aggregate of porous anhedral hematite (hematite I) with inclusions of magnetite relics and several internal irregularities that could not be identified.

Hematite II, granular crystals, which occur intergrown with marlrite and hematite I, formed by the partial recrystallization of the ore during metamorphism. They locally contain magnetite relics, indicating their formation by recrystallization of hematite I. Although most of the grains are now free of fluid inclusions, many of them also contain large two- and single-phase inclusions. The single-phase inclusions (FI-2) are interpreted as the product of necking down and leading of pre-existing fluid inclusions during metamorphism, culminating with a multitude of empty inclusions scattered in the core of many crystals. The two-phase fluid inclusions (FI-1) are thought to be related to a low-temperature hydrothermal event, involving low- to medium-salinity fluids that were responsible for the formation of new crystals of hematite II.

**Origin of transitional hematite II-III, hematite III, and specularite**

The textural relationship and fluid inclusion data from tabular hematite III and platy specularite crystals indicate that both morphological types developed later than the hematite II (Rosière et al., 2001; this study). The two occur in wedge-shaped shear zones that cut the massive, granoblastic ore and were formed in the presence of a high-salinity fluid that was distinctly different from the earlier, low-temperature and low-salinity fluid (Fig. 6).

Tabular-shaped hematite III crystals have grown mostly interstitial to the granoblastic hematite II aggregates but also in isolated dilational sites and as veins with sharp to fuzzy boundaries in the walls of the shear zone. Their fluid inclusions have high salinity and relatively low homogenization temperatures (ca. 140°C). Hematite III may also overgrow hematite II, forming the hematite II-III transitional type (I-hematite II-III, Fig. 3c). These composite grains still contain relic primary fluid inclusions (Fig. 4d) inherited from the original hematite II. They have the same T_h and T_melicional as FI-1 fluid inclusions but
decrepitate between 345° and 350°C, a feature that is not observed in the FI fluid inclusions in hematite II. The decrepitation of these inclusions is interpreted as resulting from the structural weakening of the crystals during shearing.

On the other hand, specularite formed at higher temperatures in the central domain of the shear zones where penetration of fluids was enhanced. The lowest temperature of formation of specularite, inferred from the partial dissolution of the daughter crystals, is about 350°C, which is much higher than the homogenization temperature for aqueous inclusions trapped both in hematite II and III.

Specularite may form by crystal-plastic deformation of hematite, through dislocation glide on the basal plane, as discussed by Rosière et al. (2001) and determined experimentally by Siemes et al. (2003), or by fluid-assisted diffusion processes (Lageiro, 1998). In the Conceição samples, basal glide on specularite is suggested by the presence of sigmoidal inclusions (Fig. 40) aligned parallel to the trace of the (001) plane, although the prevailing temperatures (ca. 350°C) were much lower than the experimental values of 600°C from Siemes et al. (2003).

Both hematite III and specularite were apparently formed from similar high-salinity fluids at different stages in the evolution of the shear zones and represent the third and last hypogene mineralization event. Hematite III precipitated in the early stages, at low temperature, during the opening of
dilatational gashes without the development of a preferred orientation, and overgrowing hematite II grains. Specularite formed at higher temperature and at higher strain.

Relationship to quartz veins

The quartz veins from the analyzed samples cut across the metamorphic schistosity (S1) or interfere with the banded microstructure of the hematite ores. They envelop all the early minerals, including specularite plates and are the product of late, aqueous carbonic hydrothermal fluids of low salinity (less than 8 wt % NaCl equiv), with total homogenization temperatures of the fluid inclusions of approximately 330°C. These fluids are of uncertain age and origin and did not participate in oxidation of magnetite or Fe mineralization processes.

Metamorphism and preservation of low-temperature fluid inclusions

Although metamorphism produced a general granoblastic fabric in the massive ores, many granular crystals have preserved magnetite and martite relics in their cores. Some of the hematite II grains contain low-temperature, two-phase fluid inclusions (FI-I) and others contain probably empty or necked inclusions (FI-2). The low-temperature two-phase inclusions are thought to be relics that have been unaffected by metamorphism. This interpretation is supported by the presence of relic magnetite and martite in the core of some crystals. Regardless of their size, the FI-I inclusions in the hematite II crystals show no indication of fluid loss by leakage or decrepitation or any metastability during microthermometric measurements, including heating up to 350°C and subsequent cooling. The fill ratio was constant for all analyzed inclusions, and both the fill ratios and homogenization temperatures remained constant, even with repeated heating and cooling. These observations suggest that some of the hematite was quite stable under the metamorphic conditions that prevailed in this part of the Quadrilátero Ferrífero. Marshall et al. (2001) pointed out that there is no clear answer to the problem of the stability of fluid inclusions during metamorphism, and there are many examples of fluid inclusions studies of amphibolite fabrics gold deposits that contain both completely recrystallized quartz with homogenized fluid inclusion populations and relatively undeformed quartz with undisturbed fluid inclusion populations (e.g., including a population of several different types, different L/A ratios, and different homogenization temperatures). Microscopic and field observations also suggest that hematite may be more stable than quartz under certain conditions of metamorphism (e.g., Hespert et al., 2001; Sternes et al., 2003).

Conclusions

The analysis of the textural relationships and fluid inclusions preserved in the Concóicao ores supports a provision genetic model for the development of two types of high-grade orebodies: hard massive, partially concordant bodies, and tabular, schistose bodies controlled by shear zones (see also Fig. 7). Mineralization developed in several recurrent stages, with the participation of fluids of different characteristics and origin, including both hypogene and supergene processes.

Magnetite growth represents an early (probably initial) mineralization stage (Fig. 7), but its age cannot be precisely determined yet. This mineral is ubiquitous in the iron formations and high-grade bodies of the Quadrilátero Ferrífero, either as relics or as the main component in the extreme western part of the region. We suggest that in the early contractual stage of the Transamazonian orogeny (ca. 2.1–2.0 Ga; Alkmim and Marshak, 1998) reduced metamorphic fluids, together with connate water, were responsible for the pervasive magnetite mineralization similar to the model proposed by Taylor et al. (2001) for the Tom Price deposit in Western Australia. The folds of this phase of deformation are of flexural or flexural slip type, commonly with disharmonic to polyharmic profiles, and did not develop a pervasive axial planar cleavage (Rosière et al., 1993, 1996; Hippert and Davis, 2000).

Hematite I formed from martite following the oxidation of the magnetite (Rosière, 1981; Rosière and Chemelle, 1991; Rosière et al., 2001), whereas much of the hematite II grains grew from low-temperature and low- to medium-salinity hydrothermal fluids during a second episode of mineralization (Fig. 7). These fluids are interpreted to have been of meteoric origin (Fig. 7a), moving downward from the surface along normal faults and fractures during progressive uplift and extension of the crust at the end of the Transamazonian event. Such fluids may have been responsible for oxidation of the banded iron formations in the entire Quadrilátero Ferrífero district, but the high-grade bodies were developed only at sites of greater permeability such as fold hinges. Faults that controlled the mineralization have not been positively identified in the Concóicao deposit, owing to overprinting by structures developed during subsequent shortening, but the main orebody occurs within a relict Transamazonian structure (i.e., refolded F2, hinge zone of the Itabira synclinorium, Fig. 2).

The peak of the regional metamorphism in the Quadrilátero Ferrífero probably occurred during the Transamazonian extensional tectonism (Alkmim and Marshak, 1998) caused by the juxtaposition of hot basement and cooler supracrustal rocks, producing a granoblastic fabric in all rock assemblages of the Minas Supergroup. During crustal extension, ascending plutonic rocks (Norce, 1993) most likely caused additional recrystallization.

Syndeformational growth of idiomorphic, tabular hematite III and of specularite plates occurred during the later Brasiliano/PanAfrican orogeny in domains of intense deformation, with the development of a pervasive schistosity, mainly in the eastern part of the Quadrilátero Ferrífero (Chemelle et al., 1994). High-salinity (probably more than 20 wt % NaCl equiv) fluids penetrated along shear zones in its early stages, causing the precipitation of hematite III (Figs. 6–7) in microscopic dilational sites. Similar but higher temperature (~350°C) and high-salinity fluids assisted deformation by facilitating hematite basal gliding, forming platy hematite crystals (specularite). SiO2 from the iron formation must also have been leached by these fluids, and cations such as Ca2+, Fe3+, and Mg2+ were probably added to the system, possibly derived from dolomitic rocks at depth (Fig. 2). The combination of these factors produced the shear zone-related, high-grade tabular orebodies (Rosière et al., 2002). Although the relationship between the microshear zone, which hosts the sample analyzed in this study, and the regional structure is not clear, it is suggested that the relatively high grade of the presently mined shered itabirite from the deposits in the
Itabira synclinorium is a result of this third, shear-related, mineralization event (Fig. 7).

A similar model may apply to other deposits of the Quadrilátero Ferrífero, particularly in the western low-strain domain, where the early Transamazônica structures are better preserved. In this region, the most significant high-grade massive bodies are located in zones of internal folding, especially close to the contact with the uplifted gneiss basement and granitic bodies. In the eastern domain, weathered, schistose and sheared itabirite bodies have developed even in the absence of an oxidized magnetite protore and may represent an important exploration target.

Acknowledgments

We wish to express our thanks to the FAPESPI Foundation (Process CRA 175/02 to FJR) and to the research staff of ECI-CDTN, especially K. Fuzikawa, J. V. Alves, J. M. Correia-Neves, W. Macedo, and E. Costa. A. Matias is thanked for the excellence of the double-polished chips of hematite. Our thanks are also extended to CVRD, especially E. M. Resende da Souza, M. L. Vidal Guimarães, and E. Caldeira Leite for generously supplying the samples and sharing useful information. Thanks to W. Tito Soares from the Laboratório de Microscopia Eletrônica e Microanálises (UFMG-CDTN-FAPESPI CEX 1074/95). C. A. Rossié particularly thanks the CNPq (Brazilian National Research Council) for the grant, CAPES, FINEP/PADCT, and DAAD for all the previous support. Special thanks are due to H. Quade, the former supervisor of CAR for introducing him and other students of the Clausthaler Group to the problems of iron ore genesis and L. Lobato, N. Beukes, M. Bailey, B. Simonson, J. Gutsmuth, and C. A. Sper for the fruitful discussions. Many thanks are indebted to V. Leidinger for the valuable explanations about methodology and technical details of the Infrared equipment and to A. Campbell for his comments on infrared radiation sources. Finally, thanks are also due to J. Hannington (editor of Economic Geology), A. Cabral, and particularly to S. Hagman whose valuable review and suggestions contributed considerably to the improvement of the paper.

November 20, 2001; December 16, 2003

REFERENCES


C) ESTUDOS SOBRE CREPITAÇÃO: Foi desenvolvido um estudo preliminar sobre fases crepitantes e não crepitantes sobre amostras da Mina do Pico e Mina do Sapecado (setor oeste do QF). Foram estudadas 8 lâminas polidas de fases crepitantes e não crepitantes, e estabelecidas comparações. A crepitação das amostras foi testada nos laboratórios da Empresa MBR.

Durante os testes com NIV, todas as amostras estudadas apresentam transparência sob luz infravermelha. Além disso, a maior parte dos cristais de hematita apresentaram inclusões fluidas, mormente monofásicas.

Inicialmente, não haveria relação entre a presença de inclusões fluidas e a ocorrência de crepitação. Entretanto, observou-se que as fases não crepitantes, sob NIV, mostraram um padrão de tonalidades característico, sempre diferente daquele observado nas fases crepitantes. A continuidade dos estudos deverá confirmar essa tendência.

D) TESTES DE MIV EM GRANADAS ESCURAS E TURMALINAS PRETAS Foram desenvolvidos testes preliminares utilizando NIV em turmalinas e granadas notadamente opacas. As turmalinas e granadas foram amostradas em pegmatitos complexos da região de Galileia (Governador Valadares, MG). Todas as amostras comportaram-se como transparentes sob luz infravermelha, sendo possível observar feições internas e inclusões sólidas e fluidas.


D) FORMAÇÃO DE ALUNOS Foi orientada a aluna de Iniciação Científica Wherínea Cotta Dias na metodologia MIV, estudando os fluidos nos Nb Tantalatos (opacos) dos pegmatitos de Santa Maria de Itabira, MG. Não existem, na bibliografia, antecedentes de estudos de MIV nesses minerais. A aluna reconheceu feições internas (zoneamentos,
planos de crescimento, inclusões sólidas e, sobretudo, inclusões fluidas). O estudo está em andamento, e a aluna possui uma bolsa PIBIC para desenvolver suas atividades.

E) RESUMOS EXPANDIDOS E ABSTRACTS PUBLICADOS EM CONGRESSOS

32nd IGC - Florence, 2004

Abstract title
PETROGRAPHIC STUDIES IN OPAQUE MINERALS (HEMATITE, MARTITE) USING INFRARED MICROSCOPY

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Keywords
infrared microscopy
iron oxides
iron ore

Abstract
The use of infrared microscopy in the study of opaque minerals was developed in the last 15 years and requires the use of an IR microscope and high definition IR cameras and TV monitors. The Fluid Inclusion and Metallogenic Laboratory (EC1) of CDTN, BH, Brazil has developed an alternative system that permits petrographic and fluid inclusion studies. It consists of a 100 W (12 V) transmitted-light illuminator using halogen light bulbs and an infrared emission regulator with a 50 W/12 V Hosobuchi iodine-tungsten lamp coupled to a Leica DRMX microscope. A Sony ExwaveHad black and white, very near-IR camera and a Eletrophysics Micronviewer Model 7290-A IR camera, both coupled to a high-resolution Sony-Trinitron monitor with a Sony video graphic printer provide the infrared images. Infrared microscopy permits the observation of several remarkable features usually unseen under normal light. The hematite crystals generally exhibit a good transparency in the very near infrared spectrum. In magnetite-martite-hematite associations, observation with the IR system of the EC1 enabled the recognition of the original shape and size of magnetite crystals, despite oxidation, that has almost totally obliterated the early fabric as seen under normal reflected light. Under infrared light, it was also possible to recognize in hematite crystals several features such as twinning lamellae, growth lamellae, birrefringence, fluid inclusions, etc that are usually hardly or not visible under polarized reflected light.

ACCEPTED as Poster Presentation
in session: "G15.11 - Mineral inclusions"
Abstract title
THE ORIGIN OF HEMATITE IN HIGH GRADE IRON ORES BASED ON INFRARED MICROSCOPY AND FLUID INCLUSION STUDIES

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Keywords
Iron Ore
Fluid Inclusions
Infrared Microscopy

Abstract
Petrographic and textural analysis combined with fluid inclusion studies by infrared microscopy of high-grade (>65% Fe) hematite ore samples from the Conceição deposit, in the northeastern part of the Quadrilátero Ferrífero, Brazil, indicate a complex process of oxidation and mineralization during two orogenic events, each developed under different conditions and involving distinct fluids. The earliest mineralization formed massive magnetite-rich orebodies under relatively reducing conditions in the early stages of the Transamazonian orogeny. Magnetite was oxidized (martitized) with the development of porous hematite crystals (hematite I). Possibly during this stage, new hematite crystals were also formed from low-temperature, low- to medium-salinity fluids, as indicated by two-phase fluid inclusions. The origin of these fluids is still uncertain but tentatively interpreted as being modified surface water. The fluids were transported along normal faults and fractures during post-tectonic collapse following the Transamazonian orogeny (2.1 - 2.0 Ga) and creation of the dome-and-keel structural pattern of the Quadrilátero Ferrífero. These solutions were also likely responsible for the initial oxidation of the iron formations and the development of hematite I. Subsequent uplifted hot basement rocks or post-tectonic plutons were probable heat sources for the regional metamorphism and development of a granoblastic fabric of hematite II grains in the iron formations and high-grade orebodies. However, the ore was only partially recrystallized, as several relics of the early magnetite, martite and hematite are still preserved in the granular hematite II crystals. During the Brasiliano/Pan-African orogeny (0.8 - 0.6 Ga), high-salinity fluids, with temperatures varying from ~120°C to a maximum of approximately 350°C, penetrated the iron formations along shear zones, crystallizing initially tabular and thereafter platy hematite crystals (hematite III and specularite) forming schistose orebodies. Quartz veins that cut across the ore and envelop specularite plates and ore fragments formed from late stage, high temperature and low salinity fluids containing CO₂. These later fluids did not alter the hematite I ore.

Each of these stages of mineralization produced orebodies with distinct features. Recurrent hydrothermal mineralization is thought to have been responsible for the development of giant, high-grade iron ore deposits in structurally favorable sites.
RESULTADOS PRELIMINARES DO ESTUDO METALOGENÉTICO DO MINÉRIO HEMATÍTICO DE ALTO TEOR DO DEPÓSITO DE FERRO N5 – CARAJÁS, UTILIZANDO MICROSCOPIA E MICROTERMOMETRIA DE INFRAVERMELHO

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Os depósitos de ferro de Carajás inserem-se na sequência metavulcanossedimentar do Grupo Grão Pará, Supergroup Itacaiúnas. Jasplitos e minérios de alto teor (>85 % Fe) compõem a denominada Formação Carajás, sobre e sotoposta por rochas máficas. Jasplitos correspondem ao protominério e caracterizam-se por micro- e mesobandamento dado pela alternância de jaspe e óxidos de ferro.

O uso da microscopia de infravermelho aplicado ao estudo dos fluidos que originaram o minério de ferro apresenta poucos antecedentes. Na atual pesquisa, estão sendo estudadas amostras de minério de alto teor (denominada como hematita dura – HD – pela CVRD) de testemunhos de sondagem do depósito N5, Província Mineral de Carajás. As amostras estudadas são do furo 484, profundidade 230 m, quase no contato da HD com rochas metabásicas inferiores do Grupo Grão Pará. Nas amostras foi verificada a presença de: (a) hematita microcristalina bandada, com duas gerações diferentes, a primeira constituindo as bandas (hematita 1) e a segunda formando “olhos” de hematita 2; (b) veios monomineralícios, milimétricos até centímetros de hematita que cortam o bandamento. Esses veios são constituídos por, pelo menos, duas gerações diferentes: cristais lamelares (hematita 3) e cristais tabulares (hematita 4).

A pesquisa consiste em verificar a presença de inclusões fluidas e outras feições petrográficas nos cristais de hematita, utilizando a microscopia de infravermelho (MIV).

Até o momento, não foi observada transparência nos cristais de hematita 1 do bandamento. Porém, a MIV permitiu verificar que os olhos de hematita 2 estão formados por inúmeros microagregados milimétricos. Esses agregados mostram transparência sob MIV, embora não apresentem inclusões fluidas.

Os resultados obtidos com MIV nos veios monomineralícios de hematita são muito promissores já que a transparência obtida, durante a incidência do feixe de NIR (near infrared), é muito boa.

A hematita 3 geralmente aparece disposta em forma radial, constituindo o esqueleto dos veios, ou apresentando textura em pente (lembrando feições epitemais). Costuma apresentar macias (geminações) reticulares (em alguns cristais foi observada uma incipiente geminación polissintética). Além dessas feições, a MIV permitiu identificar dois tipos de inclusões fluidas: (I) inclusões primárias, pequenas (<10 µm), regulares até pseudopoligonais, momento bifásicas (L + V), onde a fase gasosa aparece muito subordinada), com preenchimento 0,9/0,95, muito provavelmente tratam-se de soluções de baixa temperatura de homogeneização, seguramente <150°C; e (II) inclusões fluidas tardias (secundárias), monofásicas (formadas por uma fase líquida) e <10 µm.

A hematita 4 (cristais tabulares) apresenta boa transparência sob MIV. Entretanto, as inclusões fluidas observadas são muito pequenas (< 5 µm e, aparentemente, monofásicas) o que dificultará seu estudo microtermométrico.
TIMING OF HYDROTHERMAL IRON MINERALISATION, CARAJÁS PROVINCE

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Together with under- and overlying basaltic rocks, jaspilites and iron ores constitute the Grão Pará Group in the Carajás Province, and represent an Archaean metavolcano-sedimentary sequence. The former exhibits evidences indicative of spilitic sea-water hydrothermal interaction and the latter have a typical geochemical signature of volcanogenic banded iron formations. The mineral associations of these rocks apparently reflect anchimetamorphic conditions, and both preserve an abundance of their original textural characteristics. The basin-closure structures are consistently preserved in brittle and brittle-ductile regimes throughout the Province, with cataclastic and fracture-cleavage features predominating and displaying oriented-mineral structural fabrics only along very discrete local structures.

World-known, huge iron deposits developed along the lower contact where both the underlying mafic rocks and jaspilites are mineralised, with the latter hosting ore in various places. In jaspilites, martitic magnetite and morphologically-variable hematite crystals replace the original microcrystalline hematite "dust", typical of jasper bands, and are themselves replaced (and/or recrystallised) by other generations of hematite (lamellar, microamellar/fibrous, anhedral/subhedral), which ultimately develop veins containing coarser-grained, spectacular tabular-shaped crystals; tabular hematite also cements ore breccias.

Lamellar and tabular, comb-textured hematite forms internal selvages to discordant and banding-parallel milli- to centimetric veins. The intensity of hematite enrichment may be accompanied by progressive destruction of the original fabric, including banding, and (i) jasper recrystallisation, developing clear, fine-grained granoblastic quartz, with attendant microcrystalline hematite substitution; (ii) quartz leaching. Late-stage, quartz and/or carbonate veinings are widespread. In the basal basaltic rocks, lamellar hematite fills in amygdules, veinlets, and can replace silicates. Presently-martitic, porphyroblastic magnetite is commonly surrounded by haloes of recrystallised jasper. Such porphyroblasts are recrystallised by anhedral to subhedral hematite.

These are the dominant peak hydrothermal textures in the high-value massive hematite ore, where only rare (some or few) isolated metric volumes of altered jaspilites are preserved. The textures indicate development at typical epithermal temperatures (>180°C). Such temperatures and the very high fluid-to-rock ratios attendant at hydrothermal alteration, associated with the mineralisation, are in excess of the ambient metamorphic conditions registered in the host rocks. The mineralisation therefore appears incompatible with its development during the peak metamorphic, or relatively dry deformation conditions. Conversely, retrograde metamorphism or deformation textures are not registered over the mineralisation. It is confidently concluded that hydrothermal, iron mineralisation in Carajás was developed over exhumed, very-low grade metamorphic chemical sediments, deformed under a brittle regime.

A hydrothermal fluid at epithermal temperature conditions was capable of leaching silica from the host rocks, forming the high-grade orebodies. The early-hydrothermal nature of magnetite suggests an original fluid at an fO2 that was in disequilibrium with the microcrystalline hematite of jaspilites. The fluid evolved through mineral reactions to produce the different, recrystallised hematite generations.

Mafic metavolcanic extrusive rocks of the Grão Pará Group are 2.76 Ga old. Preliminary Sm-Nd geochronological data of non-altered jaspilites, sampled at deposits N4, N5 and N8, indicate their formation at >2.59 ± 0.2 Ga. Considering the metamorphic peak to be Neo-Archaean, closely following back-arc basin formation, the age of iron mineralisation is inferred to be younger.
ACCEPTED as Poster Presentation

in session: "G14.09 - Metallogeny of large and super large mineral deposits"

Geocongress 2002 & 11th Quadrennial IAGOD Symposium, Namibia, 2002

DEFORMATION-CONTROLLED HIGH-GRADE IRON ORES

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In the Quadrilátero Ferrífero massive hematitic high-grade iron ore occurs usually as lenticular bodies partially controlled by the banding of the ilbitite iron formation while others are clearly related to shear zones cross-cutting the layering. These are almost entirely constituted of oriented specularite platelets. Specularite develops both as a product of crystal-plastic deformation of hematite, or as product of solution-precipitation processes. Both mechanisms interact in the partitioning of the strain while solution-precipitation also is responsible for the localized leaching of silica in order to produce syn-tectonic ores. A first attempt to determine the characteristics of the fluids involved in the processes by infrared microscopy in specularite crystals have indicated the presence of higher saline aquo-carbonic H2O N NaCl fluids containing other additional cations (Ca, Fe, Mg) in solution and T, averaging between 130 N 150 °C.

Structure framework of the Quadrilátero Ferrífero and relation to high-grade iron ores

The structure of the Quadrilátero Ferrífero (Dorr, 1969) is delineated by a roughly square arrangement of synclines, with Paleoproterozoic metasediments of the Minas Supergroup, separated by antiformal structures with Archean greenstone terrains of the Rio das Velhas Supergroup and domes of Archean and Proterozoic crystalline rocks (Machado et al., 1992; Machado and Carneiro, 1992; Noce, 1995). The thickest sequence of iron formations from the Minas Supergroup (ilbitites) together with enclosing high-grade iron ore bodies belong to the Itabira Group (2419 +/- 19 Ma - Pb-Pb isochron date, Babinski et al., 1995).

The regional structure is the result of the superposition of two main deformatinal events (Chemaie Jr. et al., 1994): the first produced the nucletation of regional synclines in the supracrustal sequence, and the uplift of the gneissic domes during the Transamazonian Orogenesis (2.1 N 2.0 Ga); the second is related to a west N verging thrust belt of Pan-African/Brasiliano age (0.8 N 0.6Ga). Two main structural domains (Rosière et al., 2001) can be regionally delimited: the Eastern High-Strain Domain has thick shear zones and important thrust systems, whereas the Western Low-Strain Domain displays well preserved megasynclines that are discontinuously cut by discrete shear zones and faults.

An eastward increasing metamorphic zoning is also regionally defined by the presence of chlorite, biotite and staurolite respectively (Herz, 1978), and follows the deformatinal gradient. These were later redefined as zones of grunerite (GZ), cummingtonite (CZ), actinolite (AZ) and
tremolite-antophyllite (TAZ) (Fig 1B) by Pires (1995), based on the metamorphic parageneses of the iron formations of the Itabira Group.
High-grade iron ore bodies (>64% Fe) occur both as soft and hard hematite bodies. Hard hematite bodies may occur as
1. compact (massive or banded) that represent the first iron-ore generation in the area. These bodies present more or less lenticular shape, with variable dimensions from few centimeters to hundreds meters, discontinuously distributed along the strike of the itabirite layer. When present, the banding is defined by alternating compact and porous layers of iron oxides (marlrite with magnetite relics and granoblastic hematite) with interstitial gangue minerals (quartz and phyllosilicates).
2. schistose ores that occur in shear zones related to the Brasiliano thrust belt, pervasively in the Eastern High-Strain Domain. Hematite commonly occurs as euhedral platelets that are referred to as specularite when its basal plane defines a schistosity of variable intensity developing a shape- and lattice-preferred-orientation and composing a lepidogranoblastic to lepidoblastic fabric (Rosière et al., 2001).

Origin of specularite

Specularite may form by crystal plastic deformation of hematite by dislocation glide on the basal plane (hier called specularite I), (Rosière et al., 2001) but also by fluid assisted diffusion processes (Lagoi, 1999) with dissolution of Fe²⁺ from magnetite in a similar manner to that proposed by Davis et al. (1968) and Morris (1983). Subsequently Fe²⁺ reacts with oxygen and precipitates as specularite (specularite II) at extensional sites together with quartz or carbonates along magnetite grain boundaries parallel to the foliation, particularly in strain shadows, but also in veins, filling fractures and vugs (Lagoi, 1998; Rosière et al., 2001).
Specularite I is the product of activation of crystal plastic processes during iron ore deformation and it's presence does not have any implication in the iron enrichment. Specularite II, otherwise, is the product of syntectonic hematite precipitation and growth in an anisotropic environment, controlled by the instantaneous stretch direction. These crystals evidence iron and silica remobilization by hydrothermal fluids contributing, at least, for the partial enrichment of the protore.

Characteristics of the fluids from some quartz-hematite veins

Several hematite N'quartz veins cut the metamorphic schistosity (s₁), some of them disregarding structural control, while others are lodged along the s₁- or s₂ schistosity planes. Both quartz and hematite usually grow more or less randomly but may be locally oriented parallel or perpendicular to the vein walls.
First petrographic and fluid inclusion analysis from a cross-cutting vein in the Conceição iron ore deposit, (NE- Quadrilátero Ferrifero) with support of infrared microscopy, allowed the recognition of two forms of hematite
- hypidiomorphic to idiomorphic tabular crystals (<500µm) with polysynthetic twin lamella of primary origin (growth twins), entirely devoid of magnetite inclusions.
  These crystals present minute (<5 m) two-phase fluid inclusions, of primary origin, some of them with negative crystal shape. Most of them are oriented along growth planes, probably parallel to the basal pinacoid. Due to the minute size of the inclusions it was not possible to clearly determine phase changes during cryometry but homogenization temperature Tᵢ was determined in the range between 120 °C to 140 °C.
- very long specularite platelets in radial disposition, commonly slightly flexured (specularite II), Fluid inclusions in specularite II are primary, two-phase (L+V, f 1= 0.8 N 0.9), usually with negative crystal shape and of variable dimensions, but usually smaller than 20µm (Fig. 3e and 3f). Inclusion trails were identified in several grains concentrated along the basal planes of the specularite plates. In slightly deformed crystals these inclusions display sigmoidal
shape. There was no evidence of carbonic phases in the analyzed inclusions and $T_e$ (eutectic temperature) was not precisely determined. In closely associated quartz, carbonic phases (CO$_2$) were identified in very subordinate amounts. The eutectic temperatures in some inclusions appear to range between $95^\circ$ and $96\pm9^\circ$ C (mean $92.5^\circ$ C), indicating the presence of different cations (Ca, Fe, Mg) besides Na in the solution. $T_{mp}$ values varied between $99^\circ$ C and $117.9^\circ$ C, corresponding to intermediate to high salinity (12.8 to 20.8 wt% equiv. NaCl; Bodnar and Vityk, 1994). Homogenization temperatures ($T_h$) ranged between 130 and 160 $^\circ$ C.

Genetic considerations

Hard massive high-grade iron ore bodies are bedding-concordant and don’t present any clear straight forward relationship with tectonic structures but several schistose bodies are clearly related to shear zones developed during the Brasiliano thrust tectonic. Field evidences and textural/microstructural studies on the hematite shear zones indicate that both crystal plastic mechanisms and pressure solution processes played a role in the development of the discordant schistose iron ore bodies found mainly in the Eastern High-Strain Domain from the Quadrilátero Ferrífero. Syntectonic fluids whose origin is still being subject of studies contributed to remove a considerable amount of silica leaving a residual enrichment of iron oxides in a similar way as described by Taylor et al. (2001), but a critical eye must be thrown on the effectiveness of this mechanism as solely responsible for the formation of economically important deposits.

At least part of the remobilized silica and iron precipitate as quartz-hematite veins proximate to the schistose orebodies. Preliminary studies about the characteristics of the fluids from inclusions in hematite crystals indicate a metamorphic origin, under slightly higher temperature compared to the ones associated with the hard massive bedding-concordant bodies (see also Rosière and Rios this band). The upper temperature values are somewhat lower than the ones determined from quartz fluid inclusions by Hagemann et al. (1999), as well as reported by Barley et al. (1999) and Taylor et al. (2001), from hematite-quartz veins found at the giant Mount Tom Price deposit (Hamersley Range).

Geocongress 2002 & 11th Quadrennial IAGOD Symposium, Namibia, 2002

INFRARED MICROSCOPY AND FLUID INCLUSIONS ANALYSIS IN HEMATITE FROM HIGH-GRADE IRON ORE. GENETIC CONSIDERATIONS

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Infrared microscopy studies in itabirites and its hosted high-grade hematite ores from several deposits of the Quadrilátero Ferrífero mining district, Brazil, complementary to routine optical petrography in reflected light give a new insight in the understanding of the relationship between microscopic fabric, mineralogical transformations and metamorphism. Through infrared microscopy it is often possible to better estimate the oxidation/martilization degree of magnetite, to recognize completely inverted hematite and also to distinguish a zoning in most hematite crystals due to the distribution of inclusions. While the core domain is inclusion-rich the borders are
inclusion free. The analysis of the textural relationship conjugated with the salinity and microthermometric data of the fluid inclusions allow important genetic considerations.

The Itabirite-iron formation in the Quadrilátero Ferrifero

In the Quadrilátero Ferrifero two main structural domains (Rosière et al., 2001) can be regionally delimited: the Eastern High-Strain Domain has thick shear zones and important thrust systems, whereas the Western Low-Strain Domain displays well preserved megasynclines that are discontinuously cut by discrete shear zones and faults. An eastward increasing metamorphic zoning is also regionally defined (Herz, 1978, Pires, 1995), and follows the deformational gradient.

Itabirites are metamorphosed, intensively oxidized and heterogeneously deformed banded iron formations, with a variety of mineralogical and textural types according to the presence and relative abundance of gangue minerals: quartz itabirites, dolomitic itabirites and amphibibolitic itabirites. Considerable mineralogical and geochemical variation are explained by the original composition of the sediments, intensity of deformation and degree of metamorphism and hydrothermal alteration processes.

A thick sequence (ca. 250-300m) of itabirites intercalated with hematite phyllites, dolomitic phyllites and marbles comprise the Cauê Formation that, together the dolomites of the Gandarela Formation (2419 +/- 19 Ma, Pb-Pb isochron data, Babinski et al., 1995) constitute the Itabira Group. The Itabira Group is a sequence of predominantly chemical sedimentary rocks from the Paleoproterozoic Minas Supergroup (Dorr, 1969) deposited in a shallow basin to open platformal environment.

These iron formations delineate the main regional structures of the entire Quadrilátero Ferrifero as a guide horizon and massive high-grade deposits are disseminated along the megasynclines or may also occur as schistose bodies in shear zones on the Eastern High-Strain Domain.

Fabric relationships in hard massive high-grade iron ores

In the Low-Strain Domains lenses of high grade iron ore occur as massive bodies, have their shape totally or partially controlled by the original sedimentary bedding of the BIF protore and usually reproduce the original structure in a granoblastic fabric. They represent a first, pre-tectonic mineralization pulse.

The massive high-grade ores are constituted of hematite and marlites aggregates with magnetite relics (Mg Ni Fig. 1a) These fabric relationships have been described in high-grade ores and itabirites from several areas in the Quadrilátero Ferrifero (Hackspacher, 1979; Rosière, 1981; Rosière and Chemal, 1991; Rosière et al. 1996) as well as in Australian ores (Morris 1980, 1983). Marlites aggregates appear in reflected polarized light as -porous- hematite with microscopic to submicroscopic inclusions (Fig. 1a), but are totally opaque under near infrared light not allowing a determination of its nature and composition (Fig. 1b). IR microscopy results in the image of opaque magnetite -phantoms- (Mg Ni Fig. 1b) enclosed in hematite aggregates. This circumstance does enable, however, recognition of the original shape and size of early magnetite crystals even though martilization has almost totally obliterated the fabric as seen under normal polarized reflected light.

Hematite appears in two different forms:

- Granoblastic aggregates. They are recrystallized grains (<200µm) with straight to slightly curved boundaries that surround and are intergrown with martilized magnetite.
- Small oriented platelets dénominated specularite I developed by deformation and anisotropic recrystallization of granoblastic hematite (Rosière et al. 2001). Specularite plates define a more or less continuous schistosity surrounding and cutting through the granoblastic aggregates.
Granoblastic hematite grains contain numerous isolated primary fluid inclusions (Roedder, 1984) with variable dimensions (5–50 μm). These inclusions are restricted to the center of the grains (Fig. 1c) while the borders are inclusions-free.

Fig. 1. Microscopic features and fluid inclusions in hematite: (a) Porous microfabric of massive ore with granular hematite crystals surrounding martite with magnetite relic (Mg). Reflected light, partially XPL, sample CO260N1. (b) Same as (a) under IR radiation. Original shape of incomplete martitized magnetite is recognized as opaque mineral (Mg). Complete inverted hematite crystals are transparent. Sample CO260N1. (c) Monophase aqueous Fl in granoblastic hematite. (d) Two-phase aqueous Fl in granoblastic hematite (e) Fl in granoblastic hematite with most monophase inclusions, concentrated in the central part of the crystal. Borders are Fl-free probably due to recrystallization. (f) Oriented specularite I crystals (Spec I) defining the schistosity with martitized magnetite (Mg) crystals. The iron oxides are surrounded by coarse remobilized quartz. Transmitted light. PPL, sample CO260N5.

Fluid inclusions

Cores of hematite grains from hard massive ore, recrystallized from oxidized (martitized) magnetite, contain an abundance of primary, predominantly monophase aqueous fluid inclusions, but two-phase inclusions (L+V, fill = 0.90 N 0.95) are also present. They are usually near spherical or elongated without solid phases or daughter saturation crystals with T, ≤ 120°C and low salinity (4.6 N 10.2 wt% equiv. NaCl, Bodnar and Vityk, 1994). Similar inclusions are absent from grain margins, possibly a result of synmetamorphic deformation and consequent recrystallization. The final product of metamorphism is a granoblastic to lepidoblastic fabric composed of subhedral and platy hematite crystals (specularite I) almost completely devoid of fluid inclusions.

Fig 2 Schematic representation of the mineralogical changes in the iron oxides in ores from the Quadrilátero Ferrífero and its relation to the fluid inclusions.

Genetic consequences

The analysis of the textural relationships between magnetite, martite and the granoblastic hematite grains on samples of high-grade ore, conjugated with salinity and microthermometric data, supports the conclusion that the fluid inclusions of the hematite crystals are inherited from early, pre-tectonic martization process of pre-metamorphic magnetite, that probably occurred in low depth, possibly during the late diagenesis of the lower Minas Supergroup sequence. With increasing temperature, during recrystallization of hematite, these fluid inclusions were probably preserved in the nuclei of the crystals, while in the borders they were allowed to escape, assisted by deformation. Lattice distortion, followed by recrystallization by grain boundary migration and crystal growth, resulted in totally inclusion-free grains such as the specularite I platelets (Fig. 3c). Although metamorphism has produced recrystallization of hematite, several evidences indicate that the characteristics of its fluid inclusions did not change during the geological evolution of the Quadrilátero Ferrífero. Salinity and temperature values probably reflect the conditions prevailing during oxidation and mineralization of the iron formation protore at temperatures lower than 150°C, as already recognized by Gruner (1926).

Acknowledgements
We wish to express our thanks to the research staff of the CDTN (CT2), especially K. Fuzikawa, J. V. Alves, W. Macedo, J. M. Correa-Nunes, E. Costa, L. Andrade, M. Martins and P. Gasteoios. A. Mathias is thanked for the excellence of the doubly polished chips of hematite. Our thanks go also for the CVD's specially E. M. Resende de Souza, M. L. Vidigal Guimarães and E. Caldeira Leite, and MBR, specially C. A. Spier for generously supplying the samples and sharing valuable information. C. A. Rosiète particularly thanks the CNPq (Brazilian National Research Council) for the grant, CAPES, FINEP/PADCT and DAAD for all the previous support. Thanks are also due to J. Gutzmmer, N. Beukev, M. Barley, B. Simonson H. Quade and H. Siemis for the fruitful discussions.

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CONSIDERAÇÕES FINAIS

Com o apoio da FAPEMIG, foi aperfeiçoada, e colocada definitivamente em rotina, a metodologia de Microscopia e Microtermometria de Infravermelho (MIV). O sistema permite que minerais opacos se comportem como transparentes quando bombardeados com radiação infravermelha.

No momento, o CDTN conta com o único laboratório de MIV para geociências em operação e rotina na América do Sul.

A viabilidade do sistema foi confirmada por especialistas do US Geological Survey, Japan Geological Survey e University of Western Australia (dentre outros). A metodologia foi testada com sucesso em várias gerações de minério de ferro (hematita granular, amorfa, tabular e especcularita) de jazidas do Quadrilátero Ferrífero (MG) e Serra dos Carajás (PA). Testes com MIV forneceram bons resultados de transparência em turmalinas pretas e granadas escuras do Setor Governador Valadares (MG), além de wolframatos (Pedra Preta, PA) e, recentemente perovskita, antimonita, sulfetos de prata e esfaleritas pretas (altamente ferríferas).

Consideramos que o Projeto atingiu amplamente os objetivos inicialmente propostos.

Desenvolvemos uma metodologia de trabalho inédita, o que permitiu o estudo de oxídos de ferro, sulfetos de prata, nióbio tantalatos, wolframatos, minerais gemológicos escuros, esfaleritas escuras, dentre outros.

Nossa experiência mostra que não é suficiente possuir um sistema de MIV instalado num laboratório. É necessário, além disso, conhecer a metodologia de trabalho e estar ciente de que as pesquisas são bem mais demoradas do que aquelas desenvolvidas com microtermometria de luz transmitida normal.

O Projeto ajudou a consolidar a parceria EC4 (CDTN) e IGC (UFMG). O trabalho publicado na Economic Geology é prova disso. Estão em andamento trabalhos conjuntos, utilizando a MIV, nos setores de N4 e N5 (Serra dos Carajás, PA) e algumas jazidas do leste do Quadrilátero Ferrífero (MG).

Finalmente, a metodologia MIV está disponível para toda a comunidade científica.
AGRADECIMENTOS

Agradecemos aos colegas do lab. de Física de Superfícies (EC1 do CDTN), especialmente ao Eng. Eden Crsitiano Souza, pelas discussões e fundamental colaboração na elaboração das fontes de infravermelho.

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