International Journal of Natural and Engineering Sciences 4 (2): 11-14, 2010
ISSN: 1307-1149, E-ISSN: 2146-0086, www.nobel.gen.tr

Characterization of Dolomite, Pyrite and Chalcopyrite Mineral Rocks of Pakistan

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Received : February 20, 2010
Accepted : April 17, 2010

Abstract

We studied the dolomite $CaMg(CO_3)_2$, pyrite FeS_2 and the chalcopyrite $CuFeS_2$ mineral rocks of Pakistan, using electronic probe micro analyzer (EPMA) of scanning electron microscope (SEM) with energy dispersive X-ray spectrum (EDS) analyzer. We observed in dolomite two phases perhaps due to crystallization in the matrix, due to calcite $CaCO_3$ and quartz SiO_2. In pyrite phase transition occurs due to pyrrhotite $Fe_{1-x}S$ (x = 0 to 0.2) and Carbon in the matrix of pyrite. In chalcopyrite two different kinds of phase transitions are observed, due to carbon, quartz SiO_2 and pyrrhotite $Fe_{1-x}S$ (x = 0 to 0.2) in the matrix of chalcopyrite. These phase transitions in respective mineral rocks show disperse crystal mineralization due to pressure and temperature changes for more than thousands of years. Phases are observed with EDS and MLA.

Key Words: Dolomite, Pyrite, Chalcopyrite, SEM, EDS, Mineral Rocks

INTRODUCTION

X-ray diffraction, infrared X-ray absorption and Mössbauer spectroscopy [1] techniques have been used for material characterization. Modern analytical techniques including, transmission electron microscopy (TEM), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), energy-dispersive X-ray fluorescence spectrometry (EDXRF), electron probe microanalysis (EPMA), optical microscopy, backscattered electron imaging (BSE), proton induced X-ray emission (PIXE), X-ray photoelectron spectroscopy (XPS) and laser ionization mass spectrometry (LIMS), secondary ion mass spectroscopy (SIMS, often employing an ion-microprobe), inductively coupled plasma-atomic emission spectroscopy (ICP-AES) with the LECO method (ASTM D4239-83), and laser ablation inductively coupled plasma mass spectroscopy (LAICP-MS), permit analysis of mineral samples with high spatial resolution [2]. The sensitivities of the techniques are such as to permit detection of many trace elements down to ppm ($\mu g\ g^{-1}$) and ppb ($pg\ g^{-1}$) levels. Using such techniques and appropriate mineral samples, the investigator is able to image the specimen prior to analysis of a selected 'spot' with spatial dimensions of several microns or less. Imaging under high magnification permits the user to avoid (or select) visible inclusions or atypical regions of the sample [3].

Analysis of limestones and dolomites has been done using x-ray fluorescence [4]**.** Selective chemical separations of mineralogical constituents and evaluation of trace and major elements of dolomites and limestones have been done [5,6]. Ying Gu [7], presented methods and techniques used in the Mineral Liberation Analyzer (MLA), consisting of a specially developed software package and a standard modern SEM, fitted with an energy dispersive spectrum (EDS) analyzer, on ore samples containing pyrite and the chalcopyrite. The decomposition and oxidation of pyrite was studied using SEM micrographs [8]. A chemical, morphological and electrochemical (XPS, SEM/EDX, CV, and EIS) analysis

of electrochemically modified electrode surfaces of natural Chalcopyrite and Pyrite in alkaline solutions was done by Velasquez Pablo et al [9]. B. Thomas et al [10], studied the layer morphology and structural properties of FeS_2 (pyrite) thin films, grown on natural pyrite and synthetic ZnS crystals by scanning electron microscope (SEM) and X-ray diffraction, etc. Energy dispersive X-ray spectroscopy (EDS) of Cu-Ni ore- bodies from different mines of Botswana, was done by Nkoma and Ekosse [11], regarding mineral characterization and elements special relationships. They also used the X-ray diffraction (XRD) technique to study these samples [12]. Using XRD data they not only found the dominant and less dominant compounds in the ore, but also obtained the lattice parameters for chalcopyrite, pentlandite and pyrrhotite, which relate to the compounds in their natural state. Hu Junqing et al studied the synthesized $CuFeS_2$ nanorods [13] and ultra fine powder [14], using X-ray powder diffraction and transmission electron microscope (TEM) results.

We report in the present paper the EPMA, SEM and EDS studies done on the ore samples of Pyrite and Chalcopyrite, obtained from rocks of Balochistan, Pakistan. These results are an extension of our previous work on the above mentioned mineral ore. We have so far studied the XRD and Magnetization [15] as well as Electrical Resistivity and Thermoelectric Power of Pyrite [16] and Chalcopyrite [17] ore samples, with theoretical calculations of Grüneisen function and various other useful parameters.

RESULTS and DISCUSSION

The surface morphology was observed by an electronic probe micro analyzer (EPMA), with scanning electron microscope (SEM) and energy dispersive X-ray spectrum (EDS) analyzer apparatus, JEOL JED-2300 Analysis Station. Chemical compositions were measured by energy dispersive X-ray spectrum (EDS) analysis, attached to the SEM apparatus.

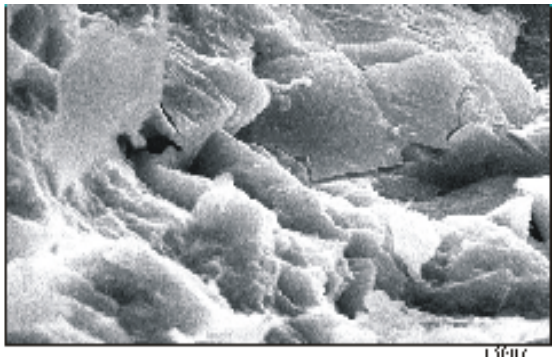

Figure 1. SEM micrograph of Dolomite showing morphology of the surface

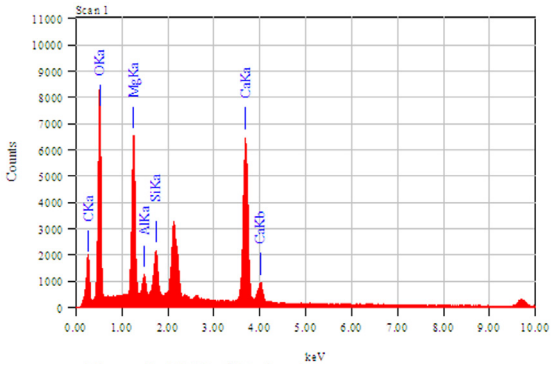

Figure 2. EDS of Dolomite

Table 1. Quantitative Analysis of Dolomite

ZAF Method Standardless Quantitative Analysis

Fitting Coeff. : 0.2515
Acqusition Parameter: Instrument: 6380(LA)
*Acc.Voltage:*20.0kV *Probe Current:*1.00nA *PHAmode:*T3
*Real Time:*76.52s *Live Time:*30.00s *Dead Time:*60%
*Counting Rate:*16878cps *Energy Range:*0-20keV

Element	(keV)	mass%	Error%	At%	K
C K	0.277	14.85	0.12	**21.88**	6.5427
O K	0.525	54.61	0.23	**60.41**	51.8418
Mg K	1.253	11.86	0.12	**8.64**	10.8355
Al K	1.486	1.49	0.12	**0.98**	1.3993
Si K	1.739	2.65	0.11	**1.67**	3.0806
Ca K	3.690	14.53	0.14	**6.42**	26.3001
Total		100.00		100.00	

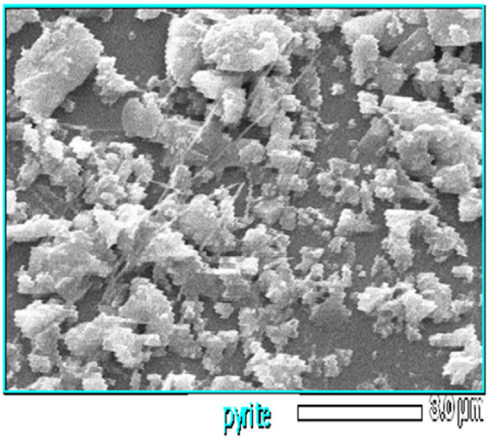

Figure 3. SEM micrograph of Pyrite showing morphology of the surface

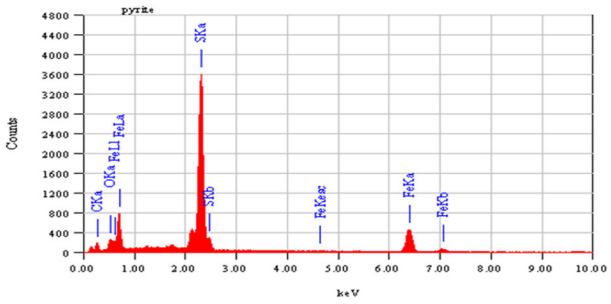

Figure 4. EDS of Pyrite

Table 2. Quantitative Analysis of Pyrite

ZAF Method Standardless Quantitative Analysis

Fitting Coeff. : 0.1815
Acquisition Parameter: Instrument: 6380(LA)
*Acc.Voltage:*15.0kV *Probe Current:*1.00nA; *PHAmode:*T3
*Real Time:*35.92s *Live Time:*30.00s *Dead Time:*16%
*Counting Rate:*3318cps *Energy Range:*0-20keV

Element	(keV)	mass%	Error%	At%	K
C K	0.277	17.84	0.19	**39.85**	2.1178
O K	0.525	5.25	0.16	**8.81**	4.2184
S K	2.307	40.40	0.09	**33.80**	52.3561
Fe K	2.307	36.50	0.58	**17.53**	41.3078
Total		100.00		100.00	

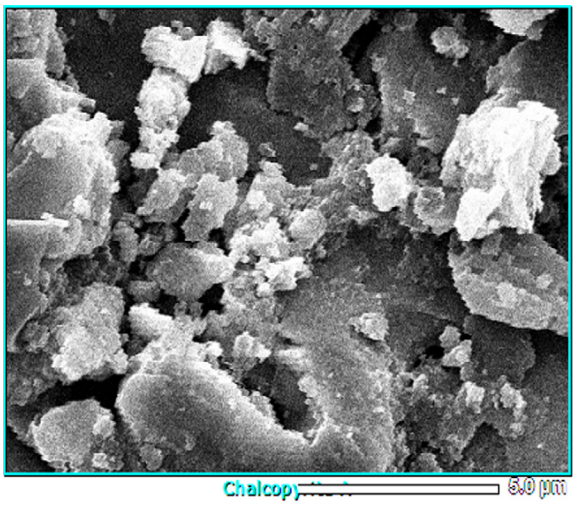

Figure 5. SEM micrograph of Chalcopyrite showing morphology of the surface

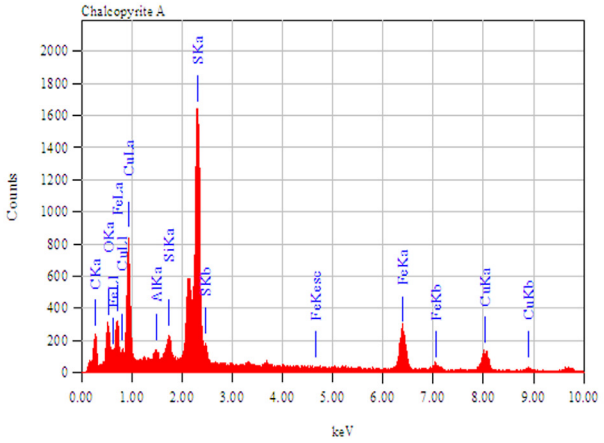

Figure 6. EDS of Chalcopyrite

Figures 1, 3 and 5, are the micrographs of the ore samples, showing morphology of the surface of Dolomite, Pyrite and Chalcopyrite respectively.
Figures 2, 4 and 6 are the EDSA of the ore samples, showing crystal information of Dolomite, Pyrite and Chalcopyrite respectively. The elemental analysis indicates the presence of Silicon in dolomite. In pyrite, Carbon in elemental form with some inclusion of CO_2 is indicated.

Table 3. Quantitative Analysis of Chalcopyrite

ZAF Method Standardless Quantitative Analysis

Fitting Coeff. : 0.2752
Acquisition Parameter: Instrument: 6380(LA)
*Acc.Voltage:*15.0kV *Probe Current:*1.00nA; *PHAmode:*T3
*Real Time:*35.05s *Live Time:*30.00s *Dead Time:*14%
*Counting Rate:*28548cps *Energy Range:*0-20keV

Element	(keV)	mass%	Error%	At%	K
C K	0.277	18.63	0.19	**42.77**	3.2683
O K	0.525	7.53	0.18	**12.97**	7.3962
Al K	1.486	0.94	0.16	**0.96**	0.7637
Si K	1.739	1.55	0.15	**1.52**	1.5434
S K	2.307	22.03	0.13	**18.94**	28.0777
Fe K	6.398	24.01	0.76	**11.86**	29.9231
Cu K	8.040	25.30	1.97	**10.98**	29.0276
Total		100.00		100.00	

The analysis of chalcopyrite also shows Carbon in elemental form with some inclusion of CO_2 but Silicon is also detected.

We observed in dolomite two phases, perhaps due to crystallization in the matrix. A phase transition is observed due to the presence of calcite $CaCO_3$ and the other due to quartz SiO_2. In pyrite a phase transition occurs due to pyrrhotite $Fe_{1-x}S$ *(x = 0 to 0.2)* and Carbon in the matrix of pyrite. We observe in chalcopyrite two different kinds of phase transition, due to presence of quartz SiO_2, pyrrhotite $Fe_{1-x}S$ *(x = 0 to 0.2)* and Carbon. These phase transitions in respective mineral rocks show disperse crystal mineralization due to pressure and temperature changes for more than thousands of years. Phases were observed with EDS and MLA.

REFERENCES

[1] Hang Nam Ok, Kyung Seon Baek, and Eun Jung Choi, *Mössbauer study of antiferromagnetic* $CuFeS_{2-x}Se_x$, Physical review B, **14**, 50, 10327-30, **(1994)**

[2] Abraitis P.K, Pattrick R.A.D, Vaughan D.J, *Variation in the compositional, textural and electrical properties of natural pyrite* , Int. J. Miner. Process. **74,** 41-59, **(2004)**[3] Yang Shao-Horn, Quinn C. Horn, *Chemical structural and electrochemical comparison of natural and synthetic* FeS_2 *pyrite in lithium cells,* Electrochimica Acta. **46**, 2613-21, **(2001)**

[4] Brander D. Wheeler, *Analysis of limestones and dolomites by x-ray fluorescence*, The Rigaku Journal, **1**, 16, 17-24 **(1999)**

[5] Barber C., *Major and trace element associations in limestones and dolomites*, Chemical Geology, **4**, 14, 273-80, **(1974)**

[6] Tzen-Fu Yui, Shou-Yeh Go, *Stoichiometry effect on stable isotope analysis of dolomite*, Chemical Geology Volume **3-4**, 201, 359-68, **(2003)**

[7] Ying Gu, Journal of Minerals and Materials

Characterization & Engineering, **1,** 2, 33-41, **(2003)**

[8] Guilin Hu, Kim Dam-Johansen, Stig Wedel, Jens Peter Hansen, *Decomposition and oxidation of pyrite*, Progress in Energy and Combustion Science **32**, 295-314, **(2006)**

[9] Velasquez Pablo, *A chemical morphological and electrochemical (XPS, SEM/EDX, CV, and EIS) analysis of electrochemically modified electrode surfaces of natural Chalcopyrite and pyrite in alkaline solution*, Journal of Physical Chemistry B **11**, 109, 4977-88, **(2005)**

[10] Thomas B., Ellmer K., Mueller M., Hoepfner C., Fiechter S., Tributsch H.; J. Cryst. Growth (Netherlands), **1-4,** 170, 808-12 **(1997)**

[11] NKoma, J.S.; Ekosse, G.; Coetzee, S. H, *Energy dispersive X-ray spectroscopy (EDS) of Cu-Ni ore bodies,* Proceedings–microscopy society of Southern Africa **33**, 33,**(2003)**

[12] J S Nkoma and G Ekosse, *X-ray diffraction study of chalcopyrite $CuFeS_2$, pentlandite $(Fe,Ni)_9S_8$ and pyrrhotite $Fe_{1-x}S$ obtained from Cu-Ni ore bodies*, J. Phys.:Condens.matter **11**, 121-128, **(1999)**

[13] Hu J., Lu ,Q.; Deng, B.; Tang, K.; Quian, Y.; Li Y.; Zhou, G.; Liu, X. **,** *A hydrothermal reaction to synthesize $CuFeS_2$ nanorods,* Inorganic chemistry communication **2(12),** 569-571, **(1999)**

[14] Hu Junqing, Lu Qingyi, Tang Kaibin, Qian Yitai, Zhou Guien, Liu Xianming, *A solvothermal reaction route for the systhesis of $CuFeS_2$ ultrafine powder,* Journal of materials research, **14(10)**, 3870-72, **(1999)**

[15] Naqvi S.M.M.R., Shabana Rizvi, Dabir.H.Rizvi S., Abbas S.Z., Raza S.M., Mujeeb Ahmed, Kamaluddin S., & Mahmood Z., *Magnetization behavior of Pyrite and Chalcopyrite Minerals.* 9th International Symposium on Advanced Materials, Proc., 429-433 **(2005)**

[16] Naqvi S.M.M.R., Shabana Rizvi, Dabir H. Rizvi S., Mohsin Raza S., Najma Shams and Yasmin Rizvi; *Electrical Resistivity and Thermoelectric Power Measurements of Pyrite (FeS_2) in the Temperature Range 78-300 K*. Journal of the Chemical Society of Pakistan, **3**, 29, **(2007)**

[17] Shabana Rizvi, *PhD Thesis of Shabana Rizvi*. Dept. of Physics, University of Karachi, Karachi