Qatar Univ. Sci. J. (2001), 21: 97-100

Whole-rock powders and their analysis by an electron mocroprobe technique

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مساحيق الصخور وتحليلها بواسطة تقنية المسبار الإلكتروني الدقيق

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صهرت مساحيق لصخور قاعدية أنعم من ١٠٠ لشبكة المنخل، في وعاء تنجستن ومولبدنيوم في وسط نتروجيني، وتستخدم في تقنية الانصهار كميات صغيرة جداً من العينة، والتي تكون مناسبة للتحليل السريع للعينات الصخرية، خاصة ذات التركيب القاعدي.

إن الزجاج الناتج من الانصهار يكون متجانساً إلى حد ما، وملائماً للتحليل بواسطة المسبار الإلكتروني الدقيق، وقد أمكن اختبار التجانس بواسطة القيم الإحصائية المعطاة في الورقة.

إن دقة وسرعة تحاليل المسبار الإلكتروني الدقيق لزجاج العينات الصخرية يسمح بالاستعمال الروتيني لهذه التقنية، وتسمح هذه التقنية بالتحاليل الاستطلاعية للعناصر الرئيسية لمجموعة من الصخور التي لم تحلل من قبل في زمن أقصر وبتكلفة أقل.

Key Words: Electron microprobe-Homogenous – Rock powders – Fused technique.

ABSTRACT

Basic rock powders finder than 100 mesh were fushed in a molybdenum and tungsten boat in nitrogen atmosphere. The fusion technique uses very small amounts of sample which is suitable for rapid analysisof rock sample, especially of basic composition. The glasses are fairly momogenous to be suitable for electron mocroprobe analysis. The nomogeneity of the rock glasses was tested by CV values.

The accuracy and rapidity of the mocroprobe analyses of the rock glasses allow routine use of such technique. This technique allows major element reconnaissance analyses of rock suites not analyzed before in very short time and withless cost.

INTRODUCTION

Geochemists have discussed the possibility of analyzing rocks by the electron mocroprobe technique [1]. Brown [1] discussed techniques used in electron microprobe major elements analysis of whole rock samples. He also described a new technique for fusing powders in molybdenum boats in a pressurized (60 psi) argon atmosphere.

The purpose of this paper is to investigate the direct fusion of basic rocks in molybdenum and tungsten boats in a nitrogen atmosphere, using a strip furnace, and to analyze the resulted glasses by the electron microprobe technique..

It is expected that this experiment will develop a rapid and simple method of fusion of rock powders into representative, homogenous beads suitable for analysis by electron microprobe, without the need of an argon pressure chamber.

METHODS OF STUDY

The technique used for this study are those usually used in the Mineral Science Department, Smithsonian Institution, Washington, U.S.A.

The Mo and W boats used in the fusion are inexpensive and readily available from most SEM suppliers. They are very practical for holding all molten glasses. The best sizefor routine sample fusion is a 17/8 inch strip containing a boat 7/16 inch long, 3/16 inch wide and 3/32 inch deep.

The melting boat, connected between two posts, is covered by a glass bell jar that is continuously flushed by nitrogen. After placing the 200mg powder, ground finer than 100 mesh, in the fusion boat and covering the fusion assembly by the bell jar, it takes about 1min., to establish an atmosphere of almost pure nitrogen. This is manifested by the lack of oxidation of a Mo strip heated in this atmosphere at 1700° to 1750°C for 25 min.

Samples were heated for about 10 sec at low temperature to drive off absorbed water and water of hydration if present. The temperature was then rapidly increased to approximately 1700°C. The powders usually melt in 3-5 sec but total heating at the final temperature for about 10-30 sec promotes diffusion and thus formation of more homogenous glasses.

Quenching of the melts is done by simultaneously shutting off the power to the fusion bridge and directing a stream of nitrogen onto the bottom of the fusion boat. Cooling is sufficiently fast to prevent crystallite formation.

The glasses were analyzed by a 9-spectrometer computer-automated ARL-SEMQ electron microprobe. The spectrometers were equipped with the following analyzing crystals. Si-EDDT, AL-EDDT, Fe-LiF, Mg-ADP, Ca-LiF, Na-RAP, K-Lif, P-ADP.

The data were corrected by an on-line computer using the method of [2]. During the analysis, 15 kV accelerating potential and 20mA sample current were used. A defocussed beam 20-50 μm in diameter, practically eliminates Na volatilization during analysis and also helps to average local, small inhomogeneities which may exist in the glass. 10 sec counting times were used and the analysis were repeated at least ten times to accumulate a statistically significant body of data. The repeated analyses help to average possible larger scale inhomogeneties.

RESULTS

Five rock powders were fused by the described technique. All rocks are from a basic intrusion from the Khaybar area, southwestern part of Saudi Arabia [1]. Rocks contain variable amounts of plagioclase, augite, olivine, orthopyroxene and some opaque minerals [4]. Table 1 shows the analyses by the electron microprobe technique, while Table 2 shows comparison between analyses by the wet chemical and electron microprobe methods. The electron microprobe data show good agreement with the wet chemical analyses, which were recalculated volatile free in order to make direct comparison with the electron microprobe analysis.

Table 1

Analyses of 5 fused rock powder samples of electron microprobe technique.

Sample No.	SH-1	SH-2	SH-3	SH-4	SH-5
SiO_2	43.41	45.39	48.32	47.28	48.50
TiO_2	1.75	1.55	1.03	0.67	1.21
Al_20_3	15.43	16.03	17.47	17.48	17.77
FeO	13.83	10.05	7.69	7.20	9.61
MnO	0.25	0.19	0.12	0.12	0.13
MgO	11.74	10.39	9.61	10.62	8.39
CaO	10.71	13.17	11.42	14.18	11.68
Na_2O	2.02	2.55	2.87	1.78	1.86
K_2O	0.13	0.52	0.49	0.23	0.38
P_2O_5	0.10	0.03	0.15	0.06	0.07
Total	99.37	99.87	99.17	99.62	99.60
		Fusion conditions			
Temp. (°C)	1700	1700	1700	1700	1650
Time (Sec)	30	30	30	30	30

Table 2

Comparison between analyses of 5 rock samples by wet chemical and electron microprobe methods.

Sample No.	**SH-1**		**SH-2**		**SH-3**		**SH-4**		**SH-5**	
	C	M	C	M	C	M	C	M	C	M
SiO_2	43.40	43.41	45.37	45.39	48.37	48.32	47.26	47.28	48.47	48.50
Ti_2O	1.73	1.75	1.59	1.55	1.01	10.03	0.65	0.67	1.22	1.21
Al_2O_3	15.39	15.43	15.98	16.03	17.45	17.47	17.44	17.48	17.74	17.77
FeO	13.80	13.83	10.07	10.05	7.71	7.69	7.23	7.20	9.63	9.61
MnO	0.23	0.25	0.17	0.19	0.11	0.12	0.12	0.12	0.12	0.13
MgO	11.78	11.75	10.37	10.39	9.59	9.61	10.69	10.62	8.42	8.39
CaO	10.69	10.71	13.15	13.17	11.39	11.42	14.42	14.18	11.71	11.68
Na_2O	2.10	2.02	2.58	2.55	2.89	2.87	1.80	1.78	1.89	1.86
K_2O	0.12	0.13	0.50	0.52	0.45	0.49	0.21	0.23	0.40	0.38
P_2O_5	0.09	0.10	0.04	0.03	0.16	0.15	0.05	0.06	0.05	0.07
Total	99.33	99.38	99.82	99.87	99.12	99.17	99.65	99.62	99.65	99.60

Table 3 shows the CV values, CV equal to standard deviation X 100/mean. These values reflect not only the homogeneity of the glasses, but also include a component of variation due to instrumental instability.

The standards used in the glass analyses were analyzed by wet chemical techniques at the Department of Mineral Science, Smithsonian Institution.

CONCLUSIONS

The described fusion technique used smallamounts of sample. Major elements concentrations can be measured more rapidly and quite accurately for basic to intermediate rocks.

The fused glasses are generally homogeneous, and the electron microprobe is quite accurate to allow routine use of this technique.

The rapidity and accuracy with which samples can be prepared and analyzed allowed major element reconnaissance analyses of rock suites not analyzed before due to cost and time factors involved.

ACKNOWLEDGEMENT

I would like to thank Mr. J. Nelen, Department of Mineral Sciences, Smithsonian Institution, Washington, D.C., USA, for introducing me to this technique to analyse rock samples.

Table 3

CV values of the electron microprobe data and the wet chemical analysis.

Sample No.	SH-1	SH-2	SH-3	SH-4	SH-5
SiO_2	0.02	0.03	0.07	0.03	0.04
TiO_2	0.81	1.80	1.39	2.14	0.58
Al_2O_3	0.18	0.22	0.08	0.16	0.12
FeO	0.15	0.14	0.18	0.29	0.15
MnO	5.89	7.90	6.15	0.46	0.25
MgO	0.18	0.14	0.15	0.46	0.25
CaO	0.13	0.11	0.25	0.10	0.18
Na_2O	2.74	0.83	0.49	0.79	1.13
K_2O	5.66	2.77	6.02	6.43	3.63
P_2O_5	7.44	20.20	4.56	12.86	23.57

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