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Study of volcanic sediments by microbeam-PIXE technique

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Abstract

Single mineral grains in a suite of Cretaceous (85–90 Ma) volcanic sedimentary rocks were analyzed using the microbeam-PIXE technique to understand fundamental geological processes responsible for micro-scale variation in elemental composition across mineral zones, twinning, cleavage planes, fractures and grain boundaries. Distributions of major and trace elements show distinct geochemical features indicative of a specific geological setting and a subsequent diagenetic process in these volcanic sediments. Examples of mineral zoning, microstructures of variable chemistry and post-depositional fluid flow are discussed using the data on feldspar group of minerals.

1. Introduction

Characterization of authigenic minerals that form in response to sedimentary processes provides significant information pertaining to the sedimentary basin in terms of tectonic, thermal and chemical evolution of the associated sedimentary rocks. Detailed chemical and petrological data obtained from these minerals are typically used to discern several parameters including temperature, pressure, fluid composition, oxidation state, pH, etc. These data can be effectively applied to determine P – T (pressure–temperature) pathways of rocks in natural conditions. In addition, they also provide a viable tool for determination of provenance and permit recognizing paleotectonic setting of the sedimentary distributive provinces.

Trace element analysis of single mineral grains, fluid inclusions, mineral zones and matrix phases can help to understand fundamental geological processes responsible for micro-scale variation in geochemistry of the rocks. This in turn can lead to relating the specific geological processes to a particular thermodynamic setting. The data can also be tested against similar natural geological environments.

Proton Induced X-ray Emission (PIXE) spectroscopy has proved to be effective technique for sensitive and accurate analysis of major and trace elements in mineral samples [1,2]. Microbeam-PIXE has recently added a new dimension to research in geological science by allowing measurements of trace element distributions at a spatial resolution of a few μm [3]. Because of its high elemental detection sensitivity (at ppm levels), microbeam-PIXE has

a distinct advantage over competing Scanning Electron Microscopy (SEM).

2. Method

2.1. Sample preparation

Polished thin sections (about 30 μm) of lithified samples were prepared by standard techniques and mounted on petrographic slides with epoxy resin. The thickness of the samples was dictated by the requirement of petrological microscopes which were used to record relevant mineralogical and petrographical characteristics, and to mark areas of interest for micro-PIXE analysis. This thickness was also just about enough to stop 2.5 MeV protons or reduce their energy to an extent making them unproductive to generate X-rays from high Z traces, if any, in the glass mounts. For micro-PIXE analysis, the samples were coated with a thin layer of carbon to make the surfaces electrically conducting. The carbon layer was too thin to have any significant effect on PIXE measurements.

2.2. Analysis

The scanning proton microprobe at the Energy Research Laboratory (ERL) of the Research Institute of King Fahd University of Petroleum & Minerals (KFUPM) was used to carry out the measurements reported in this paper. The ERL microprobe facility has been described elsewhere [4,5] and some of its applications have been reported recently [6–8]. Typical spatial resolution of the microbeam for 2.5 MeV protons is about 4 μm at a current of about

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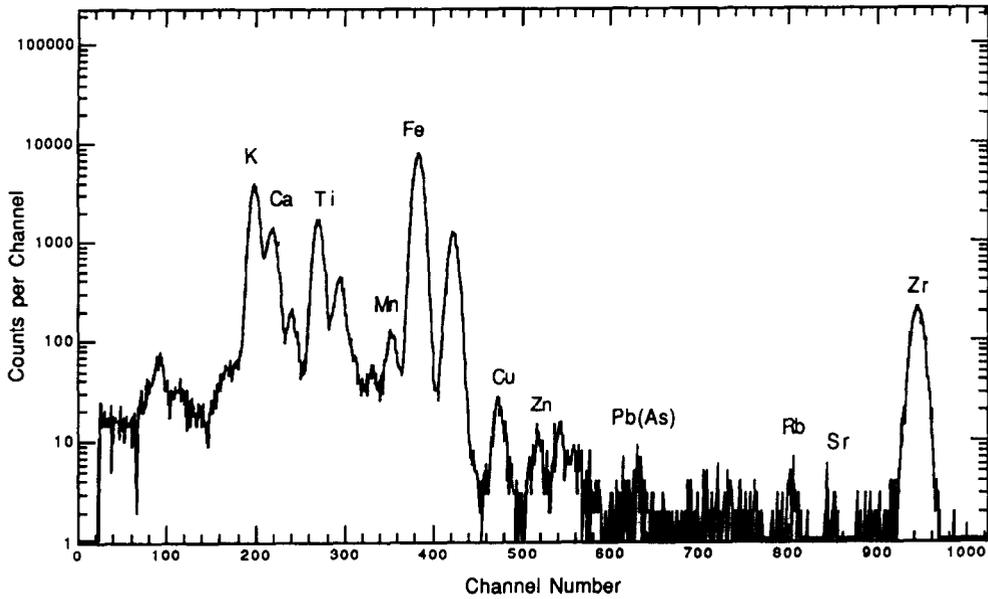


Fig. 1. A typical micro-PIXE spectrum averaged over the scanned area of $540\ \mu\text{m} \times 540\ \mu\text{m}$ on a feldspar grain at $E_p = 2.5\ \text{MeV}$ showing the presence of several trace elements.

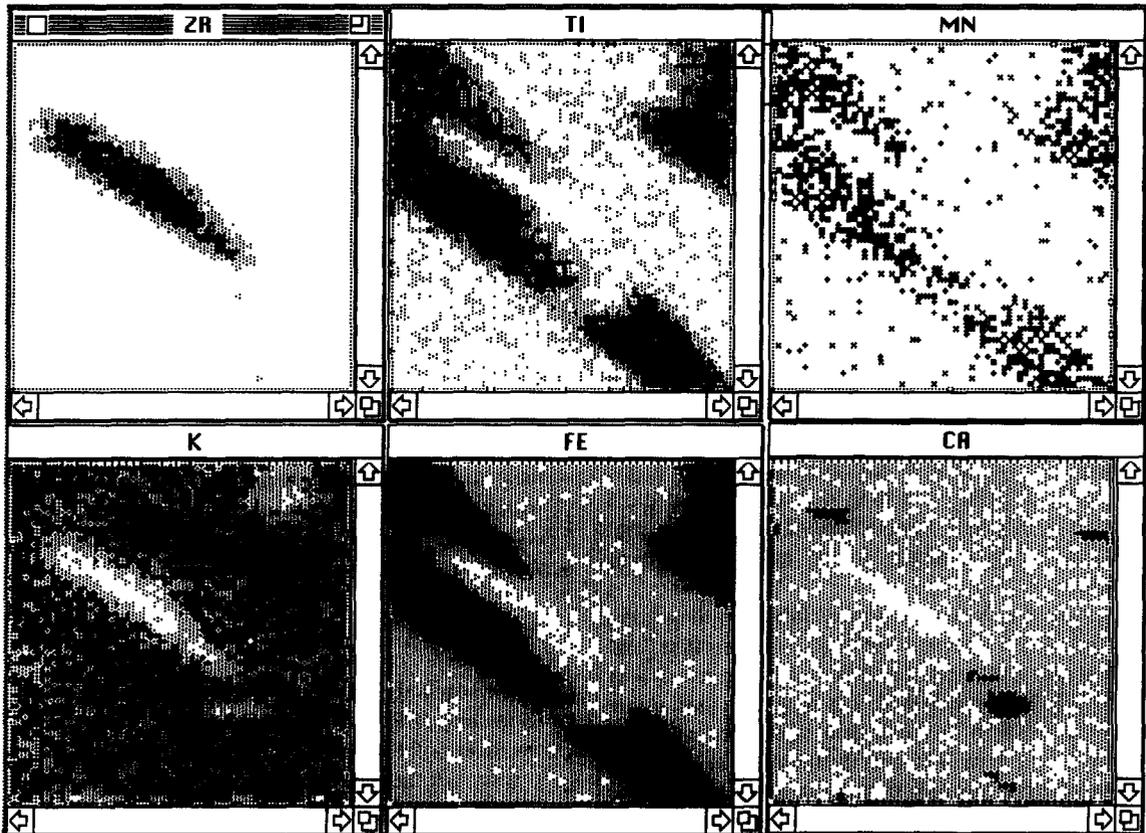


Fig. 2. Elemental distribution maps from $540\ \mu\text{m} \times 540\ \mu\text{m}$ area inside a feldspar grain showing microstructures having distinct spatial variations in chemistry.

50 pA and the maximum scanning range is 540 μm in each of the X and Y directions. A secondary electron detection system was used to quickly position the microbeam on any region of interest on the sample for subsequent PIXE analysis. From the scanned area of the sample, an average elemental composition spectrum as well as multiple elemental distribution maps have been produced simultaneously.

3. Results and discussion

Thirty seven samples of various mineral phases including feldspar, pyroxene, amphibole, biotite, chlorite and magnetite were studied. Results of these analyses show distinctive microtextural and micromorphological features associated with volcanic sediments. Some of these features will be illustrated here by using the data on the feldspar group of minerals. Fig. 1 shows a typical PIXE spectrum averaged over the scanned area of a feldspar grain. Although no absolute elemental concentration data could be made available, estimations relative to major constituents (K and Ca) indicate the presence of several trace elements (Rb, Sr, Zr, etc.) in these samples. Detection of

such trace elements in single mineral grains provides an important tool to study chemical alteration in volcanic sediments after their deposition. Some of these traces such as Zr are immobile and resistant to changes in diagenetic environment [9–11]. These immobile trace elements can therefore serve as index to the original chemistry of the volcanic sediments [12–14]. In contrast, many of the major constituents are mobile and can easily be removed from or added to the volcanic sediments, thereby altering the original mineralogy and creating a problem in interpretation of geological data on volcanic sediments [15–17]. Trace element data on geological samples are therefore very useful. In this respect, the PIXE technique, being sensitive to trace element detection, has an important advantage over other analytical techniques including electron microscopy for the study of geological samples.

Morphology of volcanic sediments are determined by several physical–chemical factors including depth of burial, post-depositional fluid flow, chemistry of the diagenetic fluid, original composition of the parent constituents, Eh–pH of the diagenetic fluid, and post-depositional tectonic overprinting. Elemental distribution maps measured in the feldspar specimen (Figs. 2–4) show evidence of some of these influences on volcanic sediments. Maps in Fig. 2,

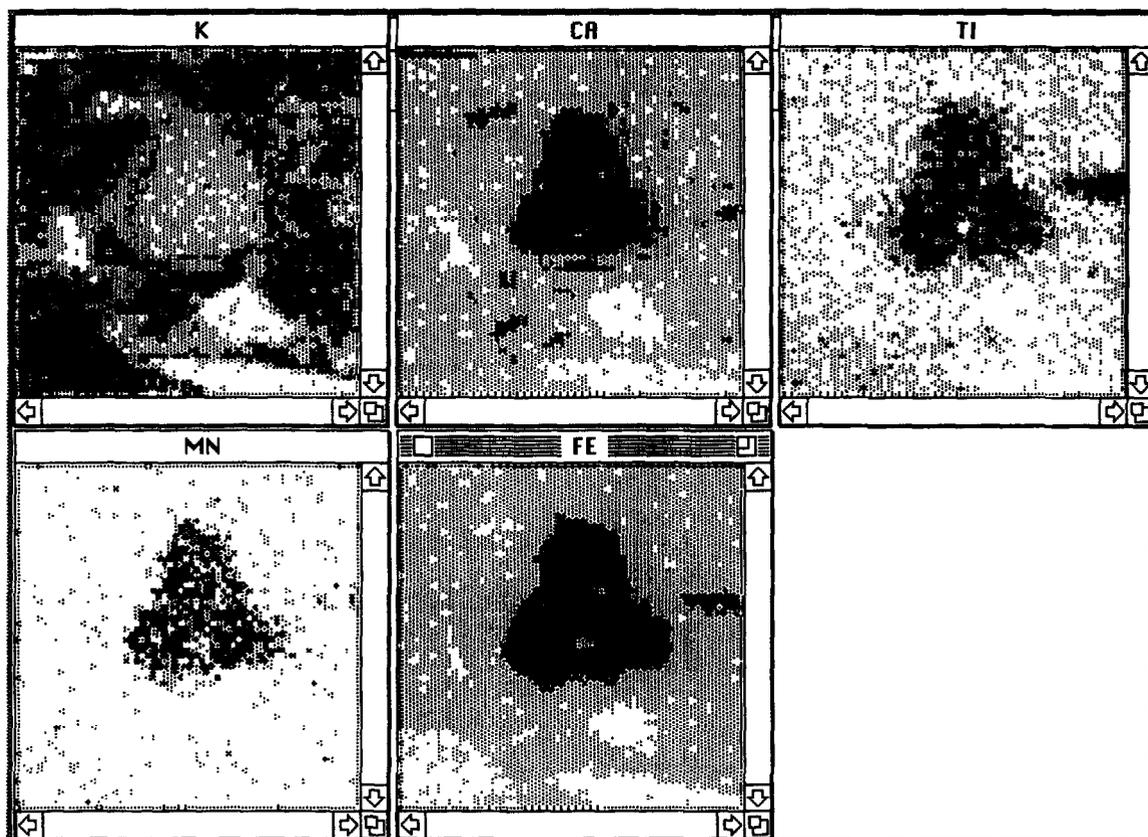


Fig. 3. Elemental distribution maps from 540 $\mu\text{m} \times 540 \mu\text{m}$ area encompassing a small feldspar grain showing an example of normal zoning.

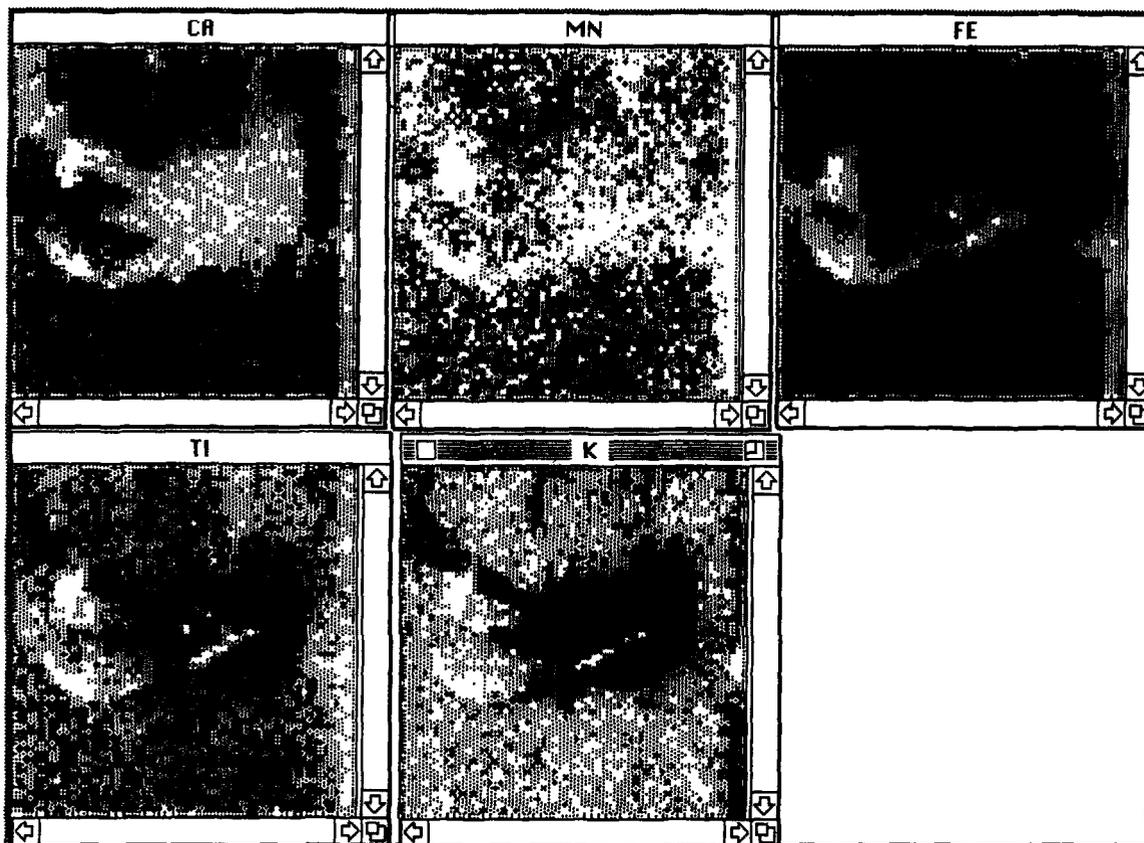


Fig. 4. Elemental distribution maps from $540\ \mu\text{m} \times 540\ \mu\text{m}$ area inside a feldspar grain showing a case of post-depositional fluid flow.

obtained from an area of $540\ \mu\text{m} \times 540\ \mu\text{m}$ within a feldspar grain, show some of the microstructures of the grain. Presence of trace element Zr is anticorrelated with the major elements K, Ca, Ti, Mn and Fe. This clear chemistry variation within the grain can be attributed to temperature effects which cause elements to segregate and crystallize at different regions. Fig. 3 maps encompassing a small feldspar grain show an example of normal zoning where the core consisting of primary elements Ca and Fe is surrounded by a secondary element K. Maps in Fig. 4 display the case of post-depositional fluid flow through microfractures and remobilization of secondary elements with the fluid flow. Individual maps in this figure show that the primary elements, Ca, Mn and Fe are completely or partially flushed out from the core by the secondary elements K and Ti brought in by the incoming diagenetic fluid.

4. Conclusions

Present study on volcanic sediments with microbeam-PIXE technique shows the following geological and geochemical features:

- Mineral zones are very pronounced in the feldspar group of minerals, particularly in Ca-rich feldspars.
- There are microstructures within a grain having distinct variations in chemistry.
- There is a strong evidence of post-depositional fluid flow through microfractures.
- Presence of immobile traces like Zr along with petrological data support an intermediate type volcanic provenance nearby for these suite of sediments.

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