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Scanning x-ray fluorescent microanalysis of rock samples

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Results are given concerning the two-dimensional scanning microanalysis of microsections of metallographic specimens of rocks. The monochromatized or white SR beam from a wiggler magnet installed at the electron storage ring VEPP-3 (2 GeV, 20 kG, 100–200 mA) was used for fluorescence excitation. The working energies ranged from 8 to 35 keV. Without focusing x-ray optics, the spatial resolution was 60 μm with the monochromatic beam and 30 μm with the white SR beam. The minimum detectable concentration of the elements (within the Fe-Sr region) is 10 ppm for 1–3 s exposures per point.

INTRODUCTION

Mircro x-ray fluorescent analysis using synchrotron radiation has some significant advantages over the broadly used electron microprobing technique.[1–3] Among the advantages of value for geological samples is the possibility of analysis of elemental mapping at a considerably lower level of concentration (100 to 1 ppm and lower), the possibility of identification of heavy elements using K series, and especially, the possibility of selective excitation of the elements in a complex matrix.

To analyze the elemental composition of microsections of metallographic specimens of racks, ores, and minerals, the authors have designed and tested a device comprising a monochromator, mechanical scanner, and a semiconductor detector. At the first stage, measurements have been made for test objects (wires, grids, geological samples). Scanning analysis of minerals has been performed for identification of elements (Cl to I).

I. EXPERIMENTAL DEVICE

The sample is excited by the monochromatized or white SR beam produced by a wiggler magnet installed at the storage ring VEPP-3 (2 GeV, 20 kG, 100–200 mA). The working range of a pyrolytic monochromator is 8–35 keV. To achieve a maximum flux of quanta, a single-crystal scheme of monochromatization was used.

A flat 50×50×3 mm crystal of pyrolytic graphite was used as a monochromator; the angular mosaic spread was 50 in., and the reflectivity factor was 20%. We have also realized a variant where the crystal monochromator focused the beam along the horizontal axis. In this case, a conic-formed crystal of pyrolytic 40-mm-long graphite with 20 (entrance) and 12 mm (exit) bending radii was formed according to the procedure suggested at the Institute of Graphite Based Materials of Construction (Moscow). Such a crystal makes it possible to obtain a quasimonochromatic x-ray beam within 20–29 keV energies with a spectral width of 20%–30% and a minimal focal size of 1.5 to 2 mm.[4]

The air-filled sample chamber was made from tungsten. In the chamber, there is a scanner allowing a shift, with a 10-μm step, of the sample in two mutually perpendicular direc-

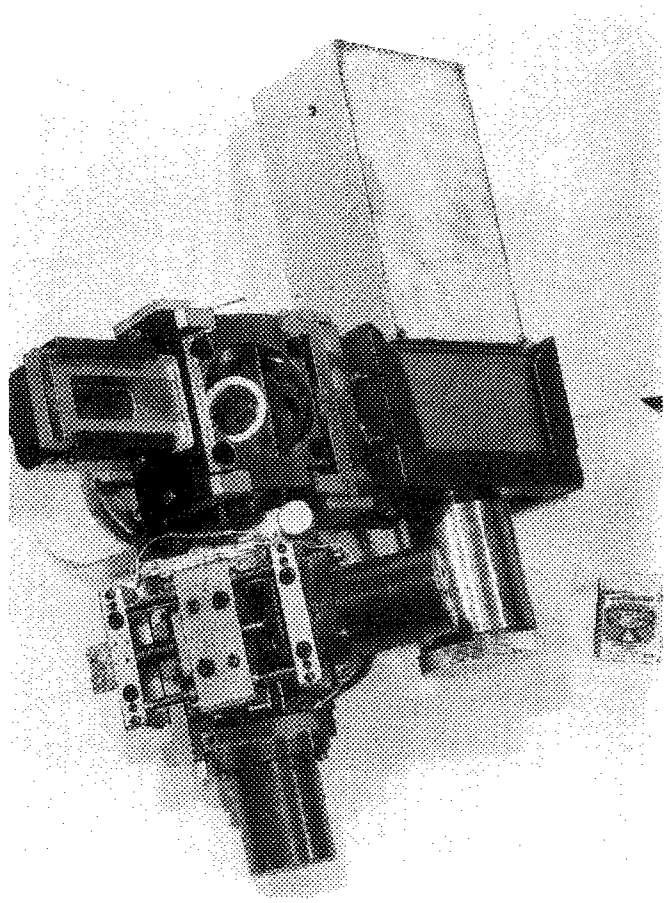

FIG. 1. The general view of the scanner.

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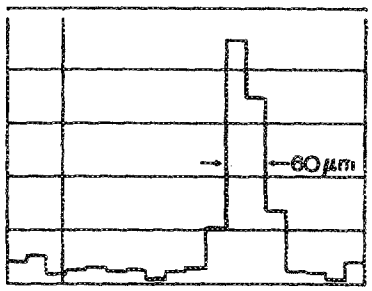

FIG. 2. The profile radiograph obtained during the intersection of a nickel wire of 50 μm diam. The scanning step is 30 μm and the effective diameter of the collimator is 40 μm.

tions within the 20×20 mm area. The scanner is motor driven, and two stepping motors for this purpose are computer controlled.

During the measurements, the following signals were monitored: (a) a signal from the ionization chamber placed in the monochromatized beam in front of the last collimator, (b) the area of Compton scattering peak due to the exciting radiation, and (c) the area of the fluorescent peak from a metallic foil located at the back of the sample being examined (substrate technique). The general view of the chamber with the sample, the scanner, and the ionization chamber is shown in Fig. 1.

II. RESULTS

As collimators, we have employed conic holes drilled in a 1-mm-thick lead sheet placed on a hard metallic plate. The effective diameter of such a collimator depends on the working range of energies and was determined experimentally, with respect to the broadening of the image lines when scanning a thin nickel grid (see Fig. 2).

To obtain a sufficient x-ray fluoresence intensity with good statistics, the x-ray spot size should be compromised with the incident x-ray intensity and the measurement time at a point, depending on the concentration of an analyte element. Without focusing x-ray optics, the spatial resolution was about 60 μm in the monochromatic beam and about 30 μm in the white SR beam for an exposure time of 1 s/point. The minimum detectable concentration of the elements ranging from Fe to Sr was 10 ppm at a measurement time of 1–3 s/point (with monochromatized excitation).

To study the element mapping, Cl to I, in minerals, we have prepared thin (0.3–0.5 mm) planar and parallel plates of up to 1-in. in diameter. During the scanning we managed to simultaneously identify eight elements with a Si(Li) semiconductor detector. Figure 3 illustrates the Fe and Sr maps in the rock section with garnet grains. The circular regions of high concentration of Sr is the result of segregation as the garnet crystals grow. The Sr concentration in the map varies from 10 to 300 ppm.

The two-dimensional Cl and I maps in the (002) cross sections of the pyrolytic graphite crystals, chemically treated by halogens, have been obtained with the same procedure. This technique has also been successfully applied to some biological problems, for example, to the measurement of the images of the lymphonodes in which a gold-containing colloid is introduced for x-ray contrast.

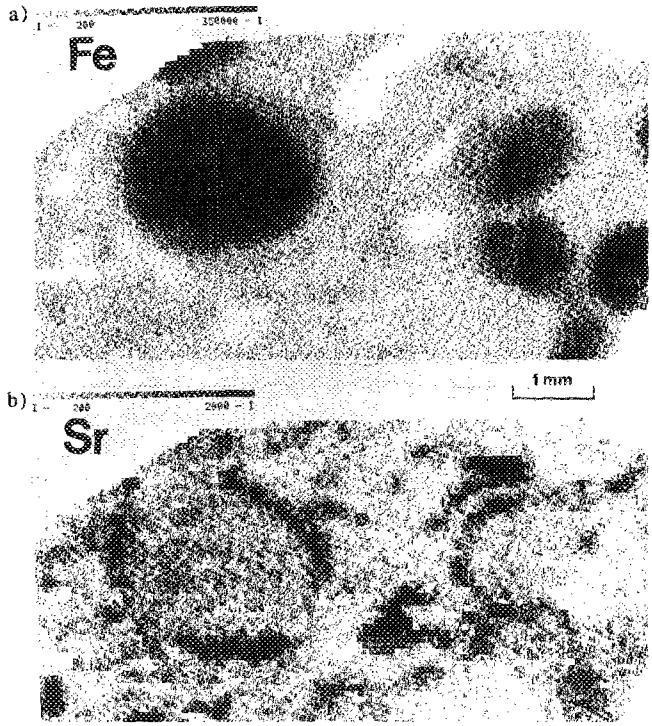

FIG. 3. Two-dimensional chart of ion (a) and strontium (b) distribution in the microsection metallographic specimen of a rock. The regions with a high content of iron are the garnet grains. 100×60 points; measurement time, 1.5 s/point; scanning step, 80×80 μm^2. Monochromatic excitation; $E = 20$ keV.

III. CONCLUSION

The insufficient efficiency of the planar crystal monochromator in the scheme described above imposes some restrictions on the spatial resolution and the sensitivity of the technique. After the installation of the short-focus monochromator made from the formed pyrolytic graphite (see Ref. 4) and a new scanner comprising the components manufactured by the firm Microcontrole (France) with a scanning field of 50×50 mm and 1-μm step, scanning radiographs with a spatial resolution of about 10 μm, and a sensitivity of up to 1 ppm are assumed to be obtained.

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