The essence of rocks

USP starts using a new electronic microprobe to analyze chemical elements in minerals

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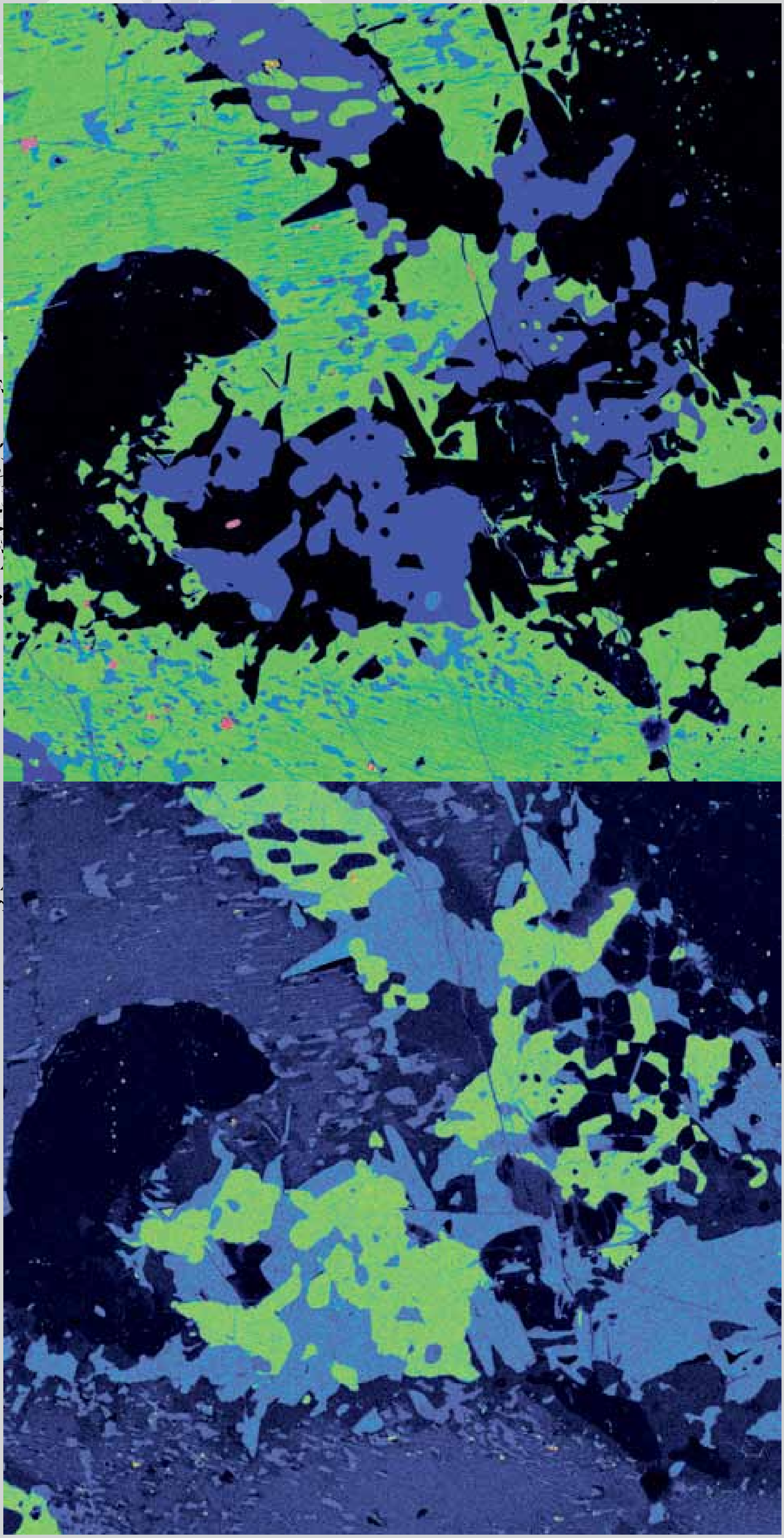
The newest version of a device that gained notoriety among geologists when Apollo astronauts brought rocks back from the Moon between 1969 and 1972 is now up and running in a custom-built facility at the Geosciences Institute (IGc) of the University of São Paulo (USP). The electronic microprobe is a research instrument capable of quickly identifying and quantifying the chemical elements in a mineral, which became important immediately after the Moon missions, when NASA sent samples to institutions around the world. Knowing whether a rock contains calcium, iron, or any type of rare earth is important not only for learning more about the geologic properties of a given place but also for determining whether any valuable materials for mining or other industrial purposes can be found there. This knowledge can also be used in metallurgy to analyze the components of metallic alloys or even to reveal the chemical details of how teeth are formed.

The equipment, purchased from one of its two manufacturers, the Japanese company Jeol (the other is Cameca in France), cost US$ 1.6 million and was fully funded by FAPESP. It will replace and upgrade a microprobe purchased in 1992 with money from a funding program by USP and the Inter-American Development Bank (IDB), with supplemental funding from FAPESP. The older device had replaced an American model from 1971, the first ever installed in Brazil, which was fully funded by the IDB. Its data-capturing process was complicated and hand-operated, and it recorded information on

IMAGES MICROPROBE LABORATORY AT IGC-USP



Images obtained
with the microprobe,
using wavelength-dispersive
X-ray spectroscopy.
A silicate mineral sample
with aluminum (in blue,
top image) and
calcium (in green, bottom
image) is shown

punch cards in a computer attached to the machine, which is still stored at the IGc. The oldest apparatus had three X-ray spectrometers, while the new one has five such spectrometers attached to the microprobe. This type of spectrometer analyzes the chemical elements present in a mineral by reading the wavelength generated by an electron gun when the beam hits the sample. The result is X-ray radiation with the specific wavelength irradiated by the material under analysis. The wavelength is captured by a crystal inside the microprobe, which matches it to a chemical element and recognizes its intensity at specific points of the sample.

"Operating the first device was very difficult, and preparing and analyzing the sample was a complicated and lengthy process; the second one already had five spectrometers and the most recent one allows a more automated analysis, with better resolution in the graphical interface and brighter images. It will be possible to get better quality photos of minerals such as those containing manganese or cadmium, which can emit light when an electron beam is aimed at them. Also important is the fact that the vacuum conditions in which the electron beam is emitted are more sophisticated now, without the electrons being absorbed by air molecules before they hit the sample," says Celso de Barros Gomes, Professor Emeritus of the IGc, who installed and directed the Institute's Electronic Microprobe Laboratory and was responsible for purchasing the three devices in 1971, 1992 and 2012. "It's a historical cycle," he says.

The microprobe is used by master's and doctoral students, but is also used in studies by companies, including Vale and Petrobras

SAND CUSHION

To house the new microprobe, a new, 90-m2 laboratory had to be built on the ground floor of the IGc. The new equipment is more sensitive, and it required a setting in which it would receive no interference from the magnetic fields produced by the institute's other equipment. The microprobe stands 1.8 meters high and is connected to a table with supporting equipment and three viewing screens on which the results can be observed. The apparatus sits on a type of cushion, consisting of a sand-filled hole that is one meter deep and was designed to absorb the vibrations produced by automobile traffic near the building. The microprobe is also encased in a Faraday cage, a type of metal framework that provides electrostatic shielding.

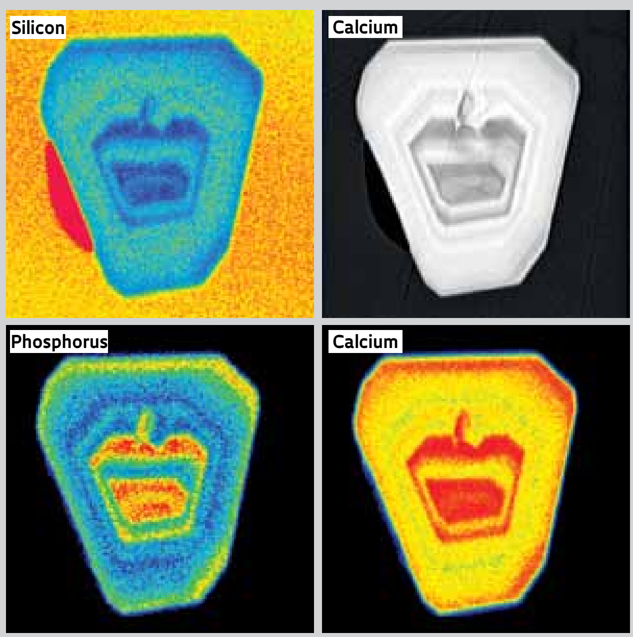
Opened on December 14, 2012, the new laboratory is ready to efficiently receive the partners and inquiries that used to be directed at the older microprobes. "We provide services to mining companies like CPRM and especially Vale, which demands a wide diversity of mineral products," Gomes says. "We also conduct studies for Petrobras." The fees for these services are used to maintain the laboratory. However, the microprobe's most frequent users over the years, according to Professor Celso Gomes, are master's and doctoral students. They schedule dates and times to use the equipment, coming not only from USP but also from the Universidade Estadual Paulista (Unesp), the State University of Campinas (Unicamp), and the federal universities in the states of Paraná (UFPR), Pernambuco (UFPE) and Bahia (UFBA). "We also receive researchers from several Latin American countries and from Mozambique and Angola, in Africa," says Gomes. "With the new equipment, I would like to make arrangements for studies in odontology, to analyze the distribution of chemical elements in teeth and study how nature handles teething," he affirms.

"Having a new microprobe is excellent," says Professor Marcos Aurélio Farias de Oliveira of the Geosciences and Exact Sciences Institute at the Universidade Estadual Paulista (Unesp), in the city of Rio Claro. "It has more features and enables us to complete experiments in less time. Its predecessor was already quite good, but it had to be shut down for maintenance more often and the waiting

Element composition

Microprobe images show chemical zoning within a mineral

On the right is a grain of britholite, a mineral that contains calcium phosphate. The colored X-ray images quantify the presence of specific chemical elements. The shades of gray show the presence of calcium in the darker areas



5μm (micrometers)

LESS MORE

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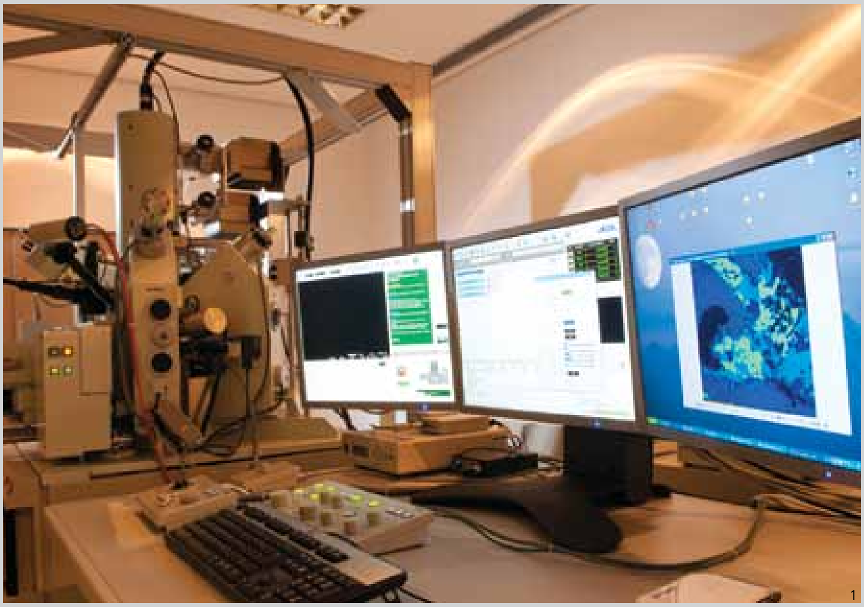
PHOTOS EDUARDO CESAR



1 Microprobe (in the back) and the screens and devices that make up the full apparatus

2 Glass slide with rock substrate prepared for analysis

line could take up to six months," says Oliveira. "For about three years, it was the only one in the country available for academic studies, as all the other ones, like the one at the University of Brasília [UnB], were out of order," he explains. He mentions that the Federal University of Rio de Janeiro (UFRJ) and UnB recently bought their own microprobes and that Unesp has already approved the purchase of one, to be funded by the Brazilian Innovation Agency (Finep), under the Ministry of Science and Technology.

GENETIC INFORMATION

The central purpose of academic microprobes is training new geologists, as this type of equipment is also used by major industries such as metallurgy. In addition, training qualified professionals is especially important when companies such as Petrobras and Vale are expanding. "Element quantification is the driver of geology. Quantification makes it possible to determine the pressure and temperature conditions at which these minerals were formed, many kilometers under the surface. This is like genetic information about the chemical composition of the material you are analyzing," says Gomes. In applications related to ore content, this and other information obtained using the microprobe is very important in making an accurate assessment of a mine's potential yield. The data also support its installation and indicate the necessary infrastructure for a mining operation.

> "Today the microprobe is part of the scientific culture of geology," says Celso de Barros Gomes, from USP

To begin to analyze rock samples collected in the field, microprobe users prepare their specimens — with the help of a technician, in USP's case — by cutting them into slices just micrometers thick (a micrometer is one thousandth of a millimeter). This thin layer of rock is attached with transparent glue to a glass slide, which is inserted in the microprobe. Then, the specimen is worn down until it is practically incorporated into the glass. The sample is first examined under an optical microscope to measure its thickness, which should be in the range of 30 to 40 micrometers. It is subsequently placed in a device called a metalizer, where it receives a coating of carbon that turns the material into a conductor.

"When we have an unknown sample whose minerals we have not identified, we use the technique called energy-dispersive spectroscopy (EDS), which allows us to scan for every chemical element in the periodic table and specify if that material is a feldspar, for instance, or some other type of mineral," says Professor Celso Gomes. It is possible to identify a mineral based on its chemical composition. To quantify elements or determine the percentage of specific elements such as silicon, iron, aluminum, or magnesium in each rock, the geologists set the microprobe to use wavelength dispersive spectroscopy (WDS), which not only meets that main objective but can also indicate the conditions under which the material was formed.

"Today, these techniques are well known and disseminated, but in the past, when microprobes first became commercially available in the 1960s and 1970s, it was difficult to convince the academic community of their importance. And the fundamental effect of that persuasion was to disseminate the technique through data presented in congresses and conferences, as well as courses and internships, and through the publication of papers," says Gomes. "We started a persuasion process forty years ago and now the microprobe is part of the scientific culture of Brazilian geology," he affirms. ■

Project

MUE: Acquisition of a new electronic microprobe for the Geosciences Institute at USP – No. 2009/53835-7; **Grant mechanism** Multi-User Equipment Program; **Coordinator** Celso de Barros Gomes – USP; **Investment** R$473,729.58 and US$ 1,662,330.00 (FAPESP).