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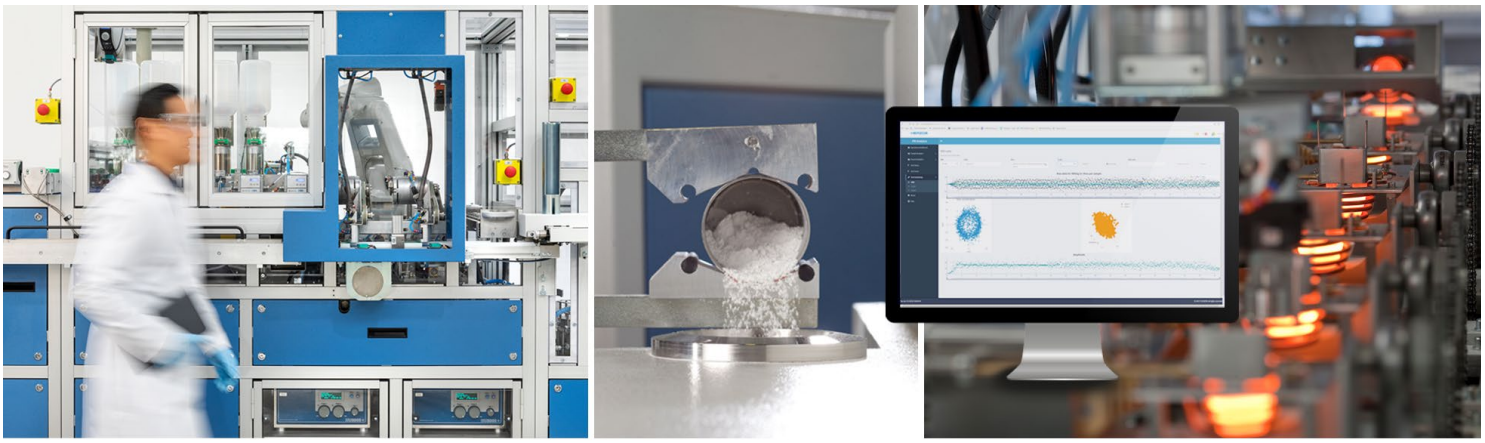

Automated sample preparation of Garnet and Titanium bearing Rutile as a pressed pellet for XRF analysis

Abstract

Metamorphic eclogite deposits are often sources of two primary raw materials: Garnet and Rutil/ Titanium. Extracted from host rock, both raw materials are used in various industrial applications. Here we present primary test results obtained during a basic application development. The analysis during the raw material beneficiation is often carried out using X-ray fluorescence and X-ray diffraction. In both cases, the sample is prepared as a pressed pellet (51.5 mm steel ring).

Key words

• Garnet • Rutile • Grinding • Pressing • Automation • HP-MP • XRF

Introduction

Rutile is a primary source of titanium, which is used in many specialized applications. It is well known as a pigment in paints and as a light but very durable metal that is for example used in aircraft engines. Due to its particularly high Mohs hardness (6.5 up to 7.5), garnet is used as an abrasive, in sandblasting, water cutting and in filters for water treatment. In order to obtain the raw materials, the ore is mined, crushed and often enriched via density separation using spiral separation and flotation. Analysis must be carried out routinely in all processing steps, to achieve energy efficient optimized process control and to finally identify the product qualities.

Fast and cost-effective preparation of samples for X-ray fluorescence can be achieved using the HP-MP mill and press combination (see figure 2). This allows the sample material to be efficiently crushed and pressed into a steel ring.

Methods and Results

The automated mill and press HP-MP is a reliable machine that combines both sample preparation steps in one system. With its small footprint and functionality, it is an optimal solution for reproducible and contamination-free sample preparation. Due to the specific hardness of the sample material and the element to be analyzed, only a tungsten carbide grinding vessel can be used.

As in addition to X-ray fluorescence supplementary X-ray diffraction is also applied, therefore it is recommended that the HMPA100 grinding aid is used. This resin-based grinding aid is characterized by its good binding properties and its particularly low measurement background. The target grain size for an optimal XRF measurement was 100% smaller than 75 µm after grinding.

Since the chemical composition of the provided three sample types was unknown, a smart standardless analysis of the samples has been done using an ED-XRF spectrometer. The samples contained two similar garnet samples and one rutile matrix.

Based on the obtained chemical data a basic calibration with fixed measurement conditions has been set up. This enabled reproducibility testing of the sample preparation.

To avoid cross contamination between both matrixes (high Titanium with 95% and less than 1 %) a sand cleaning needed to be interposed between both sample types.

In order to determine the reproducibility of the sample preparation method, 10 duplicates have been prepared from each sample type. Since both garnet samples are very similar in their composition only one is presented here (Table 1).

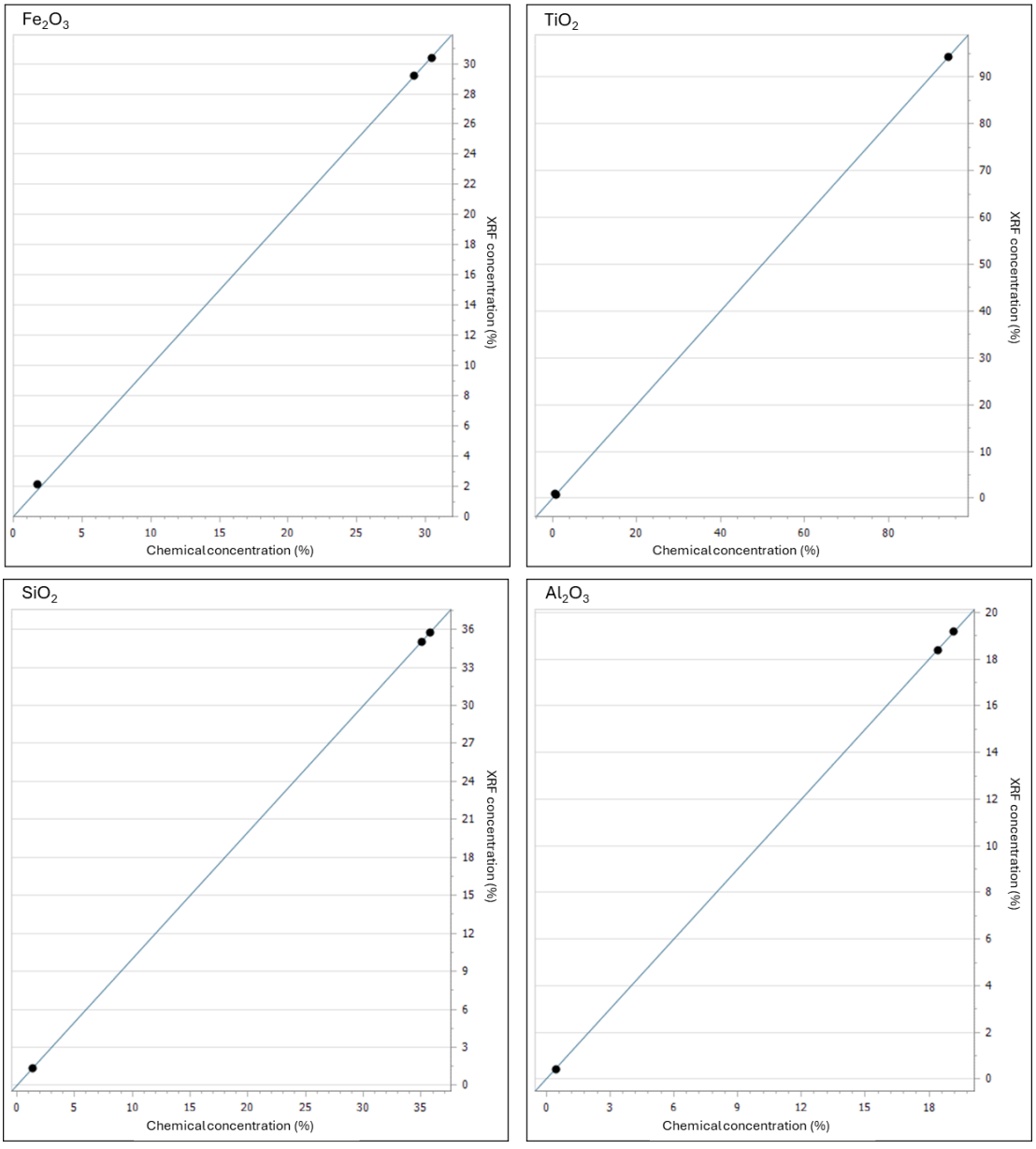

Figure 1: *Exemplary three-point calibration curves for Fe2O3, TiO2, SiO2 and Al2O3. This basic calibration has been used for the reproducibility testing.*

Sample ID	Na_2O (%)	MgO (%)	CaO (%)	K_2O (%)	TiO_2 (%)	Mn_2O_3 (%)	Fe_2O_3 (%)	SiO_2 (%)	Al_2O_3 (%)	SO_3 (%)
Garnet	0,81	5,48	8,86	0,08	1,01	0,42	29,3	36,21	18,7	0,06
Garnet	0,79	5,48	8,88	0,08	1,03	0,41	29,26	36,35	18,63	0,06
Garnet	0,77	5,26	8,81	0,08	1,01	0,41	29,38	36,08	18,52	0,06
Garnet	0,78	5,24	8,77	0,07	1,01	0,42	29,36	35,89	18,34	0,06
Garnet	0,77	5,27	8,83	0,07	1,02	0,42	29,36	35,97	18,46	0,06
Garnet	0,75	5,25	8,83	0,08	1,01	0,42	29,36	35,99	18,5	0,06
Garnet	0,77	5,31	8,83	0,08	1	0,42	29,34	36,14	18,56	0,06
Garnet	0,74	5,41	8,85	0,07	0,98	0,42	29,42	36,2	18,66	0,06
Garnet	0,74	5,25	8,81	0,08	0,99	0,42	29,47	35,97	18,56	0,06
Garnet	0,81	5,48	8,86	0,09	0,99	0,42	29,33	36,26	18,62	0,06
Average	**0,77**	**5,34**	**8,83**	**0,08**	**1,01**	**0,42**	**29,36**	**36,11**	**18,56**	**0,06**
Standad deviation	**0,02**	**0,10**	**0,03**	**0,01**	**0,01**	**0,00**	**0,06**	**0,14**	**0,10**	**0,00**

Sample ID	Na_2O (%)	MgO (%)	CaO (%)	K_2O (%)	TiO_2 (%)	Mn_2O_3 (%)	Fe_2O_3 (%)	SiO_2 (%)	Al_2O_3 (%)	SO_3 (%)
Rutile	0	0,26	0,37	0,08	94,32	0,06	1,36	1,37	0,68	0,56
Rutile	0,02	0,27	0,32	0,05	94,52	0,06	1,37	1,33	0,57	0,55
Rutile	0,01	0,24	0,32	0,05	94,42	0,06	1,36	1,3	0,57	0,55
Rutile	0,04	0,24	0,32	0,05	94,54	0,06	1,37	1,32	0,56	0,55
Rutile	0	0,25	0,32	0,05	94,43	0,06	1,36	1,32	0,56	0,55
Rutile	0	0,24	0,32	0,04	94,46	0,06	1,41	1,34	0,57	0,56
Rutile	0,01	0,23	0,32	0,05	94,49	0,06	1,38	1,31	0,55	0,55
Rutile	0,01	0,24	0,32	0,04	94,51	0,06	1,37	1,38	0,57	0,55
Rutile	0,08	0,32	0,33	0,08	94,42	0,06	1,39	1,23	0,58	0,56
Rutile	0,01	0,24	0,32	0,04	94,56	0,06	1,37	1,36	0,57	0,55
Average	**0,02**	**0,25**	**0,33**	**0,05**	**94,47**	**0,06**	**1,37**	**1,33**	**0,58**	**0,55**
Standad deviation	**0,02**	**0,02**	**0,01**	**0,01**	**0,07**	**0,00**	**0,01**	**0,04**	**0,03**	**0,00**

Table 1: *Individual results of the element concentration from the test series with ten samples of the same material for a Garnet and Rutile matrix. The results were obtained with an ED-XRF with basic calibration.*

Discussion

The results of this study clearly demonstrate that the automated sample preparation with the HP-MP delivers good repeatability for both samples types. The main compound of the garnet sample SiO2 (average concentration 36.11%) has a standard deviation of 0.14, followed by Fe2O3 (average concentration 29.36%) with a standard deviation of 0.06 and for Al2O3 (average concentration 18.56%) with a standard deviation of 0.10.

The rutile sample is mainly composed of TiO2 with an average concentration of 94.47%. Here a standard deviation of 0.07 was obtained for the tested sample set. Minor present elements like SiO2, CaO, Fe2O3 with an average concentration of less than 1.5 % have a standard deviation of maximum 0.04.

Figure 2: *Mill and press combination HP-MP for the preparation of pressed pellets suitable for XRF analysis.*

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