Characterization of Minerals, Metals, and Materials 2023

EDITED BY

Mingming Zhang Zhiwei Peng Bowen Li Sergio Neves Monteiro Rajiv Soman Jiann-Yang Hwang Yunus Eren Kalay Juan P. Escobedo-Diaz John S. Carpenter Andrew D. Brown Shadia Ikhmayies





The Minerals, Metals & Materials Series

Mingming Zhang · Zhiwei Peng · Bowen Li · Sergio Neves Monteiro · Rajiv Soman · Jiann-Yang Hwang · Yunus Eren Kalay · Juan P. Escobedo-Diaz · John S. Carpenter · Andrew D. Brown · Shadia Ikhmayies Editors

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Cover Illustration: From Chapter "Structural Characterization of Europium-Doped BaTiO₃ Obtained by Solid-State Reaction Synthesis", J. P. Hernández-Lara et al., Figure 5: Micrograph of the sample x = 0.007% by weight Eu³⁺ sintered at 1300 °C. https://doi.org/10.1007/978-3-031-22576-5_56.

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Preface

Materials characterization is a vital part of all science and engineering practice, as it is a fundamental process to achieving a good understanding of the processingmicrostructure-property relationship. Various advances in characterization techniques and instruments in recent years have contributed, in a significant way, to an in-depth knowledge of the material's properties and structure on different scales. This enhanced understanding has also profoundly impacted the efficiency of existing industrial processes and how minerals, metals, and materials are applied in many fields.

This year, the Characterization of Minerals, Metals, and Materials symposium received 146 abstract submissions, of which 62 papers were accepted in 8 technical sessions. This symposium is among one of the largest in the TMS Annual Meeting & Exhibition. The proceedings volume includes state-of-art techniques used in modern minerals, metals, and materials characterization and the latest research in materials engineering and technologies. This proceedings publication is a valuable reference for academic scholars and industry professionals interested in advanced characterization methods and instrumentations that cover a wide range of research subjects. Readers will enjoy the diversity of topics in this book with innovative approaches to process and characterize materials at various scales and levels.

The Characterization of Minerals, Metals, and Materials 2023 symposium is sponsored by the Materials Characterization Committee under the Extraction & Processing Division (EPD) of TMS. The main focuses of this symposium include but are not limited to advanced characterization of Extraction and processing of minerals, process-microstructure-property relation of metal alloys, ceramics, polymers, and composites. New characterization methods, techniques, and instrumentations are also emphasized.

As a lead organizer of this symposium, I would like to take this opportunity to express my sincere gratitude to all authors for their contribution and generosity to share their research work. On behalf of the organizing committee, I would like to thank TMS for providing us with the valuable opportunity to publish this standalone proceedings volume. Much appreciation is also extended to the EPD for sponsoring this symposium.

Most importantly, the success of this proceeding's publication would not be possible without the fabulous contribution and support from all members of the Materials Characterization Committee. I also would like to thank our publisher, Springer, for their timely and quality publication of this book.

> Mingming Zhang Lead Organizer

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Dr. Peng has published over 190 papers, including more than 140 peer-reviewed articles in journals such as International Materials Reviews; Journal of Hazardous Materials; ACS Sustainable Chemistry & Engineering; Resources, Conservation & Recycling; Journal of Cleaner Production; Waste Management; Metallurgical and Materials Transactions A; Metallurgical and Materials Transactions B; JOM; Journal of Power Sources; Fuel Processing Technology; Energy & Fuels: IEEE Transactions on Magnetics: IEEE Transactions on Instrumentation and Measurement; Ceramics International; Powder Technology; and Separation and Purification Technology. He holds 65 Chinese patents and has served as an associate editor for Mining, Metallurgy & Exploration, as a guest editor for JOM and Metals, and as an editor for PLOS ONE. He has also been a member of editorial boards of Scientific Reports, Journal of Central South University, and Journal of Iron and Steel Research International, and has served as a reviewer for more than 70 journals.

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Shadia Ikhmayies received a B.Sc. and M.Sc. from the physics department at the University of Jordan in 1983 and 1987, respectively, and a Ph.D. in producing CdS/CdTe thin film solar cells from the same university in 2002. Her research is focused on producing and characterizing semiconductor thin films and thin film CdS/CdTe solar cells. She also works in characterizing quartz in Jordan for the extraction of silicon for solar cells and characterizing different materials by computation. She has published 59 research papers in international scientific journals, 85 research papers in conference proceedings, and 3 chapters in books. She is the founder and editor of the eBook series Advances in Material Research and Technology published by Springer, and the editor-in-chief/editor of several books. Dr. Ikhmayies is a member of The Minerals, Metals & Materials Society (TMS) where she was the chair of the TMS Materials Characterization Committee (2016-2017), and the leading organizer of three symposiums;

Solar Cell Silicon 2017-2020, Mechanical Characteristics and Application Properties of Metals and Nonmetals for Technology: An EPD Symposium in Honor of Donato Firrao, and Green Materials Engineering: An EPD Symposium in Honor of Sergio Monteiro. Dr. Ikhmavies is also a member of the World Renewable Energy Network/Congress (WREN/WREC) 2010present. She is a member of the international organizing committee and the international scientific committee in the European Conference on Renewable Energy Systems (ECRES2015-ECRES2022). She is a guest editor and a member of the editorial board of several journals including JOM and the Journal of Electronic *Materials*. Dr. Ikhmavies is a reviewer of 24 international journals and several international conference proceedings. She has received several international awards including the TMS Frank Crossley Diversity Award 2018 and World Renewable Energy Congress 2018 Pioneering Award.

Part I Advanced Characterization Methods I

Characterization of Lunar and Martian Meteorites Using a Scanning Electron Microscope (SEM)



Hussain Al Halwachi

Abstract Lunar and Martian meteorites are rare rocks found on Earth and classified by meteoritical experts depending on their physical and chemical features. Three different types of certified lunar rock samples and one Martian rock sample were tested and scanned by a scanning electron microscope (SEM) to obtain the chemical composition, explore the elemental mapping, and provide a statistical comparison between the different types. The SEM was able by EDS detector to provide accurate chemical compositions, in addition to microstructure images and elements mapping. The data provided by SEM were treated statistically by the principal component analysis (PCA) technique to obtain the relation between the elements and obtain a statistical model to help in differentiating similar rocks in the future.

Keywords SEM · Lunar · Martian · PCA

Introduction

The Moon and Martian meteorites are considered among the rarest space rocks available on Earth. They usually blast off from the surface of the Moon and Mars due to collisions occurring with possible asteroids and then are captured by Earth's gravity until they fall on Earth's surface after penetrating the atmosphere [1]. Meteorite classification and identification became a crucial step in the past decade since a lot of people are interested to own a genuine piece from space. The classification is carried out by international meteoritical societies and throughout the years an international database was generated for the certified meteorites [2]. The classification is carried out by experts in this field. The sorting and classification of meteorites are carried out by scientists depending on the meteorites' mineralogy and structures [2], using several analytical tools and isotope analysis [2]. In this study, I am exploring the chemical compositions of four certified meteorite samples that had been bought from Mr. Mendy Ouzillou, a trusted dealer in this field and recommended by the

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Meteoritical Society in the United States. Since the moon is not a uniform, homogeneous planet, and consists of different rocks formed in different ways and times [3], I had selected three genuine lunar samples carefully from different kinds to have a variety of Moon meteorites, in addition to a sample for Mars meteorite. The four samples measured by scanning electron microscope (SEM) and the data provided were treated statistically by principal component analysis (PCA) to obtain a better comparison between the different types of Meteorites.

Four Meteorite Samples

The four certified meteorite samples were selected from different kinds of rocks, to allow me to study different rock natures and chemical compositions. The size and dimension of each sample were important criteria for selection. Smaller size samples will not be prepared and fit easily in SEM instrument. The price was also one of the selection criteria since the genuine certified meteorite samples are not cheap. I could not add more samples due to the high price of some rare pieces, i.e., Zagami Shergottite piece. The samples selected were big enough in size to take three SEM images for each sample with the flexibility to select the best position for mapping and measurements.

(1) NWA 6963 Martian Shergottite

Meteorite Northwest Africa (NWA) 6963 is a gabbroic Shergottite rock classified as a basaltic Martian meteorite [4]. The NWA 6963 was found in Guelmim Es-Semara, Morocco in 2011 [5], and based on its bulk chemistry and oxygen isotopes, it was classified as a Martian meteorite [4]. The mineralogical model of NWA was containing Pyroxene, Plagioclase, Opaques, and Melt pockets with no ferroan Olivine. The absence of olivine in Shergottite rocks indicates that they crystallized from fractionated magmas [6]. Shergottites are the most abundant of Martian rocks (50 out of 61), almost three-quarters of all Martian meteorites [7]. The name Shergottites is given after five Shergotties meteorites, which fell in India in 1965 in an area called Shergotty [2]. Figure1 shows the four meteorite samples.

(2) Lahmada 020, Lunar rock

Meteorite Lahmada 020 is a lunar meteorite that was found in Saguia el Hamra, Western Sahara in Morocco in November 2018 [5]. The rock was classified as feldspathic regolith melt breccia. The feldspathic lunar meteorites likely originated from the inner and outer regions of Fledspathic Highlands Terrane (FHT) on the Moon [8]. Furthermore, the lunar surface is covered by a layer of unconsolidated debris known as lunar regolith [3, 9]. Lunar regolith are rocks that were broken from underlying bedrock by collision with meteorites [1]. Lunar regolith meteorite is ejected from the Moon by the impact of the cratering process [1]. This type of meteorite reflects a broad range from different lunar lithological terraces [10], including FHT [1]. One type of lunar lithologies is





known as lunar regolith melt, which is formed when a meteorite impact melts the material, and then ejected from a crater, then solidifies before they land on earth [3, 11]. The geochemistry of Lahmada 020 rock includes Olivine, Pigeonite, and anorthite [5].

(3) NWA 11474 Lunar

Meteorite (NWA) 11474 was found in Northwest Africa in 2017 and classified as lunar feldspathic breccia [5]. The geochemistry includes Olivine, Pigeonite, pyroxene, and Plagioclase [5].

(4) Meteorite NWA 10203

Meteorite (NWA) 10203 was found in Northwest Africa in 2015 and classified as Polymict lunar [5]. Polymict (or polylithologic) breccias are a mixture of many different types of rocks, which result from the mixing of different lithologies formed under different conditions [9]. The Geochemistry of NWA 10203 includes Olivine gabbro, clinopyroxene, and plagioclase [5].

Experimental Method

SEM is a non-destructive technique used to magnify images up to at least 1,000,000X times [12]. SEM was used to produce magnified images for the four meteorite samples. The samples were fitted with special clamps to avoid destruction during measurement under a vacuum medium. Micro-mapping of the sample was carried out to determine the microtopographic locations of each element. SEM is equipped with an EDS detector that can produce quantitative analysis. The measurements were carried out in Louvre Museum Laboratory in Abu Dhabi, using SEM instrument from Thermo Fisher company (Fig. 2 and 3).



Fig. 2 SEM image for sample NWA 6369 overlapped with EDS maps, showing the microtopographic locations of the elements



Fig. 3 EDS maps, the elementary micro-mapping of main elements for sample NWA 6369

Results

The main elements found in the four samples are oxygen, silicon, iron, carbon, calcium, magnesium, and aluminum, while other elements are found in the trace levels. The level of oxygen found in the four samples is from 40 to 45.9%, which is

NWA 6963- Martian			Lahmada 020				
Element	Image1	Image 2	Image3	Element	Image1	Image 2	Image3
	Weight %	Weight %	Weight %		Weight %	Weight %	Weight %
0	42.0	43.0	42.9	0	42.8	42.6	43.9
Si	18.3	18.6	17.3	Si	18.6	15.4	18.3
Fe	16.2	16.2	14.3	Fe	4.8	3.6	2.9
С	8.5	8.7	9.0	C	5.7	9.7	5.9
Ca	5.5	4.6	6.1	Ca	9.4	13.1	11.2
Mg	5.2	5.4	4.6	Mg	3.7	1.8	1.8
Al	1.9	1.0	2.7	Al	13.7	12.8	15.2
Na	0.7		1.0	Ba	0.6		
Р	0.6	0.6	0.6				
Mn	0.5	0.5					

Table 1 Semi-quantitative EDS analysis for NWA 6963 and Lahmada 20

expected since minerals always occur in oxide form and exist in crystalline form. The level of silicon was almost around 18% in NWA6963, Lahmada 020, NWA10203, and lower than 18% in NWA11474. The level of iron was one of the characteristic elements for each sample. Iron was high in NWA 6963 at the level of 15%, and at the level of 10% in NWA10203, while it was lower in Lahmada 020 and NWA11474. Carbon: the level of Carbon differed from one sample to another. It was high in NWA11474 at around 20% and lower in NWA 6963 and Lahmada and no carbon was detected in NWA 10203. Lahmada 020 and NAW 10203 contained a high level of calcium (higher than 10%) compared with NWA 6963 and NWA 11474. The level of magnesium was almost lower than 4% in Lahmada 020 and NWA 11474, and slightly higher in NWA6963 and NWA 10203. The level of aluminum was characteristic for NWA 6963. The level in NWA 6963 was low compared with the other three samples (Tables 1 and 2).

PCA Multivariate Statistical Analysis

In order to identify the similarities and differences between the four meteorite samples, an unsupervised type of statistic was applied. It should be remembered that an unsupervised statistical technique allows a display of multivariate data (multiple variables) and allows for the identification of groups within the proposed data set. For our research, we have a data set consisting of 15 variables (O, Si, Fe, C, Ca, Mg, Al, Na, P, Mn, K, S, Ba, Ni, Sn). The samples consist of 3 Moon rocks and one Mars rock. The three Moon rocks are from different types of lunar rocks. Before starting the statistical treatment, the dataset was cleaned and the missing values were