Editorial for Special Issue "Nanomineralogy"

Yiwen Ju [1],*, Quan Wan [2] and Michael F. Hochella, Jr. [3]

[1] Key Laboratory of Computational Geodynamics, College of Earth and Planetary Sciences,
    University of Chinese Academy of Sciences, Beijing 100049, China
[2] State Key Laboratory of Ore Deposit Geochemistry, Institute of Geochemistry, Chinese Academy of Sciences,
    Guiyang 550081, China; wanquan@vip.gyig.ac.cn
[3] Department of Geosciences, Virginia Polytechnic Institute and State University, Blacksburg, VA 24061, USA;
    hochella@vt.edu
* Correspondence: juyw03@163.com

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Nanoscience and nanotechnology study the properties of materials within the range 0.1~100 nm. Materials at the nanoscale have special physicochemical properties owing to their surface and interface, small size, quantum tunneling, and quantum size effects. Nanotechnology has crossed over into many areas of Earth science, driving important advances in mineralogy, petrology, geochemistry, structural geology, energy geology, mineral deposits, geologic hazards, and environmental geology [1–7]. Studying problems and phenomena at the nanometer scale, which reveals mechanisms as well as processes, is the inevitable future of developments in Earth science. Nanogeosciences are the global frontier for the contemporary cross-development of geosciences and nanotechnology, and the rise of nanoscience is set to bring about a revolutionary leap in the development of Earth science in the 21st century, driving breakthroughs in Earth science on the ultra-micro scale [1–3].

Nanomineralogy, an important aspect of nanogeoscience, is characterized by high-resolution transmission electron microscopy (HRTEM); scanning probe microscopy (SPM); and other high-resolution microscopies, several of them synchrotron-based, in combination with an array of spectroscopic and gas adsorption methods. It focuses on the composition, structure, and physical and chemical properties of nanoscale minerals and their interrelations with other Earth critical components [2–4], involving everything from nanominerals and geochemistry, mineral nanostructures, and nanomineral deformation to nanopores in oil and gas reservoirs, nanomineral deposits, and nanomineral materials. The exploration and application of the physical and chemical properties of nanomineral resources and environment broaden mineralogy's scope for the development of practical applications as well as the prospects of such applications.

Nanominerals have both resource and environmental attributes [8–11] and offer an extensive record of nanoscale substances and geological processes. With recent developments in nanoscience, research into nanomineral particles, nanomineral solids, nanomineral structures, and resources and the environment have also deepened [1–13]. In many cases, nanominerals or minerals with nanostructures appear in aggregate form. These include mineral particles with a grain size at the nanoscale, minerals of one-dimensional nanostructure (such as halloysite of nanotube structure), and layered minerals of two-dimensional nanostructure (such as clay minerals). Studies of actual minerals by HRTEM, STM, and AFM have demonstrated the existence of nanoparticles and nanostructures, with such nanophenomena more common in crystal surfaces and interfaces. The special nanostructure and composition of a crystal's surface determine its surface properties, which are significant in discussing the physical and chemical environment for the formation and genesis of minerals. Between two mineral phase transitions, not only do nanoscale particles exist, but also nanoparticle aggregates appear under particular physical and chemical conditions—including clay minerals, zeolites, colloidal minerals, volcanic glass, meteorite glass, fusion crust, and tectonite—and fall primarily into four categories:

(1) clay minerals, (2) quasicrystal nanostructures, (3) nanostructures in colloids, and (4) nanostructures in crystalline rocks. Minerals with grain size larger than 1 μm can provide information about minerals' late growth stage, whereas those with grain size between 0.1 and 100 nm can provide records about their initial crystallization. The polymorph, polytype, multi-body, and micro-intergrowth of minerals at the nanoscale are the actual minimum bodies capable of reflecting geological information at present. Accordingly, this kind of information must be integrated to more fully reflect the physical and chemical environment in which minerals were formed. At present, the active exploration of techniques and methods for developing and using nanoscale minerals, together with the quest for breakthroughs in both theory and practice, is heightening mineralogists' ability to understand and transform nature [2,4].

Some of the papers [14–20] in this issue deal with nanominerals and geochemistry. They describe the morphology and structural phase transition of various natural nanominerals, as well as the experimental synthesis of nanomineral crystals; the adsorption, percolation, dissolution, and metasomatism of nanominerals; and the environmental transformation conditions of nanomineral composition. Hong and coauthors [14] have reported two structural phase transitions and metallization for nanocrystalline rutile using a diamond anvil cell at ~7.2, ~12.3, and ~14.5 GPa. During compression, a structural phase transition from rutile to baddeleyite occurs, with the high electrical conductivity value offering a crucial clue regarding metallization, as confirmed by temperature-dependent electrical conductivity measurements. On decompression, a structural phase transformation from baddeleyite to columbite occurs. HRTEM observations regarding the starting and recovered samples indicate that phase transformations from rutile to baddeleyite to columbite are irreversible under high pressure. Bjorn von der Heyden and coauthors [15] investigated analytical approaches to characterizing natural Fe nanoparticles before detailing a dedicated synchrotron-based X-ray spectromicroscopy investigation into the speciation of suspended Fe nanoparticles collected from fluvial, marine, and lacustrine surface waters. They identified ferrous, ferric, and magnetite classes of Fe nanoparticles (10–100 nm), all of which exhibited a high degree of heterogeneity in the local bonding environment around the Fe center. The results provide an important baseline for natural nanoparticle speciation in pristine aquatic systems, highlighting the degree of interparticle variability. Nie and coauthors [16] employ such an experimental approach to study the facile hydrothermal synthesis of nanocubic pyrite crystals using greigite (Fe_3S_4) and thiourea (NH_2CSNFH_2) as precursors. Nanocubic pyrite (FeS_2) crystals with exposed (100) crystal faces and sizes of 100–200 nm were successfully synthesized via a facile hydrothermal method using greigite as the iron precursor and thiourea as the sulfur source. These results demonstrate the critical influence of sulfur source on pyrite morphology. These findings also provide new insights into the formation environments and pathways of nanocubic pyrite under hydrothermal conditions. Gu and coauthors [17] explore the variability in rare earth elements (REEs) and trace metal concentrations in dolostone samples of the Cryogenian Nantuo Formation of the Neoproterozoic Era, taken from a drill core in South China. Petrological evidence suggests that the dolostone in the Nantuo Formation was formed in nearshore waters. All the examined dolostone samples featured a significant enrichment of manganese and middle rare earth elements (MREEs), suggesting that the dolostone samples were deposited from suboxic to iron-enriched and anoxic waters. The MREE-enriched dolostone were transported by colloids and nanoparticles in meltwaters. Liu and coauthors [18] report the results of the geochemical alteration and mineralogy of coals under the influence of fault motion. Five coal samples were carefully collected from a reverse fault zone in Qi'nan colliery, China. Systematic detection methods were employed to analyze the different chemical and physical characteristics of the fault-related coal samples. The frictional heat and strong ductile deformation generated by the fault motion led to the dissociation of phenol and carboxyl groups in coal molecules, which sharply decreased the concentrations of elements Co and Mo bound to these functional groups. The disruption and delamination of laminar clay minerals by strong compression–shear stress significantly increased the adsorption sites for related elements, especially heavy rare earth elements (HREE) and MREE. Nanoscale clay minerals resulting from stress-induced scaly exfoliation also enhanced the retention capability of REE. Zhang and coauthors [19] present a

model for the metal-bearing nanoparticles observed in soils and fault gouges over the Shenjiayao gold deposit and their significance. Their findings indicate that ore-forming elements in soils can come only from deep-seated ore bodies and that they occur in nanoparticles, and an obvious relationship between metal nanoparticles in fault gouges and soils, with the metallic nanoparticles in fault gouges representing a transitional phase along the whole vertical migration process. Pyrite is the most common authigenic mineral preserved in many ancient sedimentary rocks. Liu and coauthors [20] demonstrate pyrite morphology to be an indicator of paleoredox conditions and shale gas content in the Longmaxi and Wufeng shales in the Middle Yangtze area, South China. Based on the formation mechanism, pyrites in the Longmaxi and Wufeng shales can be divided into syngenetic pyrites, early diagenetic pyrites, and late diagenetic pyrites. The size of the pyrite framboid microcrystals is used to distinguish syngenetic pyrite and diagenetic pyrite, owing to their different formation mechanisms. The sedimentary environment of the Longmaxi and Wufeng shales is mainly a euxinic environment with good reduction conditions, but some parts of the Longmaxi Formation reflect an oxic–dysoxic environment. The process of thermochemical sulfate reduction (TSR), accompanied by the formation of diagenetic pyrite, results in oxidation of organic matter (OM) and depletion of the H bond of OM, which influences the amounts of alkane gas produced by organic matter during thermal evolution. Consequently, the size of pyrite framboids and microcrystals could be widely used to rapidly evaluate paleoredox conditions and the gas content in shales.

Verberne and coauthors [21] deal with structural geology, including the mechanism of nanomineral movement in ductile shear zone theory. Principal slip zones (PSZs) are narrow (<10-cm) bands of localized shear deformation that occur in the cores of upper-crustal fault zones, where they accommodate the bulk of fault displacement. Natural and experimentally formed PSZs consistently show the presence of nanocrystallites in the <100-nm size range. The physical properties of nanocrystalline materials may profoundly affect fault rheology and the structural characteristics of nanocrystalline (NC) PSZs observed in natural faults and in experiments. Numerous reports in the literature show that such zones form in a wide range of faulted rock types, under a wide range of conditions pertaining to seismic and aseismic upper-crustal fault slip, as well as that they frequently show an internal crystallographic-preferred orientation (CPO) and partial amorphization and form glossy or "mirrorlike" slip surfaces. It is suggested that the widespread occurrence of NC PSZs in upper-crustal faults is of general significance. Specifically, the generally high rates of (diffusion) creep in nanocrystalline fault rock may play a key role in controlling the depth limits of the seismogenic zone.

Several papers [22–27] in this special issue focus on energy geology, addressing the structure and composition of coal and shale nanominerals as well as changes in nanopores and their fractal dimensions. They also describe structural characteristics such as the size, shape, and connectivity of nanopores in unconventional oil and gas reservoirs, along with their evolutionary laws and control factors. Chen and coauthors [22] focus on an experimental methodological issue involving total porosity measured for shale gas reservoir samples from the Lower Silurian Longmaxi formation in southeast Chongqing, China. Measuring the total porosity in shale gas reservoir samples remains a challenge because of their fine-grained texture, low porosity, ultra-low permeability, and high content of organic matter (OM) and clay mineral. The composition content porosimetry method, which is a new method for evaluating the porosity of shale samples, was used in the study to measure the total porosity of shale gas reservoir samples, based on bulk and grain density values. The findings indicated that the composition content porosimetry porosity generally increases with increasing OM and clay content and decreases with increasing quartz and feldspar content. Huang and coauthors [23] evaluated the pore structure characteristics of Lower Silurian Longmaxi shale gas reservoirs in Well LD1 of the Laifeng–Xianfeng Block, Upper Yangtze region in South China. N_2 adsorption and helium ion microscope (HIM) were used to investigate various pore features, including pore volume, pore surface area, and pore size distribution. The calculated results show a good hydrocarbon storage capacity and development potential for the shale samples. Meanwhile, the reservoir space and migration pathways may be affected by the small pore size. The authors found that large pores usually have large values of

fractal dimensions and identified a strong positive correlation between the fractal dimensions and pore volume, as well as pore surface area. Fractal dimensions can be taken as a visual indicator of the degree of development of a pore structure in shale. Zhu and coauthors [24] studied tectonic and thermal controls on the nano–micro structural characteristics of a Cambrian organic-rich shale. Naturally deformed samples from the main source rocks are selected from the Lower Cambrian Lujiaping Formation in the Dabashan Thrust–Fold Belt to investigate nanometer to micrometer-sized structures. Their findings indicated that fracture-related pores predominate over mineral-hosted pores, as well as that these two pore types account for 90% of total pore space. Overall, Lujiaping deformed over-mature samples have abundant nano to micro-sized inorganic pores. High-resolution SEM images provide direct evidence of the formation of nano- and microsized structures such as OM–clay aggregates and silica nanograins. The characteristics of nanopore structure in shale play a crucial role in methane adsorption and determination of the occurrence and migration of shale gas. Using an integrated approach of X-ray diffraction (XRD), N_2 adsorption, and field emission scanning electron microscopy (FE-SEM), Yu and coauthors [25] systematically focused on eight drilling samples of marine Taiyuan shale from well ZK1 in southern North China to study the characteristics and heterogeneity of their nanopore structure. Their findings indicated that different sedimentary environments may control the precipitation of clay and quartz between transitional shale and marine shale, leading to different organic matter (OM)–clay relationships and different correlations between total organic carbon (TOC) and mineral content. Taiyuan shale of higher heterogeneity is highly fractal, and its fractal dimensions are principally related to micropores. Li and coauthors [26] present a thorough investigation of the impacts of matrix compositions on the nanopore structure and fractal characteristics of lacustrine shales from the Changling Fault Depression (CFD), Southern Songliao Basin. The Lower Cretaceous Shahezi shales are targets for lacustrine shale gas exploration. The Shahezi shales were investigated to explore the impacts of rock composition, including organic matter and minerals, on pore structure and fractal characteristics. Seven lithofacies can be identified on a mineralogy-based shale classification scheme. Inorganic mineral–hosted pores are the most abundant pore type, whereas relatively few organic matter (OM) pores are observed in FE-SEM images of the Shahezi shales. The primary controlling factors for pore structure in Shahezi shales are clay minerals rather than OM. Clay content is the most significant factor controlling the fractal dimensions of the lacustrine Shahezi shale. Pore connectivity of lacustrine shales has been inadequately documented in previous papers. Li and coauthors [27] examine findings relating to lacustrine shales from the lower Cretaceous Shahezi Formation in the Changling Fault Depression (CFD) based on investigations using field emission scanning electron microscopy (FE-SEM), mercury intrusion capillary pressure (MICP), low-pressure gas (CO_2 and N_2) sorption (LPGA), and spontaneous fluid imbibition (SFI) experiments. Their findings indicate that pores observed from FE-SEM images are primarily interparticle (interP) pores in clay minerals and organic matter (OM) pores. Pore connectivity proceeds in the calcareous shale > argillaceous shale > siliceous shale. The connected pores of Shahezi shales are affected mainly by the abundance and coexistence of OM pores and clay carbonate mineral host pores.

The other articles [28–32] in this special issue are devoted to ore deposits and mineral materials. The genesis of Carlin-type gold deposits is analyzed at the nano scale; the significance of using metallic nanoparticles to search for concealed deposits is discussed; and the preparation, characterization, and physicochemical properties of nanomineral materials are explored. Tan and coauthors [28] present two hydrothermal events at the Shuiyindong Carlin-type gold deposit in southwestern China from Sm–Nd dating of fluorite and calcite. The Shuiyindong Gold Mine hosts one of the largest and highest-grade strata-bound Carlin-type gold deposits discovered to date in Southwestern China. The ore is hosted mainly in Upper Permian bioclastic limestone near the axis of an anticline. The gold is hosted mainly in arsenian pyrite and arsenopyrite, primarily as crystal lattice gold, submicroscopic particles, and nanoparticles. Fluorite commonly occurs at the vicinity of an unconformity between the Middle–Upper Permian formations, proposed as the structural conduit that fed the ore fluids. Calcite commonly fills fractures at the periphery of decarbonated rocks, which contain high-grade orebodies. Their study was

intended to verify the occurrence of two distinct hydrothermal events at the Shuiyindong, based on Sm–Nd isotope dating of the fluorite and calcite, which contains considerable concentrations of REE, and exhibit variable Sm/Nd ratios, facilitating the direct dating of associated hydrothermal events. Two groups of Sm–Nd isochron ages suggest two episodes of hydrothermal events in Shuiyindong. The age of the calcite likely represents the late stage of the gold mineralization period. Initial Nd isotopic compositions indicate that the Nd in the fluorite and calcite was likely derived from mixtures of basaltic volcanic tuff and bioclastic limestone of the Permian formations. Lu and coauthors [29] contribute to a better understanding of the green preparation and performance and mechanism of nanoporous pyrrhotite by thermal treatment of pyrite as an effective Hg(II) adsorbent. The removal of Hg(II) from aqueous solutions using pyrrhotite derived from the thermal activation of natural pyrite was explored by batch experiments. The adsorption isotherms demonstrated that the sorption of Hg(II) by modified pyrite (MPy) can be fitted well by the Langmuir model. The removal of Hg(II) by MPy was attributed mainly to a chemical reaction that resulted in cinnabar formation as well as electrostatic attraction between the negative charges in MPy and positive charges of Hg(II). The results of this work suggest that the thermal activation of natural pyrite is highly important for effective use of ore resources to remove Hg(II). Ca-bentonite was used as the feedstock material for the synthesis of hydroxysodalite because of its high Al, Si content, good chemical reactivity, and natural abundance. Liu and coauthors [30] report the results of one-step synthesis of hydroxysodalite using natural bentonite at moderate temperatures. The Na/Si molar ratio and reaction temperature both played important roles in controlling the degree of crystallinity of the synthetic hydroxysodalite. Although clays are widely used as sorbents for heavy metals due to their high specific surface areas, low cost, and ubiquity in most soil and sediment environments, they still face inherent limitations owing to the low loading capacity of heavy metals. Yao and coauthors [31] studied enhanced potential toxic metal removal using a novel hierarchical SiO_2–$Mg(OH)_2$ nanocomposite derived from sepiolite. The structural characteristics of the resulting modified samples were characterized by a wide range of techniques, including field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM), energy dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD), and nitrogen physisorption analysis. The results show that a hierarchical nanocomposite constructed by loading $Mg(OH)_2$ nanosheets onto amorphous SiO_2 nanotubes can be successfully prepared, with the nanocomposite having a high surface area (377.3 m^2/g) and pore volume (0.96 cm^3/g). Batch removal experiments indicate that the nanocomposite exhibits high removal efficiency toward Gd(III), Pb(II), and Cd(II). Zhou and coauthors [32] address the characterization and amoxicillin adsorption activity of mesopore $CaCO_3$ microparticles prepared using rape flower pollen. $CaCO_3$ adsorbent was characterized using X-ray diffraction (XRD) and scanning electronic microscopy (SEM). The equilibrium adsorption data on amoxicillin were explained using the Langmuir, Freundlich, and Temkin adsorption isotherm models, whereas equilibrium adsorption of as-prepared $CaCO_3$ was better depicted using the Langmuir adsorption model, indicating the favorable adsorption of amoxicillin.

The understanding and cognition of the Earth directly affect human beings' ability to survive and develop. The exploration and development of energy and mineral deposits, disaster and environmental prediction and management, and the like are all urgent problems in Earth science and relate to its resources, disasters, and environment. Using the nanoscale to re-understand and analyze these problems will open new perspectives and directions for researchers, whose object in conducting nanomineralogy is related chiefly to minerals and their pores in different spheres of the Earth system.

As a geoscientific frontier, nanomineralogy faces novel opportunities and challenges. Inevitably, some results of prior research, and the knowledge based on them, will be overturned. Nanomineralogy is still in its infancy, however, and although certain achievements have already been made, understanding is still lacking concerning the formation mechanism of mineral elements; the migration process, structural form, and disaster-related and environmental effects of nanoparticles; and the mineralization of nanopores. Taking full advantage of the advantages of multidisciplinary cross-development; promoting international cooperation among scientists; and systematically researching nanomineralogy,

including by seeking concentrated solutions to key scientific problems, will enrich and spur the development of nanomineralogic theory and methods. Such an approach will provide an important theoretical basis for use of new mineral- and carbon-based materials, the exploration and exploitation of energy and mineral resources, environmental protection, and disaster prediction.

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