



ARCHIVES

of

FOUNDRY ENGINEERING

ISSN (2299-2944)

Volume 2025

Issue 4/2025

10 – 15



10.24425/afe.2025.155374

2/4

Published quarterly as the organ of the Foundry Commission of the Polish Academy of Sciences

The Effect of Addition of the Natural Zeolite on the Microstructure and Properties of Sintered Iron Matrix Composite

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Received 12.02.25; accepted in revised form 06.05.25; available online 27.10.2025

Abstract

The paper presents the results of research on the production and application of a sintered iron-based composite reinforced with natural zeolite particles. Mechanical properties were tested and the quality of the connection between the particles and the metal matrix was assessed. Before the composite production process, the chemical composition and morphology of natural zeolite particles tests were carried out. Zeolite particles with a diameter of less than 0.2 mm were used to produce sintered. The zeolite particles were subjected to chemical composition (EDS) and phase (XRD) tests. Zeolite particles were introduced into the iron matrix in amount of 5, 10, 15% by weight. Before the sintering process, the zeolite particles were pressed in a hydraulic press at a compaction pressure of 400 MPa. The sintering of the green compacts was carried out in a tubular furnace at 950°C in an atmosphere of dissociated ammonia for 60 minutes. The obtained composites were subjected to porosity, hardness and density measurements. Microstructure and chemical composition studies were carried out using a scanning microscope (SEM). Iron-zeolite composites are characterized by higher hardness and porosity compared to sintered iron. The introduction of zeolite particles also reduces the density of the composites.

Keywords: Metal matrix, Iron, zeolite, Powder metallurgy

1. Introduction

Metal matrix composites reinforced with ceramic particles constitute an important group among engineering materials. They are used in many branches of industry, including aviation, automotive and as materials resistant to abrasive wear [1-3]. Metal matrix composites are most often manufactured using powder metallurgy technology. The use of powder metallurgy ensures obtaining good dimensional accuracy without the need for machining, as well as control over the chemical composition of the composites [4,5]. The most commonly used reinforcing particles are metal oxides, carbides and borides. Usually, powders up to 100 µm in size are used, and their content does not exceed

25% by weight [6-8]. Based on a review of the literature, it can be stated that light metals are most often used as the matrix of metal composites [9,10]. However, composites based on heavy metals such as copper or iron are also an important group. Copper matrix composites are used in electronics and electrical engineering due to their high electrical conductivity [11-13]. Iron is used to produce composites based on low-alloy and tool steels, where oxides, carbides and nitrides are used as reinforcing particles [14]. Research is underway to produce composites reinforced with other particles. These include volcanic tuff, fly ash or natural zeolite. Continuously conducted research on the specific properties of zeolites, which include catalytic, ion exchange and adsorption properties, indicate the versatile possibilities of using these materials, e.g. in the processing of crude oil, in construction,



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water and exhaust gas treatment. For this reason, zeolites are considered a modern raw material for the production of engineering materials [15]. Despite interesting experiments by many Polish and foreign authors on the subject of zeolites, there is little detailed research on the possibilities of using zeolite particles as a reinforcing phase of metal matrix composite materials. So far, few works have been published on the testing of sintered composites reinforced with zeolite particles in terms of compressive strength, abrasive wear resistance, hardness or thermal conductivity [16,17]. The most common zeolite minerals in the world are clinoptilolite. They are aluminosilicates with a complex chemical composition containing, among others, calcium, potassium, sodium, silicon, as well as strontium, manganese, barium and magnesium. This group was distinguished in the 18th century by the Swedish mineralogist Axel Frederik Cronstedt [18-20]. Zeolites are natural minerals that were formed as a result of volcanic ash deposition in ancient alkaline lakes. As a result of the reaction between volcanic ash and salts present in water, the volcanic ash was transformed into various groups of aluminosilicates with unique physico-chemical properties. The unique properties of zeolites result from their porous crystal structure. This structure is characterized by the presence of channels and chambers with precisely defined molecular dimensions. The basic units building the zeolite skeleton are tetrahedral $(\text{SiO}_4)^4-$ and $(\text{AlO}_4)^5-$, which are connected to each other by common oxygen atoms, with some of the silicate atoms replaced by aluminum atoms $(\text{AlO}_4)^5-$. In this way a characteristic three-dimensional structure was created with a large number of chambers connected by channels. These chambers are called pores in which metal cations and water molecules are located. The total volume of these voids is 24 – 32%. Thanks to these unique properties such as crystalline structure, thermal and hydrothermal stability, negative charge of the crystal lattice, easy exchange of off-lattice cations, uniform micropore size, are important factors encouraging the use of zeolites in many different fields such as: ion exchange, catalysis, crude oil processing, construction, water and exhaust gas treatment and others [21,22]. Taking into account the properties of zeolites that allow their use in many industries, in the presented work research was carried out on the production and application of iron-based metal composites reinforced with zeolite particles. The zeolite particles used for the study were obtained from zeolite rock mined in Slovakia. The deposits there are among those with the highest raw material purity.

The aim of the research conducted in this work was to evaluate the connection of zeolite particles with an iron matrix by means of microstructure observation, as well as to investigate the hardness and density of the composites. Based on the conducted research, the application of the produced materials was assessed.

2. Materials and methods

Sprayed iron powder ASC 100.29 produced by the Swedish company Höganäs, with a particle size of 20-180 μm and an Fe content of 99.9%, and ground natural zeolite from a deposit located in the eastern part of Slovakia in Kucin were used for the tests. A fraction smaller than 0.2 mm was isolated for testing. The shape and arrangement of powder particles used for testing are shown in Figure 1.

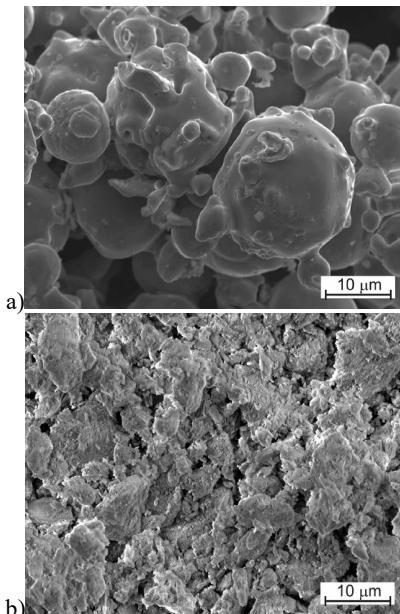


Fig. 1. Photos of powders used for testing: a) Fe powder, b) zeolite powder

Studies of the morphology of the zeolite rock as delivered showed the formation of agglomerates from powder particles. For this reason, mixing the powders before the sintering process should be carried out for a long time in order to break down the agglomerates and to achieve an even distribution of the strengthening particles in the metal matrix.

Before the process of forming the green compacts, the zeolite powder was observed on a JEOL JSM-7100F scanning electron microscope. The elements present in the zeolite particles were determined by X-ray microanalysis using an EDS analyzer.

Figures 2, 3 shows the X-ray spectrum of the surface of zeolite particles, while Table 1 shows the content of individual elements present in zeolite powder.

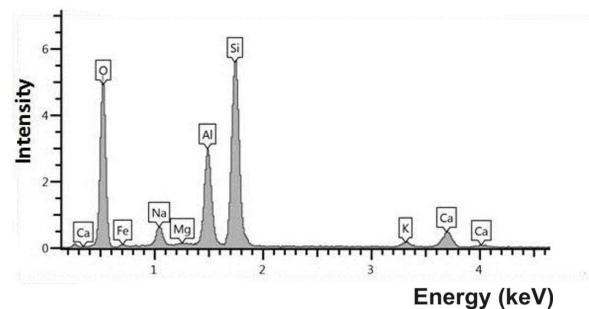


Fig. 2. X-ray spectrum acquire from the surface of zeolite particles

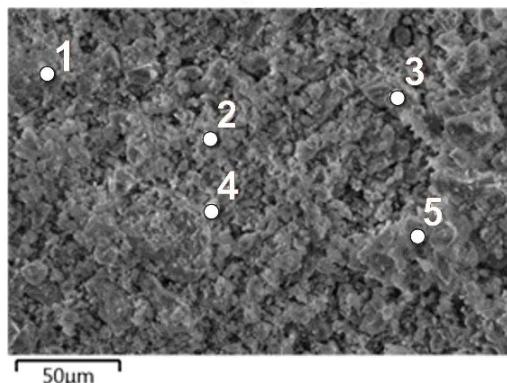


Fig. 3. Examples of point where EDS analyses were taken

Based on the conducted studies of the chemical composition of zeolite particles, it can be stated that the main elements present in it are: aluminum, calcium, silicon, potassium and iron. The studies confirm that the tested material is an aluminosilicate mineral.

Table 1.

The content of the main elements found in zeolite

Al [%]	Si [%]	K [%]	Ca [%]	Fe [%]	O [%]
5-14	23-32	2-14	1-2.5	<1.5	40-50

Before the process of manufacturing the green compacts, diffraction tests were carried out on the zeolite rock delivered for testing. Phase composition analysis was performed using the powder method (DSH) using a Cu lamp in the convergent Bragg-Brentano beam geometry using a Bruker D8 Discover diffractometer. Measurements were performed using a one-dimensional, semiconductor Lynx-Eye detector. The mineral composition was determined and calculated based on the licensed standards of the International Center for Diffraction Data and on the basis of the ICSD (Inorganic Crystal Structure Database) and NIST (National Institute of Standards and Technology) databases. DIFFRAC and Topas software were used for registration and diagnostics. The diffractometric tests (XRD) carried out on the zeolite rock sample as delivered showed the following phase composition, Table 2. The designated crystalline phases in the tested sample are marked in the diffractogram below, Figure 4.

Table 2.

Analysis of the phase composition of zeolite as delivered

Phases	Average amount [%]
Clinoptilolite	35
Feldspars	29
Quartz	11
Illite + muscovite	2
Kaolinite	2
Amorphous substance	21

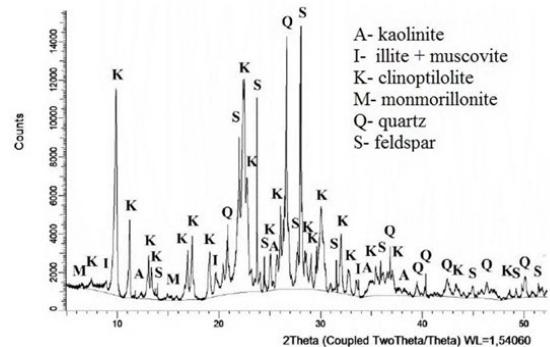


Fig. 4. X-ray diffractogram of zeolite rock as delivered

After carrying out the above-mentioned tests, powder mixtures containing 5, 10, 15% zeolite were prepared. The powders were then mixed manually until a homogeneous mixture was obtained. The finished powder mixtures before the sintering process were subjected to one-side pressing using a hydraulic press. The compaction pressure was 400 MPa. Sintering of the compacts was carried out in a laboratory tube furnace. The following parameters were set: temperature - 950 °C, sintering time - 60 minutes, protective atmosphere - dissociated ammonia. After sintering was completed, the samples were cooled with the furnace.

As a result of sintering, cylindrical samples with dimensions of $\phi 20 \times 10$ mm were obtained, which were subjected to hardness and density measurements. Density measurements were performed on a hydrostatic balance type WPA120 using the air and water weighing method in accordance with the PN EN ISO 2738:2001 standard. Hardness measurements were performed using the Brinell method in accordance with the PN EN ISO 6506-1:2014 standard. A steel ball with a diameter of 5 mm and a load of 250 kg were used for the measurements. The microstructure observations of the produced composites were performed using a scanning electron microscope (SEM) and a light microscope (OM). The porosity degree of the composites was determined by the Cavalieri-Hacqert method on a Nikon MA200 light microscope using NIS software.

3. Results and discussion

3.1. Microstructural and porosity investigations

The introduction of zeolite particles of various sizes and shapes into the iron matrix caused a change in the microstructure of the composite. The porosity of the composite increases with the increase in the content of zeolite particles. Analyzing the obtained microstructures, empty spaces between the zeolite particles and the metal matrix can be noticed. The microstructures of iron-zeolite composites with different contents of zeolite particles are shown in Figure 5.

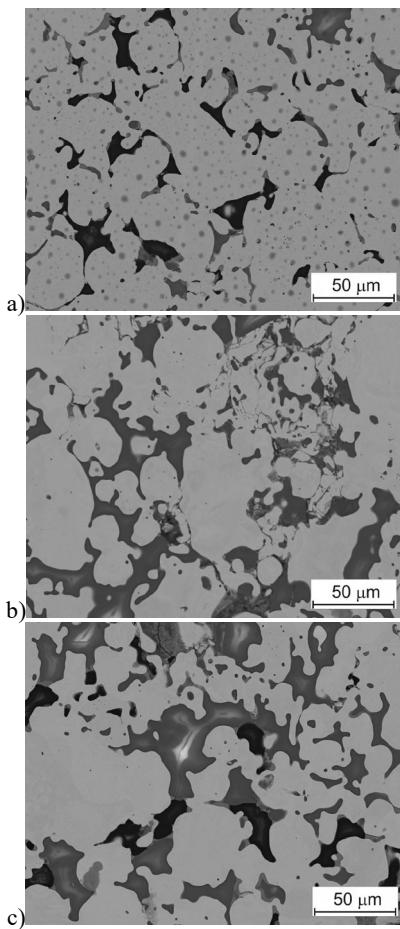


Fig. 5. Microstructure of sintered iron with zeolite, SEM.
a) Fe+5% zeolite, b) Fe+10% zeolite, c) Fe+15% zeolite

The measurement of the porosity of the composites consisted in determining the ratio of the pore area to the entire analyzed surface. Due to the difficult preparation of the surface for testing, the measurements were performed multiple times for each samples. The measurements were carried out in three selected areas of each sample in which the most uniform distribution of zeolite particles in the metal matrix was observed. ROI frames were set to binarize and analyze porous areas, ignoring possible artifacts. The results were averaged. Figure 6 shows the microstructures of sintered made using a light microscope. The photos also include frames used for porosity measurements. Table 3 shows the average results of porosity measurements.

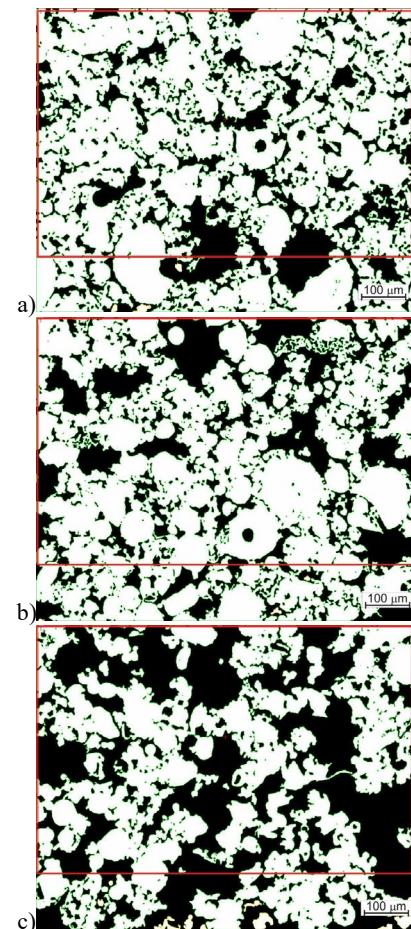


Fig. 6. Microstructure of sintered iron with zeolite, light microscope, porosity measurement: a) Fe+5% zeolite, b) Fe+10% zeolite, c) Fe+15% zeolite

Table 3.
Results of porosity measurements

Material	Porosity, average [%]
Fe	7,9 ± 1,2
Fe + 5% zeolite	21,3 ± 1,6
Fe + 10% zeolite	31,2 ± 2,1
Fe + 15% zeolite	46,6 ± 1,5

The introduction of zeolite particles into the iron matrix resulted in an increase in porosity. This is due to the structure of zeolite, which contains a large number of empty channels. The high porosity may also be related to the relatively low pressing pressure. Another reason for the increasing porosity may be that zeolites are classified as hydrated aluminosilicates. During sintering, water could have been released from the zeolite structure, which, turning into steam, could have created empty spaces. This phenomenon has also been noticed by other researchers [23,25].

3.2. Hardness and density measurements

The introduction of zeolite particles increased the hardness of the composites. This is due to the relatively high hardness of zeolite (3-5.5 on the Mohs scale). Due to the introduction of low-density zeolite particles (about 2.3 g/cm³) and the occurrence of porosity, a significant decrease in the density of the composites was observed. The phenomenon of increasing hardness and decreasing density after introducing ceramic particles into the metal matrix is often observed by other researchers [17,18,24]. The results of density and hardness measurements are shown in table 4.

Table 4.
Results of hardness and density measurements

Material	Density, (g/cm ³)	Theoretical density, (g/cm ³)	HB
Fe	6,24 ± 0,04	7,8	46,22 ± 1,5
Fe + 5% zeolite	5,80 ± 0,02	7,52	51,90 ± 1,8
Fe + 10% zeolite	5,23 ± 0,03	7,24	57,74 ± 1,1
Fe + 15% zeolite	4,96 ± 0,03	6,96	62,45 ± 1,2

3.3. Results of X-ray diffraction tests

After the consolidation process, the zeolite was crushed from the sinters and diffractometric tests were carried out again to check its phase composition after heating at 950 °C. Analysis of the phase composition of the zeolite after annealing showed the presence of quartz in the amount of 24-25%, potassium feldspar in the amount of 21-23%, cristobalite in the amount of 5-6%, mullite in the amount of 2-4%, wustite in the amount of <2% and the presence of amorphous in the amount of 43-44%.

The designated crystalline phases in the tested sample are marked in the diffractogram below, Figure 7.

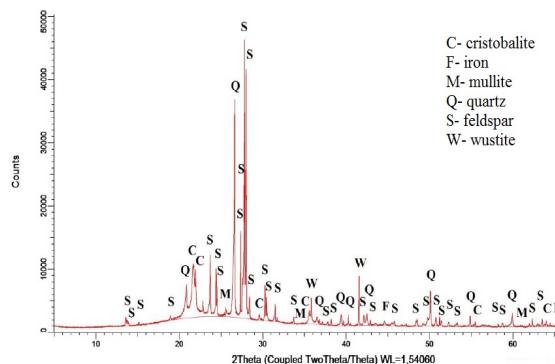


Fig. 7. X-ray diffractogram of zeolite rock after heating at 950 °C

4. Summary and Conclusions

Using commercially available powders, metal matrix composites reinforced with zeolite particles were produced using conventional powder metallurgy technology. The composite manufacturing parameters were selected based on our own research and information available in the literature. The results showed that the introduction of zeolite particles into the iron matrix causes changes in hardness, density and porosity, as well as changes in the microstructure of the composites. Phase composition studies of zeolite powder in the delivery state have shown its complex chemical composition. Diffraction studies have shown that the main compounds present in zeolite are: clinoptilolite, potassium feldspars, quartz, illite and kaolinite. A significant amount of amorphous substance is also present in zeolite powder. The relatively small amount of clinoptilolite in the zeolite as delivered may be due to the grinding process. As a result of grinding the zeolite rock, the clinoptilolite contained in it may have been damaged and thus identified as an amorphous substance during diffractometric studies.

As the zeolite content increased, a decrease in density was observed. This is caused by the increase in porosity of the sinters. The hardness tests showed that the hardness of the sinters increases with the increase in zeolite content. The introduction of 10% by weight of zeolite increases the hardness of the composites by almost 25%. This is a satisfactory result.

Studies of the porosity of iron-zeolite composites have shown that with the increase in the content of zeolite particles, the porosity reaches higher values. The porosity of iron-zeolite composites ranges from about 20-47 % and is related to the internal structure of the zeolite, as well as the parameters of the manufacturing process. For this reason, these composites can be used, among others, as absorption materials. The microstructures of the composites produced have pores and discontinuities characteristic of sintered materials. The introduction of zeolite into the iron matrix intensified this effect due to the internal structure of the zeolite. Observations have shown that the connection of zeolite particles with the iron matrix is satisfactory. No diffusion connection of zeolite particles with the metal matrix was observed.

Based on the research conducted, the following conclusions were formulated:

The use of zeolite particles as a reinforcing phase is justified due to the increase in the hardness of the composites.

The introduction of zeolite particles causes a decrease in the density of the composites and an increase in porosity, which is why these composites can be used as absorbent materials.

The distribution of the introduced particles is nearly homogeneous and the quality of their connection with the matrix is satisfactory. Based on the microstructural tests performed, no discontinuities were found at the matrix-zeolite interface. In all three cases, a very good connection of zeolite particles with the iron is visible. Based on the conducted research, it can be stated that iron-zeolite composites can find application, among others, in absorbent materials. Due to the high porosity of the composites and the ability of zeolite to absorb substances, they can be saturated by infiltration, which would allow for their use, for example, in self-lubricating materials.

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