Доклади на Българската академия на науките
Comptes rendus de l'Académie bulgare des Sciences
Tome 79, No 11, 2026

ENGINEERING SCIENCES
Materials science

INVESTIGATION OF IRON-CONTAINING WASTE GENERATED DURING MAGNESIUM ALLOY CASTING

Vladimir Petkov✉, Mihaela Aleksandrova, Stancho Yordanov, Ina Gospodinova

Received on August 25, 2026
Presented by S. Hadjitodorov, Corresponding Member of BAS, on October 27, 2026

Abstract

Technological iron-containing waste (scale) generated during the casting of magnesium alloys in steel moulds was investigated. The aim of the present study was to determine the phase and elemental composition of the waste material. For this purpose, X-ray diffraction (XRD) and X-ray fluorescence (XRF) analyses were performed. Three major iron oxide phases were identified: wüstite (FeO), hematite (α-Fe_2O_3), and magnetite (Fe_2O_4). In addition, the microstructure and elemental composition of solid waste samples collected from different parts of the scale formation on the wall of the steel mould were investigated by scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDS). The powder sample of the scale provided the most reliable characterization of the phase and elemental composition of the iron-containing waste material.

Key words: iron-containing waste, mill scale, S275JR steel, AZ63 magnesium alloy, X-ray diffraction, X-ray fluorescence

Introduction. The utilization of waste materials in modern industry is becoming an increasingly important and effective approach to improving resource efficiency and sustainability. In recent years, the term *"waste material"* has increasingly been replaced by *"by-product"* [1]. The composition and properties of industrial waste materials are closely related to the development of new technologies for their recovery and utilization. Therefore, the investigation and utilization of waste materials generated during the casting of magnesium alloys in steel

https://doi.org/10.7546/CRABS.2026.11.14

moulds are of considerable interest because of their potential environmental and economic benefits.

Magnesium alloys are well known for their low density (ρMg = 1.83 g/cm^3) and good mechanical properties, making them suitable for a wide range of industrial applications, including the automotive, aerospace, and medical sectors [2]. However, the processing and casting of these alloys can generate significant amounts of waste that require appropriate characterization and effective management.

The development of sustainable technologies for the utilization of iron-containing waste powders generated in mechanical engineering and metallurgical processes requires, as a first step, a detailed investigation of their chemical and phase composition, followed by the selection of an appropriate recycling or recovery technology. In general, iron-containing waste powders may consist of several iron oxide phases, including wüstite (FeO), hematite (α-Fe_2O_3), and magnetite (Fe_3O_4). The chemical and phase composition of mill scale may vary depending on the type of steel and the processing conditions. The iron content in mill scale is usually approximately 70%, while it may also contain traces of non-ferrous metals, alkaline compounds, and contaminants originating from lubricants, oils, and greases associated with the equipment used [1].

The reduction of iron-containing waste powders is commonly carried out in a hydrogen-containing atmosphere, such as pure hydrogen, an argon–hydrogen mixture containing 35% H_2, or dissociated ammonia, at temperatures in the range of 1050–1150 °C [3, 4].

Gravity casting is a fundamental metallurgical process that is widely used in various industries, including the automotive, aerospace, and construction sectors. Owing to its simplicity and efficiency, it remains a preferred method for the production of metal components. The present study focuses on determining the elemental and phase composition of iron-containing waste material (scale) accumulated on the walls of steel moulds used for the casting of magnesium alloy.

Materials and methods. The chemical element magnesium (Mg) is 75% lighter than steel and 33% lighter than aluminium. Therefore, magnesium alloys are widely used in various industries due to their low density and good mechanical properties [5]. The AZ63 alloy is one of the commonly used magnesium alloys and is characterized by good corrosion resistance and favourable mechanical properties.

The chemical and phase composition of the waste iron material (scale) was characterized using X-ray diffraction (XRD), X-ray fluorescence (XRF), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS).

The samples were examined using a PANalytical Aeris X-ray diffractometer equipped with Cu-Kα radiation (λ = 1.5406 Å) and operated in θ–θ Bragg–Brentano geometry. The elemental composition was determined using an Epsilon 1 spectrometer (PANalytical) equipped with an Ag X-ray tube operated at a voltage of 50 kV and a maximum power of 10 W. SEM–EDS analysis was performed using

Table 1

Chemical composition of the AZ63 magnesium alloy [6] and S275JR steel used for the moulds in which the magnesium is melted

Chemical composition of the input material, wt.%							
Element in AZ63	Al	Zn	Mn	Si	Ni	Cu	Mg
Concentration	5.5–6.5	2.5–3.5	0.25–0.45	0.06	0.06	0.005	rest
Element in S275JR	C	Mn	Cr	Ni	Si	Cu	Fe
Concentration	0.08	0.43	0.003	0.02	0.83	0.03	rest

a HIROX SH-5500P scanning electron microscope equipped with an integrated QUANTAX 100 Advanced energy-dispersive X-ray spectroscopy (EDS) system (Bruker).

Figure 1 shows the initial waste material (scale) on the left and a SEM image of an analysed fragment on the right. The analysed area is outlined by a yellow rectangle.

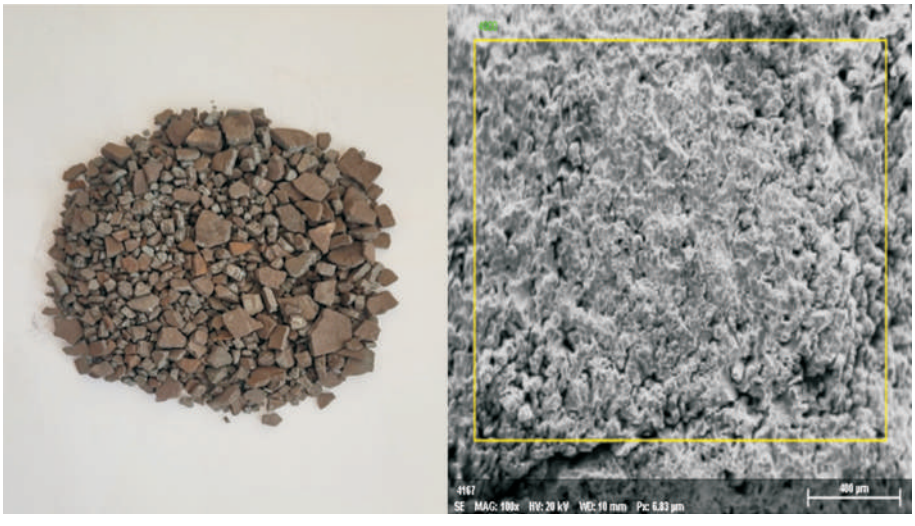
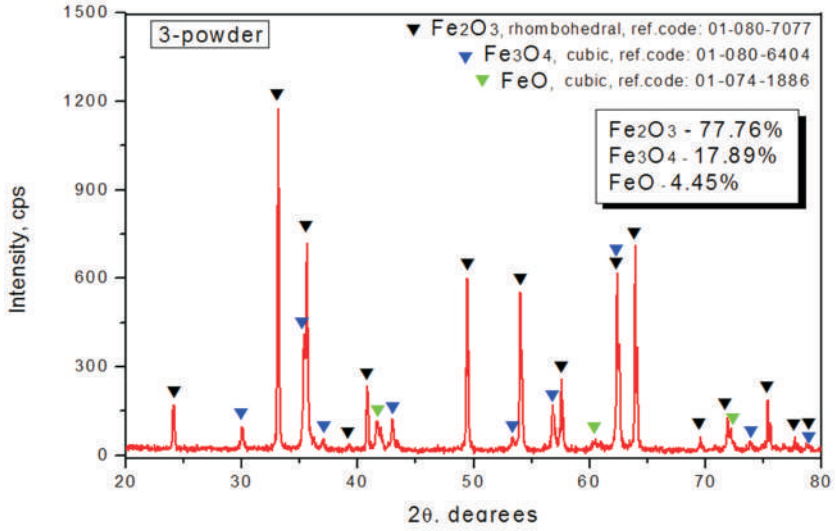


Fig. 1. Initial waste material (scale) used in the study and SEM image of a representative fragment

Results. To obtain more reliable and accurate results, X-ray diffraction (XRD) and X-ray fluorescence (XRF) analyses were performed on a powdered sample, and the results are presented in Fig. 2. Three main iron oxide phases were identified by XRD and quantitatively determined, as shown in Fig. 2A. The content of hematite (Fe_2O_3) was 77.76 wt.%, magnetite (Fe_3O_4) accounted for 17.89 wt.%, and wüstite (FeO) for 4.45 wt.%.

The XRF results presented in Fig. 2B enabled the determination of the elemental composition of the powdered sample. Iron was found to be the predomi-



A. Diffractogram of the ground initial sample (Sample 3) of the waste iron material (scale).

B. Elemental composition of waste scale of sample 3 — Normalization factor 1.650

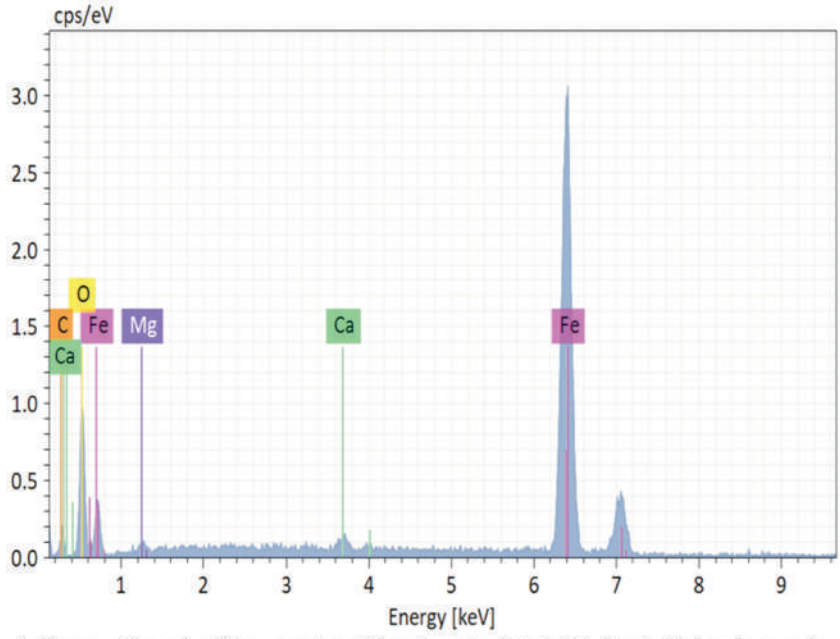
Compound	Si	P	Ca	Mn	Fe	Eu	Pb
Cons.	0.488	0.137	0.137	1.169	97.465	0.57	0.031
Unit	%	%	%	%	%	%	%

Fig. 2. A) X-ray diffractogram of the scale with the corresponding phases and crystal structures identified; B) elemental composition of the scale determined by XRF

nant element, with a content of 97.5 wt.%, followed by manganese (Mn) at 1.17 wt.%, europium (Eu) at 0.57 wt.%, silicon (Si) at 0.49 wt.%, and other elements present in smaller amounts. Manganese was introduced as an alloying element in the magnesium alloy. Of particular interest is the presence of europium, a rare-earth element of high economic value, which was introdeced into the material along with the initial raw material and was incorporated into the scale during the casting process.

The microstructure observed in Fig. 1 is characterized by a coarse-grained morphology, a relatively rough surface, and distinct porous layers located between individual agglomerates. The investigated area is outlined by a yellow rectangle at a magnification of 100×. Scanning electron microscopy (SEM) was performed on an intact solid fragment of the scale. The recorded EDS spectra indicated that iron and oxygen were the predominant elements, with a combined content of 91.64 normalized wt.%, while calcium and magnesium were detected at approximately 1 wt.% each (Fig. 3B).

Discussion. SEM–EDS analysis of the solid scale sample did not detect Eu and Mn, although, according to the available analytical data, these elements are



A. Energy-dispersive X-ray spectra of the elements detected in the studied scale sample.

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Fe	26	14558	63.24	77.27	48.27	1.80	2.85
O	8	1939	11.76	14.37	31.33	2.27	19.30
C	6	276	5.08	6.20	18.01	1.76	34.58
Ca	20	325	1.02	1.24	1.08	0.09	9.27
Mg	12	115	0.75	0.91	1.31	0.12	16.33
		Sum	**81.84**	**100.00**	**100.00**		

B. Quantitative determination of the elemental composition of the scale sample from the area outlined by the yellow rectangle in Fig. 1.

Fig. 3. SEM–EDS analysis of a fragment of the initial waste iron material

present in the composition of the waste material. The presence of carbon could not be reliably confirmed due to the relatively high analytical uncertainty associated with its determination. It is well known that light elements are characterized by greater uncertainty in SEM–EDS analysis. Consequently, the relative iron content is overestimated, reaching values close to 90%, which is in good agreement with the results obtained by X-ray fluorescence analysis of a powdered sample of the same material. It should be noted that differences in sample form and preparation (solid sample for SEM–EDS and powdered sample for XRF) may affect the accuracy and comparability of the quantitative results. Nevertheless, the combined application of XRD, XRF, and SEM–EDS provides sufficient information for the

characterization and identification of the studied iron-rich waste material.

To obtain high-purity metallic iron, the scale can be reduced using reducing agents such as carbon (coke), dissociated ammonia, hydrogen, or hydrogen–argon mixtures at temperatures of approximately 1050 °C. Carbon monoxide (CO) is another widely used gaseous reducing agent for the production of metallic iron [7]. Benchiheub et al. [8] reported metallization levels exceeding 97% following sequential reduction with CO and H_2 at 1050 °C.

In addition to the production of metallic iron, iron-rich scale can potentially be utilized in several applications, including as an additive in the production of ceramic tiles, as a raw material in the cement industry, as a catalyst or catalyst precursor in battery recycling processes, and as a source of iron for iron-deficient soils, among others [9].

Conclusions. Based on the experimental results obtained, the following main conclusions can be drawn:

- The analysed waste material generated during magnesium casting in steel moulds contains a significant amount of iron oxides, providing a potential basis for its recovery through reduction to metallic iron.
- Based on the XRD and XRF analyses, three iron oxide phases were identified in the powdered sample in the following proportions: hematite (Fe_2O_3) – 77.76%, magnetite (Fe_3O_4) – 17.89%, and wüstite (FeO) – 4.45%.
- A good correlation was observed between the XRF and SEM–EDS results regarding the elemental composition of iron in the scale.
- The detection of europium (Eu) provides a basis for further research aimed at the recovery of this valuable rare-earth element, which was detected in the scale at a concentration of approximately 0.57%.
- The iron-rich scale can be considered a valuable by-product with potential for utilization in the development of waste-minimization technologies and in accordance with the principles of circular economy.

REFERENCES

[1] Martín M. I., F. A. López, J. M. Torralba (2012) Production of sponge iron powder by reduction of rolling mill scale, Ironmak. Steelmak., **39**(3), 155–162, https://doi.org/10.1179/1743281211Y.0000000078.

[2] Giavaresi G., D. Bellavia, A. De Luca et al. (2024) Magnesium alloys in orthopedics: a systematic review on approaches, coatings and strategies to improve biocompatibility, osteogenic properties and osteointegration capabilities, Int. J. Mol. Sci., **25**(1), 282, https://doi.org/10.3390/ijms25010282.

[3] Petkov V., R. Valov (2021) Creation of a technological path for utilization of waste material from iron scale, Int. J. NDT Days, **IV**(5), 295–304.

[4] Gidikova N., V. Manolov, V. Petkov et al. (2017) Mill scale reduction, J. Mater. Sci. Technol., **25**(1), 12–19.

[5] https://waykenrm.com/blogs/lightweight-metals/2026.
[6] https://bg.wfmagnesium.com/magnesium-metal/magnesium-alloy-ingot/magnesium-alloy-ingot-az63a-good-thermal.html, 2026.
[7] Shekhawat D., V. Kukshal, M. K. Banerjee et al. (2021) Study the reduction of mill scale with lean grade coal through RI-RDI, IOP Conf. Ser. Mater. Sci. Eng., **1017**(1), 012037, https://doi.org/10.1088/1757-899X/1017/1/012037.
[8] Benchiheub O., S. Mechachti, S. Serrai et al. (2010) Elaboration of iron powder from mill scale, J. Mater. Environ. Sci., **1**(4), 267–276, https://www.jmaterenvironsci.com/Journal/vol1-4.html.
[9] https://aeiforos.bg/main_bg/13725_okalina.html, 2026.

Institute of Metal Science, Equipment and Technologies,
with Hydro- and Aerodynamics Centre "Acad. A. Balevski",
Bulgarian Academy of Sciences, 67 Shipchenski Prohod Blvd, 1574 Sofia, Bulgaria
e-mails: vladimir2pe@yahoo.com, mihaelaaleksandrova204@gmail.com, stancho14@abv.bg, inaherbal73@gmail.com