

ABSTRACT AND REFERENCES

MATERIALS SCIENCE

DOI: 10.15587/1729-4061.2020.193885**REVERSE LEACHING OF MAGNESIUM FROM FERRONICKEL SLAG USING ALKALI SOLVENT NaOH (p. 6–14)****Agus Budi Prasetyo**

Universitas Indonesia, Jawa Barat, Indonesia

ORCID: <http://orcid.org/0000-0001-7514-4648>**Darmawansyah Rahadian**

Universitas Sultan Ageng Tirtayasa,

Cilegon Banten, Indonesia

ORCID: <http://orcid.org/0000-0002-1749-3580>**Wahyu Mayangsari**

Universitas Indonesia, Jawa Barat, Indonesia

ORCID: <http://orcid.org/0000-0002-7941-5510>**Eni Febriana**

Indonesia Institute of Sciences, Tangerang Selatan, Indonesia

ORCID: <http://orcid.org/0000-0002-1816-9598>**Sulaksana Permana**

Universitas Indonesia, Jawa Barat, Indonesia

ORCID: <http://orcid.org/0000-0003-3473-5892>**Ahmad Maksum**

Politeknik Negeri Jakarta, Depok, Indonesia

ORCID: <http://orcid.org/0000-0003-1800-9137>**Oediyan Soesaptri**

Universitas Sultan Ageng Tirtayasa,

Cilegon Banten, Indonesia

ORCID: <http://orcid.org/0000-0003-1059-4597>**Florentinus Firdiyono**

Indonesia Institute of Sciences, Tangerang Selatan, Indonesia

ORCID: <http://orcid.org/0000-0003-4686-1626>**Johny Wahyuadi Soedarsono**

Universitas Indonesia, Jawa Barat, Indonesia

ORCID: <http://orcid.org/0000-0001-6051-2866>

A research based on magnesium extraction of ferronickel slag waste processed by reverse leaching using sodium hydroxide (NaOH) solutions has been carried out. The ferronickel slag has the main compositions of magnesium silicate and iron silicate. The early procedure of the research was the preparation of ferronickel slag grinding using a ball mill until it gets a size of -200 mesh. Secondly, the calcination of ferronickel slags to remove crystal water and increase the size of the porosity so that it would facilitate the leaching process. The next procedure was reverse leaching using sodium hydroxide (NaOH) to dissolve silica. By dissolving the silica, it was expected that the contents of elements such as magnesium and iron would increase in the residue. The variations in this ferronickel slag leaching research were leaching time, solvent concentration and leaching temperature. The reverse leaching of ferronickel slag was carried out with a

time variation of 15 to 240 minutes, the temperature of 30 °C, 70 °C, and 100 °C and NaOH concentrations are 9 M, 10 M, and 11 M. The XRD (X-ray diffraction) analysis, (XRF X-ray fluorescence) analysis, SEM (Scanning Electron Microscope) analysis, and ICP-OES (Inductively Coupled Plasma) analysis were used to observe the initial characteristics of the ferronickel slag and the results after the leaching process. The characterization result towards ferronickel slag samples by XRD analysis shows that the compositions of the dominant compounds are forsterite (Mg_2SiO_4), enstatite ($MgSiO_3$) and fayalite (Fe_2SiO_4). Moreover, the result is also supported by XRF analysis and SEM mapping analysis. The quantitative analysis of XRF shows that ferronickel slag contains 45.69 % of SiO_2 , 29.32 % of MgO and 16.5 % of Fe_2O_3 . The results of the SEM mapping analysis show that Mg, Si, Fe and O bond together that indicates the presence of magnesium silicate and iron silicate. The highest percentage of magnesium extraction is 73.10 % under experimental temperature conditions of 100 °C for 240 minutes, 10 M of solvent concentration and 300 rpm of stirring speed. Increasing percentage of magnesium extraction is caused by the dissolution of silica in the leaching process. *The dissolution of silica is proved by the existence of magnesium hydroxide and iron(II) hydroxide in the residue that is shown by the XRD analysis.* It resulted in the MgO content in the residue increase significantly to 42.8 % as shown by the XRF analysis. Moreover, the SEM mapping analysis shows that Mg and O bond together that indicated the presence of MgO . It also can be determined that MgO is dominant.

Keywords: ferronickel, slag, forsterite, magnesium, silica, leaching, reverse leaching, sodium hydroxide, filtrate, residue, % extraction.

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DOI: 10.15587/1729-4061.2020.194618

**INVESTIGATION OF CHARACTERISTICS
OF BINARY Ni–Co OXYHYDROXIDES FOR
SUPERCAPACITOR APPLICATION (p. 15–23)**

Vadym Kovalenko

Ukrainian State University of Chemical Technology,
Dnipro, Ukraine
Vyatka State University, Kirov, Russian Federation
ORCID: <http://orcid.org/0000-0002-8012-6732>

Valerii Kotok

Ukrainian State University of Chemical Technology,
Dnipro, Ukraine
Vyatka State University, Kirov, Russian Federation
ORCID: <http://orcid.org/0000-0001-8879-7189>

Alexey Sykchin

Ukrainian State University of Chemical Technology,
Dnipro, Ukraine
Vyatka State University, Kirov, Russian Federation
ORCID: <http://orcid.org/0000-0003-2214-4198>

Ihor Kovalenko

Ukrainian State University of Chemical Technology,
Dnipro, Ukraine
ORCID: <http://orcid.org/0000-0002-7747-0911>

Oksana Berzenina

Ukrainian State University of Chemical Technology,
Dnipro, Ukraine
ORCID: <http://orcid.org/0000-0002-1786-859X>

Viktoria Stoliarenko

Kryvyi Rih State Pedagogical University,
Kryvyi Rih, Ukraine
ORCID: <http://orcid.org/0000-0002-4665-5710>

Iryna Plaksienko

Poltava State Agrarian Academy, Poltava, Ukraine
ORCID: <http://orcid.org/0000-0002-1002-4984>

Pavlo Pysarenko

Poltava State Agrarian Academy, Poltava, Ukraine
ORCID: <http://orcid.org/0000-0002-4915-265X>

Marina Samojlik

Poltava State Agrarian Academy, Poltava, Ukraine
ORCID: <http://orcid.org/0000-0003-2410-865X>

Binary Ni–Co compounds, namely oxyhydroxides and cobaltates of nickel, are promising active compounds for supercapacitors. The characteristics of binary Ni–Co oxyhydroxides synthesized using the method of high-temperature two-stage synthesis with hot and cold hydrolysis were studied. The crystal structure of the samples was studied by means of X-ray diffraction and thermogravimetry, particle morphology –scanning electron microscopy, electrochemi-

cal characteristics – cyclic voltammetry and galvanostatic charge-discharge cycling.

By means of scanning electron microscopy, it was found that the samples of cold and hot hydrolysis are nano-structured powders composed of flower-like particles, composed of 70–90 nm thick plates. The results of XRD and thermogravimetric analyses revealed that both samples are binary Ni–Co oxyhydroxides (hydrated nickel cobaltates with different hydration levels) with the presence of pure nickel cobaltate. Hot hydrolysis samples contain less water and more nickel cobaltate. Cyclic voltammetry and galvanostatic charge-discharge cycling revealed that in the cold hydrolysis Ni–Co sample, only the nickel component is electrochemically active. The maximum capacity of the cold hydrolysis sample is 185.7 F/g (at 10 mA/cm²). With the increase of current density to 120 mA/cm², the specific capacity drops by 4.47 times. The hot hydrolysis sample was found to have both nickel and cobalt components active: the sample having increased capacity with increasing current density from 10 mA/cm² to 120 mA/cm² by 1.25 times, up to 192.5 F/g. The hot hydrolysis sample was found to possess high reversibility and high effectiveness of the electrochemical component from cycle 1.

Keywords: binary Ni–Co oxyhydroxide, nickel cobaltate, high-temperature two-stage synthesis, specific capacity, supercapacitor.

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DOI: 10.15587/1729-4061.2020.196653

REVEALING NEW PATTERNS IN RESOURCE-SAVING PROCESSING OF CHROMIUM-CONTAINING ORE RAW MATERIALS BY SOLID-PHASE REDUCTION (p. 24–29)

Viacheslav Borysov

Donbass Institute of Technique and Management Private Higher Educational Establishment “Academician Yuriy Bugay Internationalscientific and Technical University”, Kramatorsk, Ukraine

ORCID: <http://orcid.org/0000-0002-3117-2118>

Ihor Hevko

Donbass Institute of Technique and Management Private Higher Educational Establishment “Academician Yuriy Bugay Internationalscientific and Technical University”, Kramatorsk, Ukraine

ORCID: <http://orcid.org/0000-0003-1325-1434>

Oleksii Torubara

Donbass Institute of Technique and Management Private Higher Educational Establishment “Academician Yuriy Bugay Internationalscientific and Technical University”, Kramatorsk, Ukraine

ORCID: <http://orcid.org/0000-0001-7174-4611>

Svitlana Borysova

Donbass Institute of Technique and Management Private Higher Educational Establishment “Academician Yuriy Bugay Internationalscientific and Technical University”, Kramatorsk, Ukraine

ORCID: <http://orcid.org/0000-0003-0610-644X>

Dmitry Milko

Dmytro Motornyi Tavria State Agrotechnological University, Melitopol, Ukraine

ORCID: <http://orcid.org/0000-0002-0991-1930>

Dmytro Zhuravel

Dmytro Motornyi Tavria State Agrotechnological University, Melitopol, Ukraine

ORCID: <http://orcid.org/0000-0002-6100-895X>

Bohdan Tsymbal

National University of Civil Defence of Ukraine, Kharkiv, Ukraine

ORCID: <http://orcid.org/0000-0002-2317-3428>

Viacheslav Bratishko

National University of Life and Environmental Sciences of Ukraine, Kyiv, Ukraine

ORCID: <http://orcid.org/0000-0001-8003-5016>

Kyrylo Samoichuk

Dmytro Motornyi Tavria State Agrotechnological University, Melitopol, Ukraine

ORCID: <http://orcid.org/0000-0002-3423-3510>

Yulia Postol

Dmytro Motornyi Tavria State Agrotechnological University, Melitopol, Ukraine

ORCID: <http://orcid.org/0000-0002-0749-3771>

The physical and chemical properties of products from the carbon-thermal reduction of oxide chromo-containing

ore raw materials have been investigated. This is necessary to determine the parameters that reduce the loss of Cr in the processing of ore materials and the use of metallized chromium doping additives in steelmaking. It has been determined that the increase in processing temperature from 1,250 K to 1,450 K led to an increase in the manifestation of Cr_{23}C_6 and $(\text{Cr}, \text{Fe})_7\text{C}_3$. In this case, the diffraction maxima of Cr_2O_3 corresponded to the trend of weakening and, having been treated at 1,450 K, had a residual character. Cr_3C_2 on the diffractograms was only evident after processing at 1,250 K. The phase of the metallic Cr was traced in the samples after processing at 1,350 K and 1,450 K with the increased intensity of manifestation when the heating temperature rose. It has been determined that the microstructure of reduction products is heterogeneous with the presence of particles of different sizes and chemical compositions. The increase in the reduction temperature from 1,250 K to 1,350 K and 1,450 K and the development of reduction processes were accompanied by particle sintering with the formation of a spongy microstructure. We have detected regions that characterized inclusions and the phases where Cr content amounted to 65.10 % by weight, Fe – to 16.13 % by weight. Some local areas with particles with a relatively high content of ore impurities and carbon have also been found. It follows from the results of our study that the most acceptable temperature for reduction is 1,450 K. In this case, the reduction is ensured with a predominance in the phase composition of the metal Cr and carbides $(\text{Cr}, \text{Fe})_7\text{C}_3$ and Cr_{23}C_6 relative to the oxide component of Cr_2O_3 . In this case, the lower residual carbon content was due to the higher efficiency of the reducer compared to other temperature regimes.

The spongy microstructure allows for a faster dissolution compared to standard ferroalloys, thereby reducing the duration of smelting.

Keywords: chromium-containing oxide ore raw materials, phase analysis, carbon thermal treatment, carbide, doping, reduction.

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DOI: 10.15587/1729-4061.2020.193932

MICROSTRUCTURE, MECHANICAL PROPERTIES, AND CORROSION BEHAVIOR OF NEW β -TYPE Ti–Mo–Nb BASED ALLOYS BY Mn ADDITION FOR IMPLANT MATERIAL (p. 30–37)

Cahya Sutowo

Universitas Indonesia, Jawa Barat, Indonesia

ORCID: <http://orcid.org/0000-0002-3724-8443>

Sugeng Supriadi

Universitas Indonesia, Jawa Barat, Indonesia

ORCID: <http://orcid.org/0000-0001-8153-4036>

Andika Widya Pramono

Indonesian Institute of Sciences,

Tangerang Selatan, Indonesia

ORCID: <http://orcid.org/0000-0003-2920-4093>

Bambang Suharno

Universitas Indonesia, Jawa Barat, Indonesia

ORCID: <http://orcid.org/0000-0002-9029-2726>

Titanium alloys are widely used for biomaterial applications since they have special characteristics, especially better biocompatibility, superior corrosion behavior and lower modulus of elasticity compared to other conventional biomaterials. The development of existing Ti6Al4V alloys by creating new β -type Ti–Mo–Nb based alloys by modifying the addition of the Mn element as a beta phase stabilizer, so that the beta phase structure can have an effect to increase strength and reduce elastic modulus with good biocompatibility and toxicity. In the present work, Ti–Mo–Nb–(x) Mn alloys (x=0, 4, 8, and 12, mass fraction in %) were prepared using an electric vacuum arc furnace with a tungsten electrode. The samples were homogenized at 1050°C for 6 h under a controlled argon atmosphere, and the effects of adding Mn on the mechanical properties and corrosion behavior of the alloys were investigated using X-ray fluorescence spectroscopy, X-ray diffraction, optical microscopy, hardness and ultrasonic tests, and potentiodynamic polarization test.

The experimental results show that adding 4 %, 8 %, and 12 %Mn to a Ti–9Mo–6Nb alloy stabilizes the formation of the β -phase titanium, implying that the alloys have similar microstructures but different grain sizes. Potentiodynamic polarization measurements show that an increase of the Mn content in the Ti–9Mo–6Nb alloy decreases the corrosion resistance. At 4 %Mn, the alloy has an elastic modulus of 93 GPa and better corrosion resistance, with a relatively low corrosion rate amounting to 0.00290 mm per year, than those of a commercial Ti–6Al–4V alloy.

Keywords: titanium, titanium alloy, β -type titanium, TiMoNb alloy, biomaterial, mechanical properties, elastic modulus, corrosion resistance, implant material.

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DOI: 10.15587/1729-4061.2020.194164

FORMING THE STRUCTURE AND THE PROPERTIES OF ELECTRIC ARC COATINGS BASED ON HIGH MANGANESE STEEL ALLOYED WITH TITANIUM AND NIOBIUM CARBIDES (p. 38–44)

Shihab Thaer Abdulwahhab Shihab

Middle Technical University/Engineering Technical College of Baghdad, Baghdad, Iraq

ORCID: <http://orcid.org/0000-0002-8978-9436>**Pavlo Prysyazhnyuk**

Ivano-Frankivsk National Technical University of Oil and Gas, Ivano-Frankivsk, Ukraine

ORCID: <http://orcid.org/0000-0002-8325-3745>**Roman Andrusyshyn**

Ivano-Frankivsk National Technical University of Oil and Gas, Ivano-Frankivsk, Ukraine

ORCID: <http://orcid.org/0000-0003-4176-4026>**Lyubomyr Lutsak**

Limited Liability Company Interdisciplinary Research and Production Center "Epsilon LTD", Ivano-Frankivsk, Ukraine

ORCID: <http://orcid.org/0000-0002-8111-5461>**Alexandr Ivanov**

College of Electronic Devices IFNTUOG, Ivano-Frankivsk, Ukraine

ORCID: <http://orcid.org/0000-0003-4678-7956>**Igor Tsap**

Ivano-Frankivsk National Technical University of Oil and Gas, Ivano-Frankivsk, Ukraine

ORCID: <http://orcid.org/0000-0003-3085-3699>

The formation of the phase composition, structure, and properties of electric-arc coatings by electrode materials based on high-manganese austenite steel, which is strengthened by twinning steel alloyed by titanium and niobium carbides, was studied. Hardfacing alloys were applied using flux-cored arc welding (FCAW), which consisted of a shell of low-carbon steel filled with a powder mixture containing ferrosilicon manganese, graphite, rutile, fluorite, carbide of niobium or titanium.

The phase composition of the coatings was calculated by the CALPHAD method using the Thermo-Calc and Dictra software to simulate equilibrium and non-equilibrium alloy cooling, respectively. The results of calculations show that the cooling conditions at hardfacing lead to the formation of the structure of metastable manganese austenite and minor (0.2 % by weight) amount of carbide of cementite type M_3C . This amount of the carbide phase cannot adversely affect the ability of austenite to deformation strengthening. Crystallization of titanium and niobium carbides begins at a high temperature of ~2,400 K and does not affect the phase transformations of austenite into the liquid and solid state. Thus, after cooling, the coating structure mainly consists of two phases, specifically, austenite and niobium or titanium carbide MC in an amount of ~9 vol. %.

According to the results from studying the microstructure using scanning electron microscopy and energy-dispersive X-ray spectroscopy of the selected area, the MC carbide phase is released as evenly distributed small particles of the shape that is close to cubic. Carbide particles are mainly located inside austenite grains.

Measurement of the hardness of hardfacing coatings shows that at alloying high-manganese austenite steel with niobium and titanium carbides, the hardness significantly increases in hardfacing state from 22 to 35 HRC and after cold plastic deformation from 44 to 52 HRC. The research of wear resistance under conditions of wear by the "dry sand – rubber roller" pattern shows that TiC and NbC additives can significantly (by 1.6–1.8 times) improve wear resistance of hardfacing coatings.

Due to the ability to deformation strengthening in combination with high abrasion resistance, the obtained materials for hardfacing the systems Fe–Mn–Nb–Si–C and Fe–Mn–Ti–Si–C can be recommended for application to working surfaces of digging machines.

Keywords: flux-core wire, high-manganese steel, niobium carbide, titanium carbide, deformation strengthening, abrasive wear, arc welding.

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DOI: 10.15587/1729-4061.2020.156798

EFFECT OF AUSTENITE TEMPERATURE AND HOLDING TIME TO IMPACT ENERGY AND WEAR ON HRP STEEL (p. 45-51)

Yurianto Yurianto

Diponegoro University, Kota Semarang,
Jawa Tengah, Indonesia

ORCID: <http://orcid.org/0000-0002-3403-2233>

Agus Suprihanto

Diponegoro University, Kota Semarang,
Jawa Tengah, Indonesia

ORCID: <http://orcid.org/0000-0002-3321-1328>

Sumar Hadi Suryo

Diponegoro University, Kota Semarang,
Jawa Tengah, Indonesia

ORCID: <http://orcid.org/0000-0002-5476-1994>

Yusuf Umardani

Diponegoro University, Kota Semarang,
Jawa Tengah, Indonesia

ORCID: <http://orcid.org/0000-0003-3098-6408>

Padang Yanuar

Semarang State Polytechnic, Semarang,
Central of Java, Indonesia

ORCID: <http://orcid.org/0000-0002-0806-9585>

Quenched and Tempered Steel is classified as high strength and hardness steel mostly used for armor steel. Hardness and wear stability is essential for steel armor quality standards. On the other hand, austenitization and tempering temperature affect the impact value of the energy absorbed and the Quenched and Tempered Steel wear resistance value. This study aims to evaluate the effects of austenitization and holding time on energy and hardness of Hot Rolled Plate Steel. The research material is a hot roller plate made by Krakatau Steel Indonesia Company Limited with a carbon content of about 0.29 %. The method used was heating of three specimens at 900 (held 45 minutes), 900 (held 30 minutes), and 900 (held 15 minutes), and all of them cooled into the water when the end of heating. Three specimens were heated at 885 (for 45 minutes), 885 (for 30 minutes), and 885 (for 15 minutes) and cooled into the water. Three specimens were heated at 870 (kept 45 minutes) 870 (held 30 minutes), and 870 (kept 15 minutes) and cooled into water. Then the final cooling of the nine specimens was heated at 150 for 30 minutes and cooled to the atmosphere. Five specimens tested for the hardness of Charpy and Vickers. Then arranged into an orthogonal matrix; calculate degrees of freedom; signal to noise ratio, quadratic parameters; average squared response; the ratio between the average response of the square of the source and the error; contribution parameters; predictive value and confidence interval. From the calculation above, the heat treatment parameters have a strong influence on the energy impact and the specific wear obtained. The most influential parameter on the energy impact is the tempering

temperature because this parameter is softening caused by a reduction in residual stress due to the previous cooling process. The fine-grain structure increases strength and the energy impact directly.

Keywords: brittle, ductility, hardness, heating, holding, impact, quench, temper, water, wear.

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