

UNIVERSITY OF BELGRADE
TECHNICAL FACULTY BOR

**52nd International October Conference on
Mining and Metallurgy**



PROCEEDINGS

Edited by

Saša Stojadinović

and

Dejan Petrović

November 29th – 30th 2021

Bor, Serbia

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TABLE OF CONTENTS

Aleksandra Milosavljević	
THE COMPLEXITY OF SEM-EDS – WHAT AFFECTS THE QUALITY OF OBTAINED RESULTS?	1
Zoran Karastojković, R Perić, M Srečković	
LASER QUENCHING OF CUTTING TOOL STEELS - A REVIEW	5
Slavica Miletić, D Bogdanović, E Požega	
IMPACT OF EXTRAORDINARY SECURITY MEASURES TO EMPLOYEES DURING THE PANDEMIC COVID-9	15
Daniela Grigorova, R Paunova	
KINETIC STUDY OF SOLID-PHASE REDUCTION OF POLYGRADIENT IRON-CONTAINING MATERIAL	19
Emina Požega, D Simonović, S Marjanović, M Jovanović, L Gomidželović, M Mitrović, Z Stanojević Šimšić	
PART I: WHAT MAKES A GOOD THERMOELECTRIC	23
Emina Požega, D Simonović, S Marjanović, M Jovanović, L Gomidželović, M Mitrović, S Miletić	
PART II: WHAT MAKES A GOOD THERMOELECTRIC	27
Dragan Manasijević, Lj Balanović, I Marković, M Gorgievski, U Stamenković, K Božinović, D Minić, M Premović	
STUDY OF MICROSTRUCTURE AND THERMAL CONDUCTIVITY OF THE Ag–Bi–Sn ALLOYS	31
Vladimir S. Topalović, S Matijašević, S Grujić, J Stojanović, J Nikolić, V Savić, S Zildžović	
THE INFLUENCE OF THE PARTICLE SIZE ON CRYSTALLIZATION OF GLASS POWDERS FROM THE SYSTEM $\text{Li}_2\text{O}-\text{Al}_2\text{O}_3-\text{GeO}_2-\text{P}_2\text{O}_5$	35
Vesna Marjanović, R Marković, V Krstić	
TECHNOLOGIES FOR PHYSICAL TREATMENT OF WATER CONTAINING SELENIUM: A REVIEW	39
Vesna Marjanović, R Marković, V Krstić	
TECHNOLOGIES FOR BIOLOGICAL TREATMENT OF WATER CONTAINING SELENIUM: A REVIEW	43
Milenko Jovanović, M Mikić, M Maksimović, D Kržanović, R Rajković, E Požega	
USAGE SPECIFICS OF GEOGRIDS	47

Srećko Manasijević, Z Zovko Brodarac, N Dolić, M Djurdjević, R Radiša	
INTERMETALLIC BONDING BETWEEN A RING CARRIER AND AN ALUMINUM PISTON ALLOY	51
Snežana Šarboh	
PATENTED INVENTIONS OF LJUBOMIR KLERIĆ	55
Miomir Mikic, M Jovanović, R Rajković, D Kržanović, E Požega	
DEGRADED AREA OF VELIKI KRIVELJ QUARRY RECULTIVATION	59
Dragana Adamović, D Ishiyama, H Kawaraya, O Yasumasa	
EFFECTS OF TAILINGS ON GROUNDWATER ALONG BOR AND BELA RIVERS IN THE BOR MINING AREA, EASTERN SERBIA	63
Ana Kostov, Z Stanojević Šimšić, A Milosavljević,	
CHARACTERIZATION OF ALLOYS CuAlAu0.5	67
Marija Milenković, V Jovanović, J Paunković, V Krstić	
MULTICRITERIA ANALYSIS OF THE LEVEL OF SUSTAINABLE DEVELOPMENT OF THE TOPLICA DISTRICT USING THE ELECTRE METHOD	71
Daniel Kržanović, R Rajković, D Stevanović, M Mikić, M Jovanović, S Petrović	
LONG-TERM PLANNING OF MINING THE LEAD AND ZINC ORE DEPOSIT IN THE BRSKOVO ORE FIELD, THE REPUBLIC OF MONTENEGRO	75
Radmilo Rajković, D Kržanović, M Mikić, M Jovanović	
CALCULATION OF SAFETY DISTANCE FOR THE OPERATION OF MINING EQUIPMENT IN THE WORKING ENVIRONMENT WITH WEAKENED CHARACTERISTICS AT THE OPEN PIT "NORTH MINING DISTRICT" OF THE COPPER MINE MAJDANPEK	79
Zdenka Stanojević Šimšić, A Kostov, A Milosavljević, E Požega	
HARDNESS, MICROHARDNESS AND ELECTROCONDUCTIVITY OF ALLOYS WITH VARIABLE Cu CONTENT IN Cu-Al-Ag SYSTEM	83
Miodrag Banješević	
STRATIGRAPHY AND AGE OF ROCK UNITS AND MINERALIZATION IN THE TIMOK MAGMATIC COMPLEX AND THE BOR METALLOGENIC ZONE – A REVIEW	87
Milan Radivojević, Z Stević, M Tanasković	
DUALPHASED FOURWAY INTERSECTION REGULATED BY TRAFFIC LIGHTS WITH FIXED AND ADAPTIVE MOD OF OPERATION	93
Filip Gramić, N Rančić, S Filipović, J Đorđević	
USE OF COPPER TAILING AND COPPER SLAG IN 3D PRINTED CONCRETE PROCESSES	97
Filip Gramić, N Rančić, S Filipović, J Đorđević,	
POSSIBILITY OF USING MINING WASTE IN THE PRODUCTION OF BRICK PRODUCTS	101

Stepan O. Vidysh

GOLD-SILVER ALLOYS ANODIC DISSOLUTION RESEARCH IN HYDROCHLORIC ACID ELECTROLYTES 105

Milan Gorgievski, M Marković, D Božić, Vr Stanković, N Štrbac, V Grekulović, M Zdravković

ADSORPTION ISOTHERMS FOR COPPER IONS ADSORPTION ONTO WALNUT SHELLS 109

Miljan Marković, M Gorgievski, N Štrbac, V Grekulović, A Mitovski, K Božinović, M Zdravković

pH AND CONDUCTIVITY CHANGE DURING THE RINSING AND ADSORPTION OF COPPER IONS ONTO WALNUT SHELLS 113

Vesna Grekulović, A Mitovski, M Rajčić Vujasinović, N Štrbac, M Zdravković, M Gorgievski, M Marković

ELECTROCHEMICAL BEHAVIOR OF COPPER IN CHLORIDE MEDIUM IN THE PRESENCE OF WALNUT SHELL MACERATE 117

Marija Šljivić-Ivanović, S Dimović, I Jelić,

EXPERIMENTAL DESIGN APPROACH IN RADIONUCLIDE SORPTION 121

Ivana Jelić, A Savić, M Šljivić-Ivanović, S Dimović

INFLUENCE OF SILICA FUME ON SCC CONCRETE PROPERTIES 125

Milan Radovanović, A Simonović, M Petrović Mihajlović, Ž Tasić, V Nedelkovski, M Antonijević

L-LYSINE AS CORROSION INHIBITOR OF STAINLESS STEEL IN RINGER'S SOLUTION 129

Dragana Marilović, M Trumić, M Trumić, Lj Andrić

THE INFLUENCE OF CALCIUM IONS ON DEINKING FLOTATION RECOVERY UNDER DIFFERENT CONDITIONS 133

Dragana Medić, S Milić, S Alagić, M Nujkić, S Đorđević, A Papludis

OPTIMIZATION OF CATHODIC MATERIAL LEACHING PROCESS IN ACID-SULPHATE SOLUTION 137

Milijana Mitrović, D Gusković, S Marjanović, B Trumić, E Požega, U Stamenković, J Petrović

OBTAINING MULTILAYER COPPER STRIPS BY ARB (ACCUMULATIVE ROLL BONDING) ROLLING PROCESS 141

Nataša Đorđević, S Mihajlović, N Obradović, A Peleš, S Filipović

THE INFLUENCE OF HIGH COMPACTION PRESSURE ON CORDIERITE-BASED CERAMICS 145

Nataša Đorđević, S Mihajlović, M Sokić, B Marković

SEM AND X-RAY ANALYSES OF SINTERED MgO / Bi₂O₃ BINARY SYSTEM 149

Ivana Ilić, J Sokolović, M Trumić, Z Stirbanović	
COMPARATIVE RESULTS OF COPPER FLOTATION FROM SLAG BEFORE AND AFTER THE PROCESS OF MAGNETIC CONCENTRATION	153
Daniela Grigorova	
FERROSILICON OBTAINING USING IRON-SILICATE –FAYALITE	157
Slavica Mihajlović, M Jovanović, N Đorđević, A Patarić, M Vlahović, V Kašić	
THE CLAY PRELIMINARY TESTING FROM MUNICIPALITY AREA OF REKOVAC	161
Milan Milosavljević, M Premović, D Minić, Dn Mansijević, Ar Đorđević, M Kolarević	
EXPERIMENTAL AND THERMODYNAMIC STUDY OF ISOTHERMAL SECTIONS AT 600 °C AND 400 °C OF TERNARY Bi-Cu-Ge SYSTEM	165
Aleksandar Đorđević, D Minić, M Premović, D Mansijević, M Milosavljević, V Ristić	
STUDY OF TEMPERATURE PHASE TRANSFORMATION OF THE TERNARY Bi-Cu-Ge SYSTEM	169
Aleksandar Savić, I Jelić, M Šljivić-Ivanović, S Dimović, N Pudar, A Pfandler	
RECYCLED COARSE AGGREGATE AND FLY ASH EFFECT ON COMPRESSIVE STRENGTH OF SELF-COMPACTING CONCRETE	173
Vladan Kašić, D Životić, V Simić, A Radosavljević-Mihajlović, J Stojanović, S Mihajlović, M Vukadinović	
FORECAST RESOURCES OF ZEOLITHIC TUFFS OF SERBIA	177
Vladan Kašić, A Radosavljević-Mihajlović, S Radosavljević, J Stojanović, S Mihajlović, M Vukadinović	
GEOLOGICAL AND MINERAL CHARACTERISTICS OF ZEOLITHIC TUFF TOPONICA DEPOSITS NEAR KOSOVSKA KAMENICA	181
Konstantin Petkov, V Stefanova, P Iliev	
METHOD FOR UTILIZATION OF THE SULFURIC ACID OBTAINED DURING AUTOCLAVE DISSOLUTION OF PYRITE CONCENTRATE	185
Stefan Đorđević, D Ishiyama, Y Ogawa, Z Stevanović, O Osenyeng, D Adamović, V Trifunović	
MONITORING OF pH VALUE AND CONCENTRATION OF COPPER IN RIVERS DOWNSTREAM FROM BOR MINE IN PERIOD 2015-2021	189
Viša Tasić, M Cocić, B Radović, T Apostolovski-Trujić	
CHEMICAL COMPOSITION OF PARTICULATE MATTER IN THE INDOOR AIR AT THE TECHNICAL FACULTY IN BOR (SERBIA)	193
Snežana Ignjatović, I Vasiljević, M Negovanović	
DEFINING STRUCTURAL CORRELATION USING OF TOTAL HORIZONTAL GRADIENT	197

Velizar Stanković, M Janošević

**INCREASING THE CAPACITY OF THE COPPER SMELTING COMPANY IN THE COMPANY
"SERBIA ZIJIN COPPER" - CHALLENGES AND CONSEQUENCES TO THE ENVIRONMENT** 201

Vladimir Jovanović, D Todorović, B Ivošević, D Radulović, S Milićević, D Nišić

**CHARACTERIZATION OF PELLET SAMPLES OBTAINED BY PELETIZATION OF LIMESTONE
AND SEAWEED** 205

Vanja Trifunović, L Avramović, R Jonović, S Milić, S Đorđievski, M Jonović

**HYDROMETALLURGICAL TREATMENT OF ELECTRIC ARC FURNACE DUST IN AIM OF ZINC
SEPARATION** 209

Jovana Bošnjaković, N Knežević, N Čutović, M Bugarčić, A Jovanović, Z Veličković, S
Manasijević

**EVALUATION OF ADSORPTION PERFORMANCE OF PHOSPHATES REMOVAL USING CELL-
MG HYBRID ADSORBENT** 213

Dragan Radulović, Lj Andrić, D Božović, V Jovanović, B Ivošević, D Todorović,

**POSSIBILITY OF USING LIMESTONE FROM "PJEŠIVAČKI DO"-DANILOVGRAD DEPOSIT AS
FILLER IN VARIOUS INDUSTRY BRANCHES** 217

Predrag Stolić, J Ivaz, D Petrović, Zoran Stević

**ADVANTAGES OF MINING ENGINEERING CURRICULUM REALIZATION USING
SOLUTIONS BASED ON FREE SOFTWARE** 221

Slađana Krstić, E Požega, S Petrović, S Magdalinović, D Urošević, S Miletić, Z Stojanović
Šimšić

**QUALITY INVESTIGATION OF SAND FOR THE PRODUCTION OF AGGREGATES ON
VINOGRADI LOCALITY (DELIBLATSKA PEŠČARA)** 225

Saša Marjanović, D Gusković, M Mitrović, E Požega, B Trumić, U Stamenković

**INFLUENCE OF COLD ROLLING AND ANNEALING ON HARDNESS OF BIMETALLIC STRIP
Cu– Al** 229

SEM AND X-RAY ANALYSES OF SINTERED MgO / Bi₂O₃ BINARY SYSTEM

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Abstract

Magnesium oxide is a component of many ceramic materials. Bismuth oxide is added as an additive to ceramic materials in order to lower the sintering temperature. The influence of temperature was researched by sintering binary system: MgO/Bi₂O₃. The temperature of process was 820 °C and 1100 °C. Composition of this system was 80% of MgO and 20% Bi₂O₃. The effects of sintering, the composition and morphology were followed by X-ray diffraction and Scanning electron microscopy. It has been found that Bi₂O₃ forms intermediary unstable compound with MgO.

Keywords: MgO, Bi₂O₃, sintering, SEM, x-ray analysis.

1. INTRODUCTION

These studies were performed in order to determine the influence of bismuth oxide on the sintering process of ceramic materials and the possibility of reaction during sintering with magnesium oxide as one of the components of the system.

One of the most important ceramic materials whose composition includes magnesium oxide is cordierite. Its composition is 2MgO*2Al₂O₃*5SiO₂. Since the temperature range of cordierite sintering is very narrow (sintered temperature is 1300 – 1400 °C), the techniques of lowering the temperature of its formation are very interesting for research.

As the melting point of bismuth oxide is 750 °C, adding the cordierite mixture during the sintering process is the expected result of lowering the sintering temperature due to the formation of a two-phase system. During sintering, it is possible to form intermediate compounds of additives with system components, which was the aim of this research.

2. EXPERIMENTAL

During this research following components were used: MgO (Euro Hemija, Beograd), Al₂O₃ (Aluminijumskikombinat, Podgorica), SiO₂ (Bela Reka) and Bi₂O₃, p.a. (Reahim, Rusia). Chemical composition of starting compounds is given in Table 1.

Following mixture was prepared: MgO/Bi₂O₃, in relation 80% of magnesium oxide and 10% of Bi₂O₃. Samples for sintering were prepared in the tablet shape, with radius 8mm and height 4mm, under the pressure of 1 t/cm². Sintering temperature of the sample MgO/Bi₂O₃ was 820 °C and 1100 °C.

X-ray powder diffraction (XRPD) technique was used for identification and definition of the unit-cell parameters. XRPD analysis was performed using the Philips PW1710 diffractometer; with Cu K α radiation (40kV, 30mA), step scan 0.25s, 0.02° 2 θ , d range from 5° to 85°2 θ . The microstructures of the sintered samples were observed using scanning electron micrograph (SEM) with microsonde.

3. RESULTS AND DISCUSSION

Sample MgO/Bi₂O₃, sintered at 820°C (Fig.1.) showed very low level of crystallinity. Following compounds were detected: periclase (MgO), bismite (α -Bi₂O₃) and silenite (γ -Bi₂O₃). Under these conditions, only Bi₂O₃ was phase transformed, other phases were not formed since this temperature was too low for any other transformations. In the sample MgO/Bi₂O₃ after sintering at 1100°C, periclase (MgO), mixture of bismuth oxide (α , β and γ Bi₂O₃) and unstable compound - Bi₁₂MgO₁₉ were found.

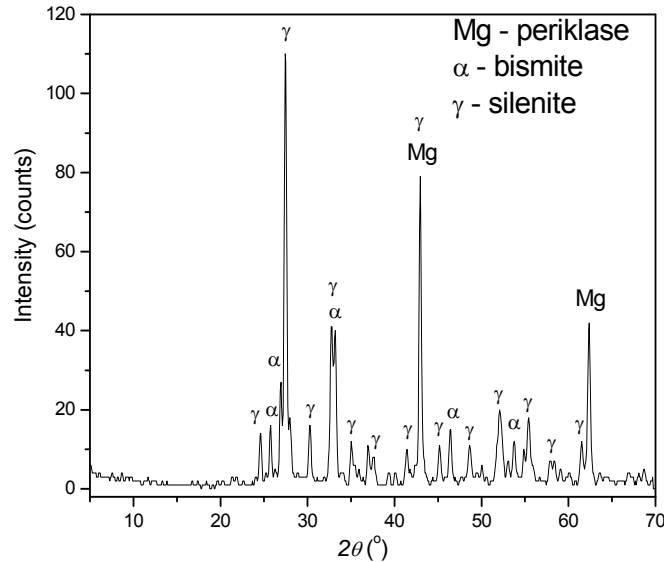


Figure 1. X-ray diffraction patterns of the sample MgO/Bi₂O₃ sintered at 820°C

SEM microphotograph of the sample MgO/Bi₂O₃, (Fig.2) sintered at 820°C, is shown in the Figure 3. The grains with flat surface could be notice. With the magnification of 2000X the pyramidal structure (5-10 μ m) and microstructure with spherical shape (dimension 100-400nm) are visible.

The microphotographs of the samples sintered at 1100°C are morphologically different from the sample sintered at 820°C. Small-grain structure of the crystal (visible with magnification of 25000X and pointed to initial sintering), by this microphotograph is well visible (Figure 4). The grains sizes \sim 100nm at 820 °C after sintering at 1100 °C have dimensions sized in μ m. Pyramidal structure could not be founded in this sample.

Crystals are isometrics, enhydral. The grains are rounded, well developed, clearly visible, with diameter of \sim 1 μ m, symmetrically formed, flat surface boarded.

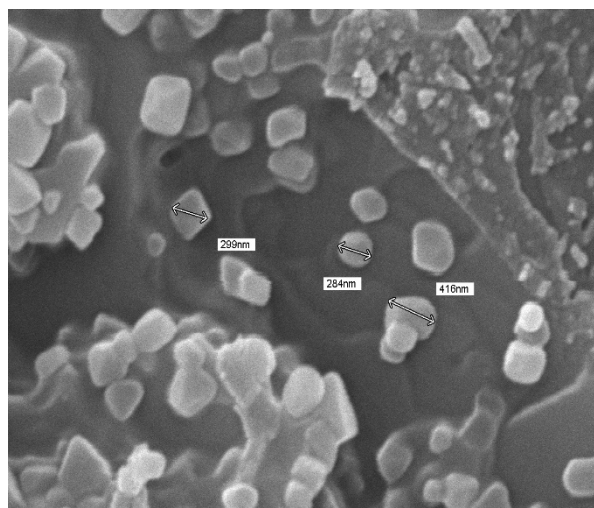


Figure 2. SEM microphotographs of the sample MgO/Bi₂O₃ sintered at 820°C (3000 X)

SEM microphotograph of the sample MgO/Bi₂O₃, (Fig.2) sintered at 820°C, is shown in the Figure 3. The grains with flat surface could be notice. With the magnification of 2000X the pyramidal structure (5-10µm) and microstructure with spherical shape (dimension 100-400nm) are visible.

The microphotographs of the samples sintered at 1100°C are morphologically different from the sample sintered at 820°C. Small-grain structure of the crystal (visible with magnification of 25000X and pointed to initial sintering), by this microphotograph is well visible (Figure 4). The grains sizes ~100nm at 820 °C after sintering at 1100 °C have dimensions sized in µm. Pyramidal structure could not be founded in this sample.

Crystals are isometrics, enhydral. The grains are rounded, well developed, clearly visible, with diameter of ~ 1µm, symmetrically formed, flat surface boarded.

4. CONCLUSIONS

In the aim of researching the reactions of cordierite synthesis, the binary systems were examined as follows: MgO/Bi₂O₃ sintered at 820°C and 1100°C. The results showed liquid phase in the system at 820°C (the melting temperature of Bi₂O₃) and producing meta-stable compounds that form MgO with Bi₂O₃ at 1100°C. This unstable compound transports through the liquid phase, which allowed (from the two aspects) the acceleration of the reaction in the more-component system.

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