

Study on the Preparation and Mechanical Properties of MgAlON Composites

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Abstract

MgAlON composites have been prepared under nitrogen atmosphere, using Dengfeng calcined alumina $w(\text{Al}_2\text{O}_3) = 85\%$ and other raw material, in order to reduce the production cost and make full use of the rich bauxite resources in Henan Province. The effects of the different sintering temperature and different matrix content on the preparation and properties of MgAlON composites were investigated. The results indicated that: apparent porosity, linear expansion, and weight gain had increased; the gradual bulk density decreased with an increased amount of MgAlON, but the room temperature flexural strength increased; the bulk density of the specimen decreased slightly; the apparent porosity increased, room temperature flexural strength increased with the temperature; the best sintered performances of the specimen was at 1500 °C. Compared with the bauxite-based MgAlON composite magnesia material, the alumina-based spinel MgAlON composite material had higher density and better strength effect of nitride.

Keywords

MgAlON composites, sintering, bauxite-based MgAlON, bauxite-based spinel MgAlON

1 Introduction

MgAlON composites have been found to possess superior mechanical properties, thermal shock resistance and slag resistance, and their oxidation resistance is better than that of carbon composite refractory materials.¹⁻³ They may replace carbon composite materials in high temperature furnaces, melting technology of ultra-low carbon steel, and more importantly, MgAlON and its composites have good application prospects in improving the quality of steel.⁴ Synthetic MgAlON and its composites with pure chemical raw materials have some disadvantages (high cost of raw materials, complex preparation process, high requirements for equipment, harsh process conditions, high energy consumption), so they are difficult to popularize in the practical application.⁵ In order to reduce the production cost and make full use of the rich bauxite resources in Henan Province, this paper examines the effects of different sintering temperature and different matrix content on preparation and properties of bauxite-based MgAlON composite.

2 Experimental

Experimental materials: Dengfeng calcined alumina (chemical composition's contents are shown in Table 1); high purity electro-fused magnesia (1–3 mm); fused magnesia alumina spinel (≤ 1 mm); Al powder (≤ 0.088 mm); active Al_2O_3 powder (≤ 5 μm); chemically pure MgO (≤ 0.04 μm); small amount of dextrin as raw material, and a small amount of sintering aids. The ingredient list of the experiments are shown in Table 2.

Table 1 – Chemical components of Dengfeng bauxite

Tablica 1 – Kemijski sastav boksita Dengfeng

Chemical composition Kemijski sastav	Al_2O_3	SiO_2	TiO_2	Fe_2O_3	CaO	MgO	K_2O	Na_2O
w/%	85.44	4.96	4.05	2.16	0.64	0.54	0.25	0.02

Table 2 – Formulation of the specimens

Tablica 2 – Priprema uzoraka

Sample Uzorak	Aggregate / % Agregat / %		Bulk material / % Rasuti materijal / %	
	MgO/MA		MgO/MA	Al (< 0.074 mm) – bauxite (< 0.088 mm) – MgO
grain size veličina zrna	3–1 mm	1–0 mm	< 0.074 mm	
MM1	45	15	20	20
MM2	45	15	10	30
MM3	45	15	0	40
JM1	45	15	20	20
JM2	45	15	10	30
JM3	45	15	0	40

Notes: MM on behalf of bauxite-based MgAlON combined with MgO, JM on behalf of bauxite-based MgAlON combined with MgAl_2O_4 , MgAlON content of 1, 2, 3 respectively 20 %, 30 %, 40 %

Design scheme of weighing various powder was placed into the planetary ball mill to grind for 3 hours; aggregate was added in the ball, dry grinding 3 minutes, add-

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ed dextrin, wet mixing for 3 minutes, and then added mixed powder mixture for mixing 20 minutes. After mixing the ingredients, the strip sample was pressed into 25 mm × 25 mm × 125 mm pieces under the pressure of 150 MPa. The test pieces were put into the drying oven (110 °C) for 24 h, the dried test pieces were put into the high temperature GN-2003-2 MoSi₂ nitriding furnace (N₂ purity of 99.99 %, flow rate of 0.06–0.1 m³ h⁻¹ nitrogen) and heated at different temperatures for about 6 h. After cooling of the specimens, the quality was measured with electronic balance, the rate of weight increase was calculated, nitrogen content was determined by using the national test standard, and other physical properties were tested (bulk density, porosity, compressive strength, and flexural strength at room temperature). The phase composition and microstructure characteristics of samples were analysed by SEM (JEOLJSM-5610LV), and EDS (OXFORD-6587) was used to test the microzone composition of the samples.

3 Results and discussion

3.1 Effect of MgAlON content

After sintering at 1500 °C, the physical properties of the samples were measured. The results are shown in Table 3. The flexural strength of the samples at room temperature are shown in Fig. 1.

Table 3 – Physical properties of the specimens
Tablica 3 – Fizikalna svojstva uzoraka

Sample Uzorak	Apparent porosity / % Prividna poroznost / %	Density / g cm ⁻³ Gustoća / g cm ⁻³	Linear change / % Promjena duljine / %	Weight gain / % Porast mase / %
MM1	28.40	2.53	1.74	0.32
MM2	31.38	2.41	1.81	1.06
MM3	31.88	2.37	1.98	1.82
JM1	23.29	2.69	0.65	1.60
JM2	25.31	2.65	1.09	2.15
JM3	25.56	2.63	1.23	4.12

Table 3 and Fig. 1 show that with an increasing amount of MgAlON content, the volume density of the sample decreases gradually, the apparent porosity, line change rate, weight gain rate increase gradually, and the flexural strength at room temperature shows an upward trend. This indicates that the sintering properties of samples are improved with an increased quantity of MgAlON, which is mainly due to the metal aluminum nitride reaction and nitridation process of weight gain. The combination between the particles and matrix can be conducive, and thereby the density and strength of the specimens can improve, but larger volume expansion in the process affects the material influence porosity and linear change rate.⁶

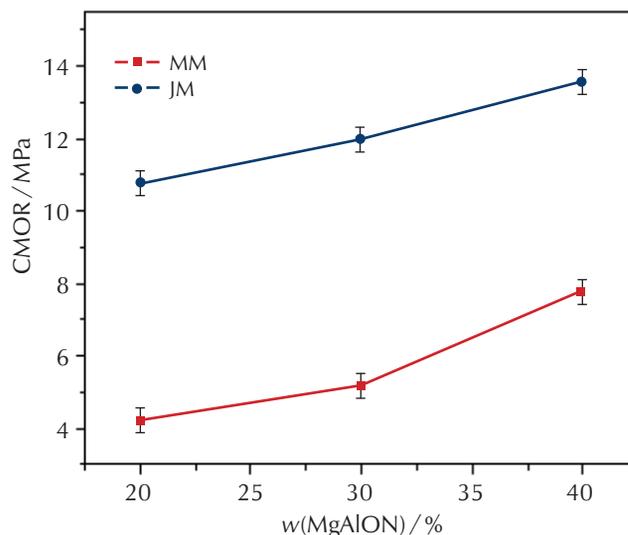


Fig. 1 – Influence of MgAlON content on the CMOR
Slika 1 – Utjecaj sadržaja MgAlON na CMOR

3.2 Effect of sintering temperature

MM3 samples and JM3 samples were used to test the effect of sintering temperature, and compare and analyse sintering properties and microstructure.

3.2.1 Properties of samples at different temperatures

The effects of sintering temperature on the sintering properties and mechanical properties of MgAlON composites at 1300 °C, 1400 °C, and 1500 °C under nitrogen atmosphere were tested. The performances of the samples after sintering temperature are shown in Table 4 and Fig. 2:

Table 4 – Physical properties of the specimens at different temperature

Tablica 4 – Fizikalna svojstva uzoraka dobivenih pri različitim temperaturama

Sample Uzorak	Apparent porosity / % Prividna poroznost / %	Density / g cm ⁻³ Gustoća / g cm ⁻³	Linear change / % Promjena duljine / %	Weight gain rate / % Porast mase / %
MM3-1300	23.57	2.64	1.27	1.71
MM3-1400	27.19	2.56	1.73	1.49
MM3-1500	31.88	2.37	1.98	1.82
JM3-1300	23.69	2.65	0.47	1.74
JM3-1400	25.97	2.63	0.65	2.00
JM4-1500	25.33	2.63	1.23	4.12

It may be seen from Table 4 and Fig. 2 that, during the firing process, the weight of the two samples increase, indicating

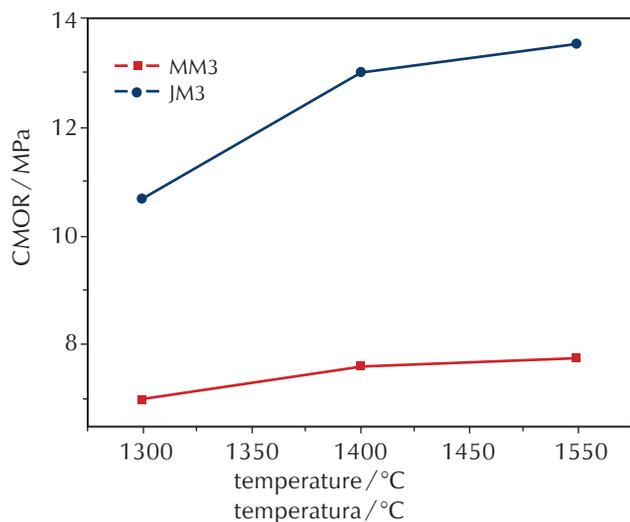


Fig. 2 – Influence of sintering temperature on the CMOR
Slika 2 – Utjecaj temperature sinteriranja na CMOR

that the samples absorbed a certain amount of nitrogen in the reaction process, and the weight gain rate of MM samples is significantly less than that of the JM samples. During the firing process, an expansion effect of the two samples occurred, the change rate of firing line increases with an increase in sintering temperature, and the change rate of the MM samples is larger. Therefore, the sintering properties and the nitriding effect of JM series samples are better than those of MM series samples.

In addition, with the increase in sintering temperature, the volume density of the samples decrease slightly, and the apparent porosity and room temperature flexural strength increase slightly. The change in bulk density and apparent porosity of JM samples is smaller than those of MM samples, so the strength of JM samples increases greatly.

With the increase in sintering temperature in the experimental range, the nitriding effect of the samples was good, and density and strength of the material had improved, especially at 1500 °C. Compared with the MM samples, the JM samples had a better effect on the nitriding, density, strength of the material, and the volume stability of the sintering process.

3.2.2 Microstructure of samples at different temperatures

At 1300 °C, as may be seen from Figs. 3 and 4, the aggregated large particles are formed by the reaction of small grains in the matrix. The small grain size of the matrix is mostly 300–600 nm, the surface of those grains is rough, and their shape is irregular. The EDS analysis shows that there are small grains of nitrogen (chemical composition in amount fraction: Al 64.76 %, O 21.21 %, N 10.09 %, Mg 3.94 % or Al 48.06 %, O 44.10 %, N 5.23 %, Mg 2.62 %), but there are also small grains that still have no nitrogen. Therefore, it may be concluded that there are some parts of the aluminum in the sample that cannot completely nitride under the temperature conditions. Since the nitride product crystal size is smaller, it is easy to build clusters and a more complicated microstructure of materials.

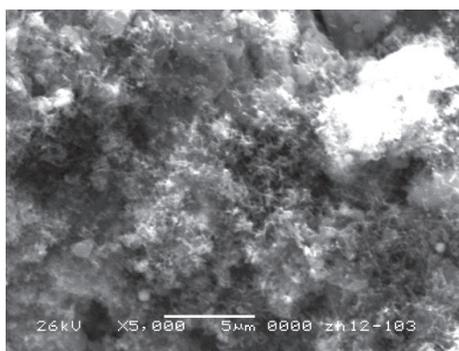


Fig. 3 – Plate-like crystals sintered at 1300 °C (MM3)
Slika 3 – Pločasti kristali sinterirani na 1300 °C (MM3)

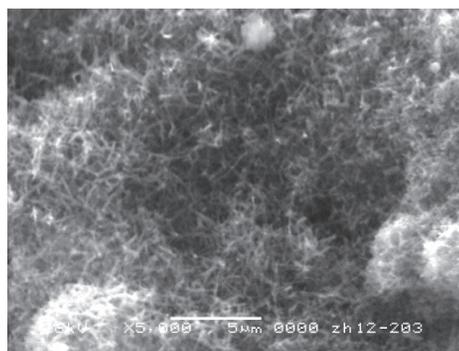


Fig. 4 – Plate-like crystals sintered at 1300 °C (JM3)
Slika 4 – Pločasti kristali sinterirani na 1300 °C (JM3)

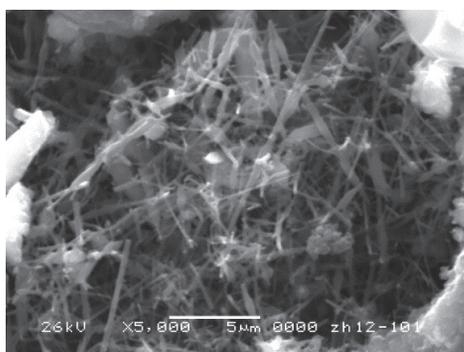


Fig. 5 – Needle-like crystals sintered at 1400 °C (MM3)
Slika 5 – Igličasti kristali sinterirani na 1400 °C (MM3)

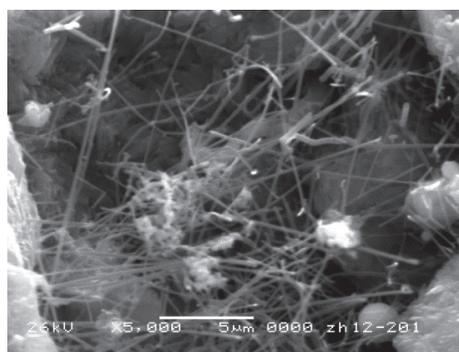


Fig. 6 – Needle-like crystals sintered at 1400 °C (JM3)
Slika 6 – Igličasti kristali sinterirani na 1400 °C (JM3)

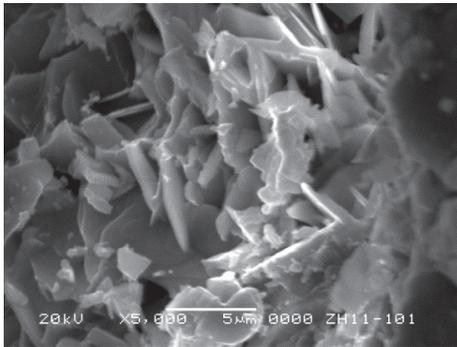


Fig. 7 – SEM photograph of MM3 sintered at 1500 °C
Slika 7 – SEM-fotografija uzorka MM3 sinteriranog pri 1500 °C

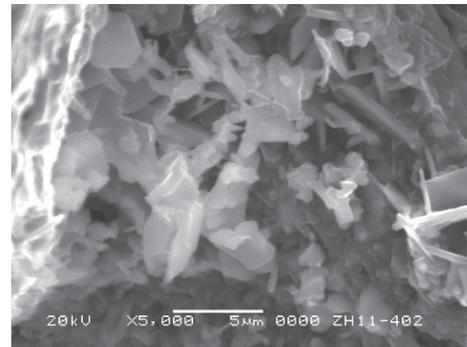


Fig. 8 – SEM photograph of JM3 sintered at 1500 °C
Slika 8 – SEM-fotografija uzorka JM3 sinteriranog pri 1500 °C

At 1400 °C, as may be seen from Figs. 5 and 6, the microstructure of the fracture surface shows that the small grains formed by the reaction in the matrix closely surround the aggregate. Energy spectrum analysis data shows that the samples' nitriding degree is high, the metal aluminum basically is completely nitrided, particles are slightly larger, but still have a large number of submicron particles, and the size is more uniform.

As may be seen from Figs. 7 and 8, at 1500 °C, the aggregated large particles are also closely surrounded by the small grains formed in the matrix, there are many small pores around the grains, and many whiskers and flake crystals in the pores. The shape of completely formed crystals is octahedron or plate-like. Therefore, there are more gas phase reactions in the firing process. The size of small particles increased significantly, and is more uniform.⁸ The EDS analysis shows that the grain nitrogen content in the sample is higher, there is more nitride, and the trace element Si and Ti can be dissolved in the nitride, so the harmful effect is reduced.

The microstructure analysis of the above samples suggests that nitrided samples level is relatively low, grain size is small, crystal growth rate is low and easy to build clusters, and at the same time, there are many whiskers in the pores, and the structure is more complex at 1300 °C or at 1400 °C. After the firing of the sample at 1500 °C, the metal aluminum nitride is more complete, and the nitrogen content of the product is higher, the particle diameter is larger, the size of the particle is uniform, the development of grains in JM samples is relatively complete, the generated grain size is several microns, and there are a variety of crystal structures and crystals crossing with each other. Therefore, according to the results of microstructure analysis, the test results at 1500 °C are the best.

3.3 Microstructure and energy spectrum analysis of the sample after sintering

3.3.1 Bauxite-based MgO-MgAlON sample

Fig. 9 shows a fracture morphology of the specimen at 1500 °C, and Fig. 10 shows an internal crystalline structure sample. The results of spectrum analysis are shown in Table 5.

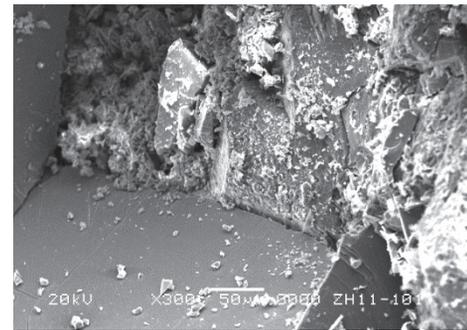


Fig. 9 – Micrograph of fracture surface of specimen
Slika 9 – Mikrografija površine loma uzorka

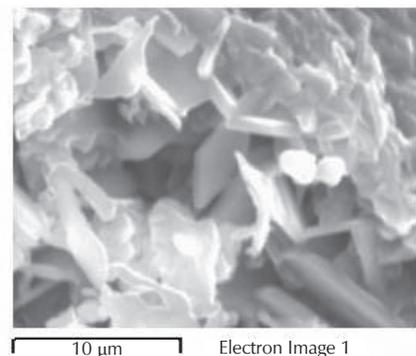


Fig. 10 – Plate-like crystals of specimen
Slika 10 – Pločasti kristali uzorka

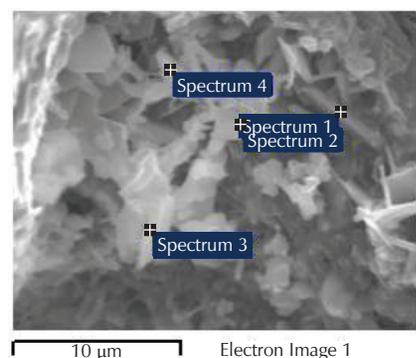


Fig. 11 – Micrograph of microcrystals of specimen
Slika 11 – Mikrografija mikrokristala uzorka

Table 5 – EDS analysis results in Fig. 11

Tablica 5 – Rezultati analize EDS-om prema slici 11

Location of analysis Mjesto analize	w / %			
	N	O	Mg	Al
Spectrum1	17.96	17.53	4.28	60.23
Spectrum2	18.63	26.74	4.46	50.18
Spectrum3	23.09	25.21	4.23	47.47
Spectrum4	26.91	24.84	3.43	44.82

Fig. 9 indicates that the magnesium oxide large particles are formed by the reaction of small grains in the matrix of uniform size,⁴ many small pores are generated during nitriding reaction, and there are many whiskers and flake crystals formed in the pores (Figs. 10 and 11), which indicates that there are more gas phase reactions in the firing process. The size of small particles is slightly larger than that of the alumina-based reaction sintered samples, and the particle size is more uniform. The EDS analysis in Table 5 shows that the nitrogen content of these small grains was between 17.96 % – 26.91 %, which was higher than that of the oxide-based materials (16.53 % – 17.69 %) in the literature.⁷ The reason perhaps being that the impurities in bauxite are beneficial to the rate increase in metal aluminum nitride, and promote grain growth and development, thus improving the nitrogen content. Therefore, compared with the oxide-based samples, the bauxite samples nitriding effects and sintering properties are better.

3.3.2 Bauxite-based $MgAl_2O_4$ -MgAlON sample

The fracture surface of SEM sample at 1500 °C is shown in Fig. 12, and the energy spectrum analysis is shown in Fig. 13 and Table 6.

Table 6 – EDS analysis results in Fig. 13

Tablica 6 – Rezultati analize EDS-om prema slici 13

Location of analysis Mjesto analize	w / %			
	N	O	Mg	Al
Spectrum2	22.44	15.02	15.88	46.66
Spectrum4	26.68	16.02	16.33	40.97

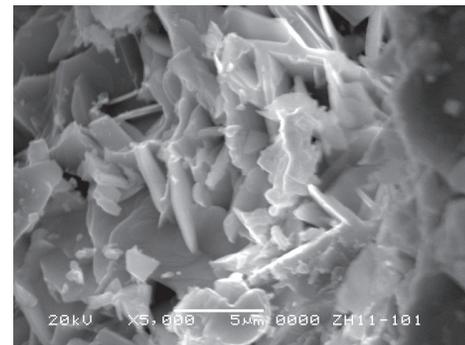
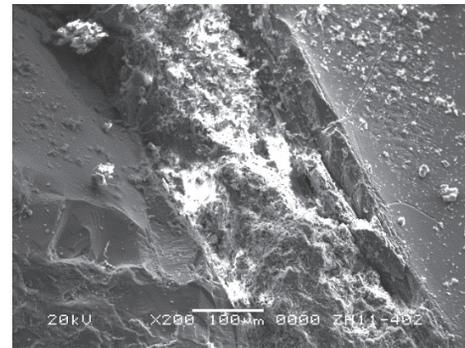


Fig. 12 – Micrograph of fracture surface of specimen
Slika 12 – Mikrografski snimak površine loma uzorka

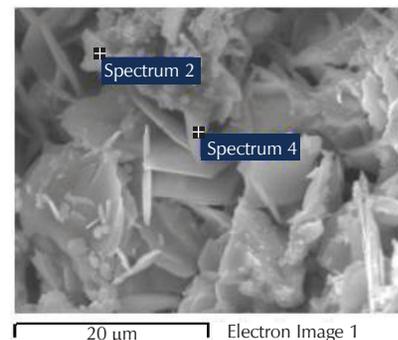


Fig. 13 – Plate-like crystals of specimen
Slika 13 – Pločasti kristali uzorka

Fig. 12 indicates that the grain size and binding state of the JM and MM samples are roughly the same. MA large particles in the sample were also closely surrounded by the reaction of the MgAlON particles in the matrix. The combination of the large particles and the matrix were denser, and the obvious dividing line could not be seen. The grain samples are a few microns in size. There is a variety of crystals cross combination structure favourable to improve the strength of the material (Fig. 13). The energy spectrum analysis data in Table 6 shows that the grain nitrogen content in the sample is higher; there is more nitride than in the bauxite-based MgO-MgAlON samples.

3.4 Performance of alumina-based MgAlON composites and bauxite-based MgAlON composites

3.4.1 Physical properties of alumina and alumina-based matrix composites

The test samples fired in a nitrogen atmosphere at 1500 °C, and the sintering performance of the sintered samples are shown in Table 7.

Table 7 – Physical properties of the specimens

Tablica 7 – Fizikalna svojstva uzoraka

Sample Uzorak	Weight gain / % Porast mase / %	Linear change / % Promjena duljine / %	Density / g cm ⁻³ Gustoća / g cm ⁻³	Apparent porosity / % Prividna poroznost / %	CMOR / MPa
AMM	1.82	1.82	2.66	23.91	10.12
BMM	0.32	1.98	2.53	28.40	7.76
AJM	4.10	0.96	2.74	22.25	19.37
BJM	1.60	1.23	2.69	23.29	13.54

Note: AMM represents the alumina-based MgAlON combined MgO, BMM represents bauxite-based MgAlON combined MgO, AJM represents alumina-based MgAlON binding MgAl₂O₄, BJM represents bauxite-based MgAlON binding MgAl₂O₄.

Table 7 indicates that compared with the oxide-based composite material, bauxite-based composites have a larger porosity rate, smaller volume density, and lower strength at room temperature. This may be due to the presence of impurities in the bauxite. Trace impurities can be beneficial for the reduction and nitridation of the bauxite-based MgAlON composites.

3.4.2 High temperature mechanical properties of alumina-based samples and bauxite-based samples

The mechanical properties of bauxite-based composites at high temperature were tested using the same method with alumina-based samples, and the high temperature mechanical properties of the two samples were compared. The results are shown in Fig. 14.

As may be seen from Fig. 14, the variation curve of MOR-T bauxite samples and alumina-based samples are consistent. There is a peak strength at a certain temperature, and then, as temperature increases, the strength starts to decline. Compared with the same aggregate, under the same temperature, the high temperature alumina specimen flexural strength is slightly higher than that of the bauxite-based samples. However, since the bauxite-based sample nitridation sintering structure is not compact enough, secondary sintering phenomenon arises as the flexural strength of test specimens at high temperature, thus the bauxite samples high temperature strength increase rate is greater.⁹ BMM sample strength increases slowly before 1000 °C, and

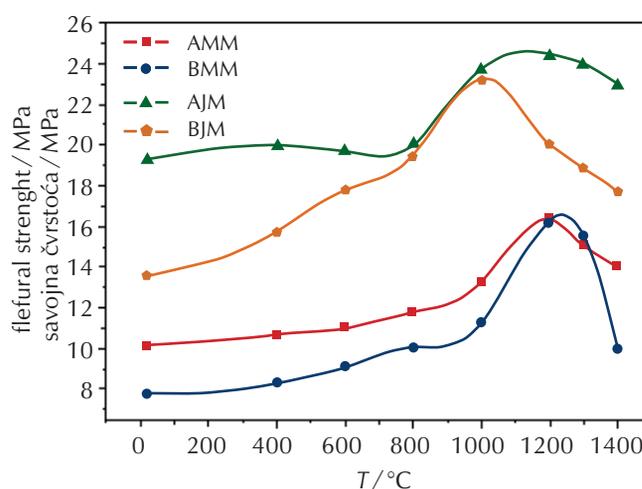


Fig. 14 – MOR-T curves of the alumina-based and bauxite-based specimens

Slika 14 – Savojna čvrstoća uzoraka na bazi aluminijske oksida i boksita

when the temperature is higher than 1000 °C, the strength increases rapidly. The maximum value of the rapid strength increase is at 1200 °C, and the average strength increases about 150 % compared to room temperature. The strength of the maximum temperature point at 1400 °C is less than that at 1200 °C, but it is still 30 % higher than at room temperature. The strength of BJM sample increases before 800 °C, the maximum strength of the sample is at 1000 °C, and the strength is increases by approximately 75 % compared to room temperature.

The composition, structure, and high temperature mechanical properties of the sintered materials of Al₂O₃-SiO₂-ZrO₂ system were studied by academician X. C. Zhong.¹⁰⁻¹¹ It was concluded that the mechanical behaviour of the material at different temperatures could be divided into three stages. The three stages represent the typical characteristics of the high temperature mechanical behaviour of the refractory materials containing liquid phase sintering: 1. Elastic deformation stage: the stress-strain curve is a straight line. Under long-term stress, the material does not produce creep, high-strength, and brittle fracture. The fracture surface of the sample is compact and smooth, which belongs to a transgranular fracture mode. 2. Plastic deformation stage: stress-strain curves deviate from a straight line. Under long-term stress, material creep phenomenon is significant, strength begins to decline, the fracture is a quasi-ductile, and the fracture is divided into smooth and rough areas, which is a mixed type of intergranular fracture. 3. Viscous flow stage: the stress-strain curve is significant and the amount of permanent deformation is large. Under the action of stress, the creep rate increases sharply, the strength becomes minimal, the fracture is a ductile, and the fracture surface is rough, which belongs to the intergranular fracture. There are two types of fracture strength with temperature changes: type I corresponds to the polycrystalline material, which is characterized by a peak value at a certain temperature. For the materials such as corundum mullite, the peak value of the strength curve is 800 °C. Type II generally corresponds to

the single crystal material, which characteristic is that the strength remains unchanged at low temperature range. When the temperature is higher than the transition temperature, the strength gradually decreases. Therefore, pure corundum material strength began to decline after 800 °C. The reasons for the emergence of two kinds of curves are residual internal stress in the samples and microcrack formation and elimination, high temperature grain boundary non-crystalline phase and glass phase softening.

From the experimental results, the two series of MOR-T curves belong to the type I, proposed by academician X. C. Zhong. This is because the MgAlON bonded magnesia composite materials are polycrystalline materials, and the phase equilibrium and the stress distribution of the material in the process of long-time thermal insulation are more balanced. During the cooling process, the thermal expansion coefficient of each phase is of larger difference between the material and the existence of residual stress, and even cracks have an impact on the strength of the material.

Fig. 14 indicates that at the same temperature, the strength of the BMM sample was lower than that of the BJM sample. The lower strength of the sample was 6–8 MPa, and the maximum strength temperature of BMM was 1200 °C, while the maximum strength of BJM was 1000 °C. There are two reasons for this: 1) The large porosity of BMM samples exists, which causes low sintering performance and low strength; 2) The reaction of MgAlON transparent crystal phase is different, and the impurity phase exists. During the cooling process, the thermal expansion coefficient of each phase is greatly different, which causes the residual stresses and cracks in the material, so the strength of the material is relatively low, but the temperature of the microcrack bridging improves.

4 Conclusion

(1) With the increase in MgAlON content, the volume density of the sample decreases, the apparent porosity, length change, and weight gain increase gradually. The room temperature bending strength has an upward trend. JM samples nitriding effect is better, they also have high density and strength.

(2) With the increase in sintering temperature, the volume density of the samples decreases slightly, the apparent porosity and the bending strength increases gradually with the temperature.

(3) Bauxite-based sample sintering properties and mechanical properties at room temperature were lower than those of the alumina-based composites. However, the difference is small, so it can replace the use of alumina-based composite materials, in order to save production costs.

(4) The MgO–MgAl₂O₄–MgAlON system MOR-T curves of samples belongs to type I curve. Strength at high temperature (≤ 1400 °C) was higher than the strength at room temperature; intensity peak temperature of the material is 1200 °C.

(5) Using the same kind of aggregate sample, the high temperature flexural strength of the alumina-based sample was slightly higher than that of the bauxite-based sample at the same temperature.

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List of abbreviations and symbols Popis kratica i simbola

CMOR	– compressive modulus of rupture at room temperature – tlačna čvrstoća na sobnoj temperaturi
MA	– magnesium aluminium oxide, spinel, MgAl ₂ O ₄ – magnezijev aluminijev oksid, spinel, MgAl ₂ O ₄
MOR-T	– modulus of rupture with temperature – savojna čvrstoća ovisna o temperaturi
w	– mass fraction, % – maseni udjel, %

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SAŽETAK

Priprema i mehanička svojstva mješavina MgAlON

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Da bi se smanjili troškovi proizvodnje i iskoristili izvori sirovina, pripremljeni su kompoziti MgAlON u atmosferi dušika od kalciniranog aluminijskog oksida. Proučen je utjecaj sastava i temperature sinteriranja na kompozitni materijal. S količinom MgAlON povećavaju se prividna poroznost, linearna ekspanzija i porast mase. Smanjuje se gustoća materijala, ali raste savojna čvrstoća. S temperaturom sinteriranja pada gustoća, a rastu prividna poroznost i savojna čvrstoća. Najbolji rezultati postignuti su sinteriranjem pri 1500 °C. Kada je kao sirovina upotrijebljen aluminijski oksid, kompozit je imao veću gustoću i pokazivao pozitivniji učinak nitrida od kompozita baziranog na boksitu.

Ključne riječi

Kompoziti MgAlON, sinteriranje, MgAlON pripremljen od boksita

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