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Wettability of the System of Cast Iron and Magnesia Ceramics with Graphite

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Abstract

In this paper examinations of high-temperature wetting tests of 3 systems of liquid alloy – cast iron in contact with ceramic materials: magnesia ceramics in combination with natural graphite were presented. After wettability testing, the microscopic observations of the morphology of the sample surface and the cross-section microstructure with the chemical composition in micro-areas were examined. One of the objective of this work was also to verify whether the graphite content would affect the wettability of the magnesia ceramics.
The study of high-temperature wetting kinetics of the liquid alloy in contact with the ceramic material, by the "sessile drop" method with capillary purification (CP) procedure was conducted. Under the test conditions, at a temperature of 1450°C and time 15 minutes, all 3 experimental systems showed a non-wetting behaviour. The average contact angle for the system with cast iron drop on magnesia ceramics was 140°, on magnesia ceramics with 10 parts per weight of graphite was 137° and on magnesia ceramics with 30 parts per weight of graphite - 139°.
Microscopic observations revealed that in the case of the sample consisting of the cast iron drop on the substrate with magnesia ceramics, the formation of fine separations was not observed, unlike the systems with the substrate with magnesia ceramics and the addition of natural graphite. Numerous, fine droplets accumulate on the graphite flakes and consist mainly of Si as well as Fe and O. On the other hand, the rough MgO grains have a gray, matt surface, without fine separations. The conducted observations indicate the mechanical nature of the bonding - liquid metal penetrates into the pores of the rough ceramics of the substrate. However, in the case of systems of cast iron drop with magnesia ceramics and addition of graphite, probably the adhesive connection and the physical attraction of elements derived from cast iron drop with the flake graphite appeared as well.

Keywords: Composites, Metal-ceramic bonding, Wettability, Sessile drop method, Cast iron

1. Introduction

The wetting phenomenon occurs when two materials: one in the liquid state and the other in the solid state, come into contact and is described as the ability of a liquid to maintain contact with a solid surface. In order to determine the wettability of such systems the measurement of the contact angle θ - formed by the tangent to the surface of the measuring drop deposited on the surface of a solid body (at the point of contact of the three phases: solid, liquid and gas) - is used [1].

There are several parameters, which affect the contact angle. The most important are [2, 3]: the surface structure (roughness) and the composition of the solid, the melted metal composition and its purity, the kind of gaseous environment (high vacuum, inert gas or oxygen atmosphere), the temperature and time of contact.

The non-wettability of the ceramic material by liquid metal is desirable in refractory materials dedicated to high temperatures processes in the metallurgical, cement and energy industries. However, there are applications like metal-ceramic composites that demand good metal-ceramic bonding and good wetting (θ‹‹90°) [1]. The process of joining dissimilar materials like metal and ceramic is complex and often difficult to obtain. Almost all non-metal/metal interfaces have poor wettability [2].

The durability of the ceramic-metal connection is determined by the structure and strength of the interface. The article [4] describes 3 types of such a connection:

- mechanical, based on the mutual anchoring of components,
- adhesive, based on the formation of bonds between atoms or molecules of adjacent surfaces,
- diffusion, involving the mutual diffusion of atoms from individual components (often associated with the formation of new phases).

The method of bonding liquid metal/solid ceramic system can be combined with the processes of mechanical bonding, dissolving, wetting, diffusion and exchange reactions [5].

Wettability in high-temperature solid-liquid systems is supported by such interactions as dissociation of surface oxides on the liquid metal, chemical dissolution of the solid in the melt, adsorption of reactive solutes and formation of an interfacial compound [6]. Metallic, ionic or covalent bonds are formed and the most desirable are interfacial metallic bonds, which provide more ductile high-strength composites [2]. The ceramic material for metal matrix composites is designed in such a way to obtain the appropriate pore structure and also to form new phases at the interface by the chemical reaction [4]. The quality of the metal-ceramics bonding is influenced by both the mechanical joining of components and the chemical interaction, as well as the imperfections of the phase boundary, such as impurities, cracks, and voids [2].

The most serious problem in metal-ceramic composite preparation is overcoming the wettability barrier. The wettability can be improved by increasing solid surface energy and decreasing the surface tension of a liquid and solid-liquid interfacial energy [5]. In [2, 5, 7] the following solutions are presented:

- coating of ceramic particles, which create an intermediate layer to the wetting process (mainly Ni and sometimes Fe, Ti, Cu, Ag and Cr are used for coatings);
- surface metallization technique leads to increase ceramics solid surface energy,
- adding some special elements, like lithium or magnesium, to the melt to reduce the liquid metal surface tension,
- alloying with reactive elements such as Ti, Si, Mg, Cr, Ca, Sr, Mn, Ce, Cu, Zr to improve the affinity between non-metal and metal matrix,
- „in situ" synthesis of composites,
- annealing of the ceramic material before its introducing to liquid metal, which reduces the metal surface energy and the contact angle.

Metal matrix composites have been developed mainly in the area of non-ferrous metal alloys. There are still few publications devoted to cast iron composites [1, 8].

The aim of this paper is to present examinations of high-temperature wetting kinetics of the liquid alloy – cast iron in contact with the ceramic material – magnesia ceramics in combination with natural graphite, with the "sessile drop" method.

The tests described in this article are focused on nodular cast iron, which is obtained by metallurgical treatment with the use of, inter alia, the magnesium compound. As the literature above indicates, Mg is the reactive element that could reduce the liquid metal surface tension. In addition, the experiment was made for a combination of ceramic materials, that wasn't tested before. One of the objective of this work was also to verify whether the graphite content would affect the wettability of the magnesia ceramic.

2. Research Methodology

The sample of nodular cast iron with pearlitic-ferritic matrix (Fig.1) was applied in the experiment. The chemical composition of this alloy was (% wt.): C - 3.74 %, Si - 2.40 %, Mn - 0.236 %, Ni - 0.125 %, Cu - 0.109 %, Cr - 0.051 %, Mg - 0.029 %, P - 0.029 %, S - 0.012 %, by atomic emission spectrometry and high-temperature combustion with IR detection.

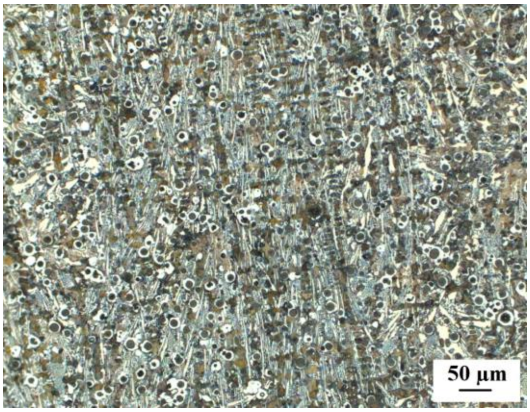


Fig. 1. Microstructure of cast iron used in the test, metallographic specimen etched in the Mi1Fe reagent (4% Nital), LM, microscope magnification 200x

The following ceramic materials were used for the tests:

1. Magnesia Ceramics - the sintered magnesia obtained as a result of the firing of natural magnesite at temperatures above 1700°C. The main mineral is periclase – MgO in an amount 94.8% and Fe_2O_3 - 2.2%, SiO_2 - 1.4%, MnO - 0.9%, CaO - 0.4%, Al_2O_3 - 0.3% by XRF chemical analysis (wt. %). The particle size (median) determined by laser diffraction for this material is d50 = 80 μm.
2. Natural Flake Graphite with the particle size (median) determined by laser diffraction d50 = 75 μm.

Table 1.
Composition of mixtures used for ceramic substrate

	Magnesia Ceramics, parts per weight	Graphite, parts per weight	Binder, % of weight
Magnesia Ceramics	100	0	34
Magnesia Ceramics | 10 Graphite	90	10	50
Magnesia Ceramics | 30 Graphite	70	30	66

A ceramic substrate was prepared with a binding material (water glass R-145 diluted with water in a 50/50 volume ratio) in a silicone mold. Three types of mixtures were used in the experiment, which compositions are given in the Table 1.

Prepared pellets with a diameter of ~ 17 mm and a height of ~ 5 mm, after taking them out from the silicone mold, were dried at the temperature of 100°C for 2 hours, and then fired at 600°C for 1 hour. The surface of the substrate samples was not modified by grinding.

The study of high-temperature wetting kinetics of the liquid alloy in contact with the ceramic material, by the "sessile drop" method with capillary purification (CP) procedure, was conducted using the experimental set-up for investigations of high temperature and surface phenomena of liquid metals and alloys described in details in [9], located in the Łukasiewicz Research Network - Krakow Institute of Technology. As a substrate, magnesia ceramic was used also in combination with natural graphite, and as a drop - a cast iron sample.

The test was carried out initially in a high vacuum (up to 500°C), and next in an atmosphere of a protective gas – argon with the pressure range of 850-900 mbar. The heating of the substrate and the capillaries with the melt was carried out at a rate of 15°C/min. After reaching the temperature of 1450°C, a drop of liquid melt was squeezed out of the capillary and deposited on the substrate. The time of testing the system at this temperature was 15 minutes, then the sample was cooled at the rate of 20°C/min. The high temperature interaction between molten cast iron sample and ceramic substrates was continuously recorded with a high-speed high-resolution CCD camera. The Astra2 program was used to perform the contact angle calculations [10, 11]. The standard deviation of the contact angle measurement by sessile drop method is ± 2°. Test conditions: temperature and time were selected from previous observation [12] and regard to the real casting process.

After wettability testing, the analysis of the morphology of the sample surface with the chemical composition in micro-areas was examined by scanning electron microscopy using TM3000 Hitachi device equipped with an energy-dispersive X-ray spectroscopy analyzer - Quantax 70. In the next stage, samples were cut perpendicularly from the center. The cross-section of ceramic substarates with the cast iron drop was analyzed using Zeiss AxioObserver Zm1 light microscope and the FEI Scios scanning electron microscope with the system EDAX Team for determination of the chemical composition by X-ray microanalysis.

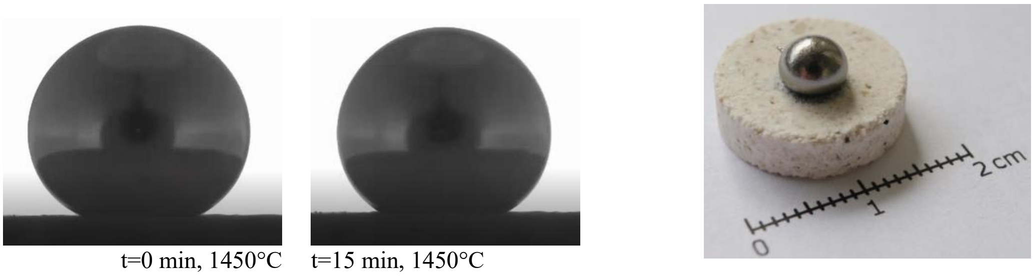


Fig. 2. Photos of the sample - a drop of liquid cast iron in contact with the Magnesia Ceramics substrate - during the test of high-temperature wetting kinetics; on the right a photo of the sample after the test

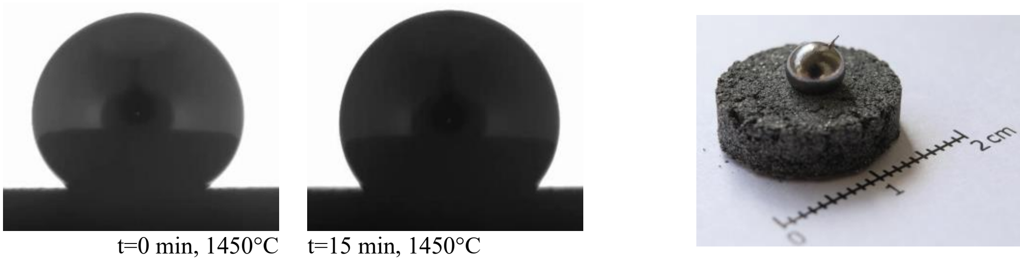
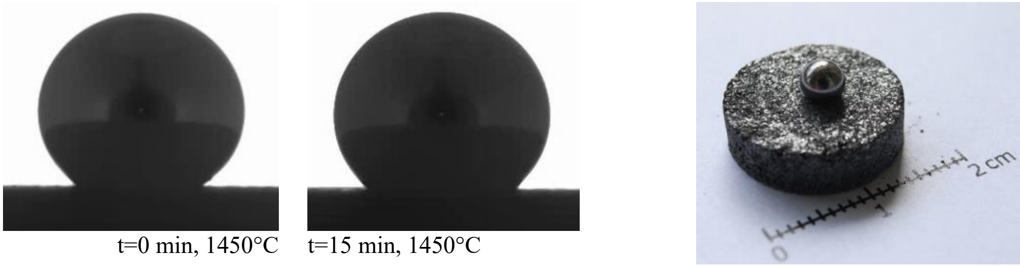


Fig. 3. Photos of the sample - a drop of liquid cast iron in contact with the Magnesia Ceramics | 10 Graphite substrate - during the test of high-temperature wetting kinetics; on the right a photo of the sample after the test



t=0 min, 1450°C t=15 min, 1450°C

Fig. 4. Photos of the sample - a drop of liquid cast iron in contact with the Magnesia Ceramics | 30 Graphite substrate - during the test of high-temperature wetting kinetics; on the right a photo of the sample after the test

3. Results of examinations

3.1. The wettability testing for systems of cast iron and ceramic substrates

Figures 2-4 show pictures of samples during the high-temperature wetting testing, at the moment of the start and the end of the experiment, recorded with a CCD camera and photos of tested systems: cast iron – ceramic substrate after testing.

Figures 5–7 show the wettability kinetics, under test conditions, for systems of cast iron and tested substrates: magnesia ceramics and magnesia ceramics with flake graphite in different contents. Table 2 shows the results of the contact angle for tested systems taking into account the left and right angle at the beginning (t=0 min) and at the end (t=15 min) of the experiment. The average contact angle for the individual system was calculated from the mean of the average of the left angle and the average of the right angle.

In Fig. 2 dark marks on the light surface of the ceramic substrate, under a drop of cast iron, are visible. In Figs. 2 and 3 and further also in Figs. 8 and 9, characteristic needles or smaller droplets „daughter" coming from the surface of the cast iron drop „mother" is observed. This phenomenon has been described in detail in the literature [1, 12] and is related to the solidification expansion of the cast iron and the difference in coefficient of thermal expansion between the metal drop and the ceramic substrate.

For the system consisting of a drop of liquid cast iron on magnesia ceramic substrate, at the beginning of the test, the contact angle (left and right) was 144°, and at the end of the test, after 15 minutes, it decreased to 136° (left angle) and 133° (right angle). A slight decrease in the contact angle was observed from 0 to 15 min by 8 to 11°. The average contact angle for this system was 140°. Similar values of the measured contact angle between the sintered MgO substrate and the liquid iron (approximately 134°) were obtained in the publication [13].

A similar characteristic of the wetting kinetic is presented by the system consisting of a cast iron sample on a magnesia ceramics substrate and with the addition of flake graphite in the amount of 10 parts per weight. Initially, the contact angle was 142° (left angle) and 144° (right angle), and after 15 min it was 131° (left angle) and 132° (right angle), respectively. Here, a minor decrease in the contact angle was observed as well, from 0 to 15 min of about 11-12°. The average contact angle for this system was 137°.

The wetting kinetic for the cast iron with magnesia ceramics and 30 parts per weight addition of graphite had a slightly different course. The initial contact angle was 136° for the left angle and 141° for the right angle. After 15 minutes of the test, the wetting angle remained at 134° (left angle) and 138° (right angle). The minor standard deviation shows that these values remained at a similar level during the test. The average value of the contact angle was 139°.

The differences between the left and right angle at the same time points of the test could be affected by the influence of surface quality of ceramic substrates. The smoothness / roughness of the surface of tested substrates was obtained from the silicone mold and the graining of the ceramic powder. Ceramic substrates were neither ground nor polished, to avoid disturbing the bonding of the matrix grains.

In all three cases, these are non-wettable systems (θ>>90°), under the test conditions, at a temperature of 1450°C and time 15 minutes. The addition of graphite to magnesia ceramics did not improve the wettability in contact with liquid cast iron.

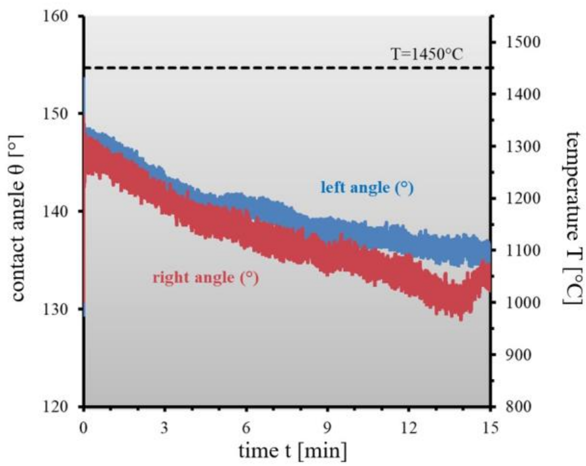


Fig. 5. Wettability kinetics for molten cast iron on Magnesia Ceramics (Al_2O_3 support) substrate under test conditions; CP/15 min/1450°C

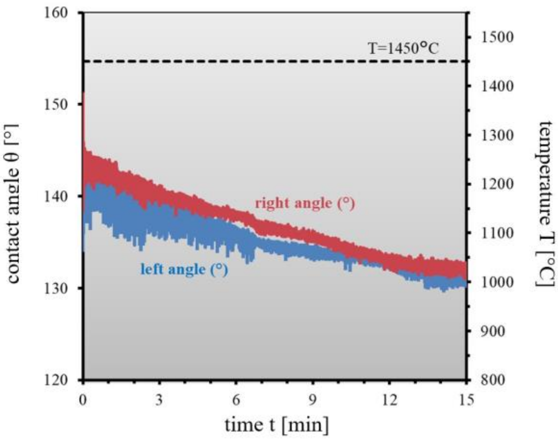


Fig. 6. Wettability kinetics for molten cast iron on Magnesia Ceramics | 10 Graphite (Al_2O_3 support) substrate under test conditions; CP/15 min/1450°C

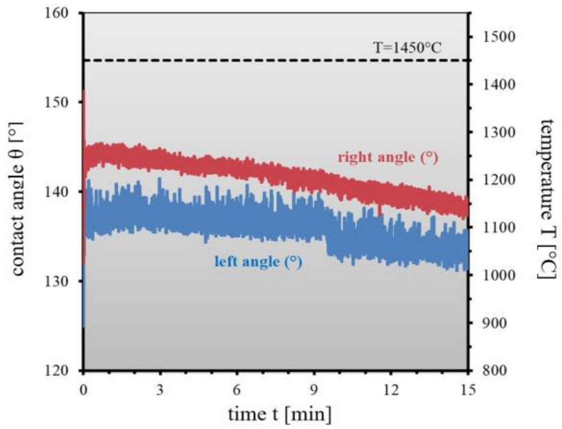


Fig. 7. Wettability kinetics for molten cast iron on Magnesia Ceramics | 30 Graphite (Al_2O_3 support) substrate under test conditions; CP/15 min/1450°C

Table 2.
List of results of the contact angle for individual systems under test conditions

	Contact angle θ [°]					
	Left angle, 0 min	Left angle, 15 min	Average left angle, 0-15 min (SD)	Right angle, 0 min	Right angle, 15 min	Average right angle, 0-15 min (SD)
Cast iron – Magnesia Ceramics	144	136	141 (4.1)	144	133	139 (5.0)
	Average contact angle θ = 140° (SD=4,5)					
Cast iron – Magnesia Ceramics | 10 Graphite	142	131	136 (3.7)	144	132	138 (3.9)
	Average contact angle θ = 137° (SD=3,8)					
Cast iron – Magnesia Ceramics | 30 Graphite	136	134	136 (1.7)	141	138	142 (1.9)
	Average contact angle θ = 139° (SD=1.8)					

Average contact angle = (average left angle, 0-15 min + average right angle, 0-15 min) / 2; SD – standard deviation

3.2. Microscopic observations of the samples after the wetting test

Microscopic observations were made on the samples after the wetting test. The aim of these studies was to analyse the morphology of the metal drop and the ceramic material and to identify changes, with particular emphasis on the contact area of these materials. The chemical composition in micro-areas on the surface of the metal drop on the ceramic substrate and on the cross-section area was investigated. The analysis of the possible cause of a slight decrease in the contact angle observed from 0 to 15 min on wettability kinetics was carried out.

In Figs. 8-9 the results of microscopic observations for the sample of the cast iron drop on the substrate based on magnesia ceramics were presented. Fig. 8 shows the surface of the solidified sample after the wetting test.

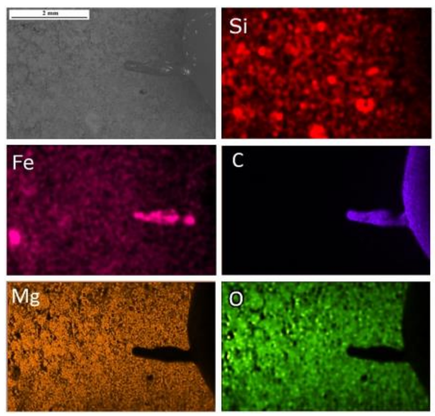


Fig. 8. Sample of cast iron drop on Magnesia Ceramics substrate after wettability test, SEM with EDS analysis in micro-area

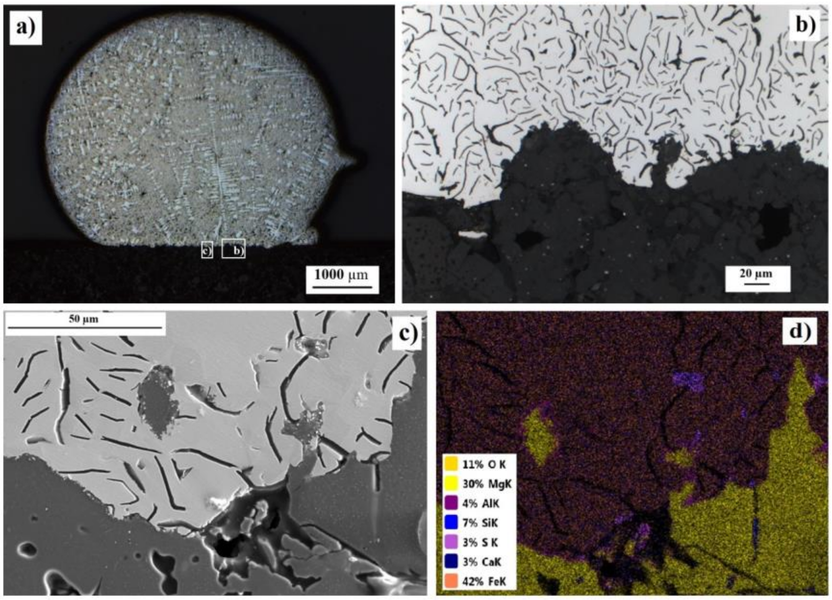


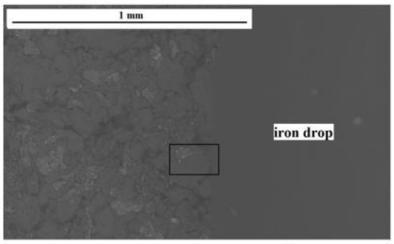
Fig. 9. Cross-sections of the sample of cast iron drop on Magnesia Ceramics substrate after wettability test; a) and b) LM, non-etched, microscope magnification 20 and 500x; c) and d) SEM with EDS microanalysis in the area of contact between metal and ceramics

The tested surface of the drop is covered with large black graphite blots. Additionally small „daughter" droplet, composed mainly of C and Fe, is presented on the surface. EDS analysis in the investigated micro-area confirms, that the metal drop is mainly composed of Fe, C and Si and the presence of Mg and O in the chemical composition of the substrate, as well as the presence of Si and Fe.

Fig. 9 presents the cross-section of the sample with particular emphasis on the contact area of the metal and ceramics. In the iron drop microstructure, there is flake graphite, due to the fading of the spheroidization effect, as a result of heating the sample to the temperature of 1450°C during the wetting tests. The conducted observations indicate the mechanical nature of the bonding - liquid metal penetrates into the pores of the rough ceramics of the substrate. The formation of new reaction products between liquid cast iron and magnesia ceramics was not observed.

In Figs. 10-11 the results of microscopic observations for the sample of the cast iron drop on the substrate based on magnesia ceramics with the addition of 10 parts per weight of flake graphite were presented.

For this sample also, flake graphite is present in the drop microstructure, and liquid cast iron penetrates into the pores of the ceramic material. On the surface of the ceramic substrate, apart from the main elements, i.e. Mg and O, could be observed the appearance of places, points enriched with elements such as Fe and Si (Fig. 10, points 1, 2 and 4).



1		2		3		4	
Element	Weight %	Element	Weight %	Element	Weight %	Element	Weight %
Fe	70.7	Fe	66.1	Mg	53.3	Si	53.4
Mg	12.9	Si	20.1	O	42.6	Fe	28.4
Si	7.1	Mg	11.0	Si	1.8	O	8.9
O	5.5	O	1.8	Fe	1.3	Mg	8.7
Ni	1.4	Cr	0.4	Mn	0.5	Cu	0.4
Cu	1.3	Ni	0.3	Cu	0.2	Cr	0.2
P	0.5	Cu	0.2	S	0.2		
Cr	0.3	P	0.1	P	0.1		
S	0.2						

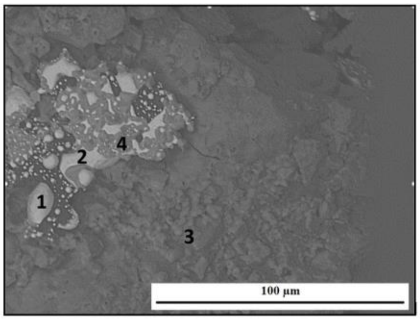
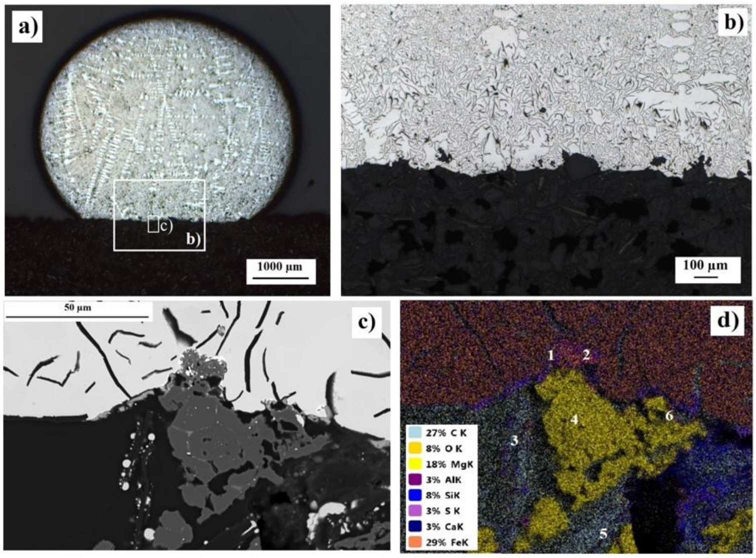


Fig. 10. Sample of cast iron drop on Magnesia Ceramics | 10 Graphite substrate after wettability test, SEM with EDS analysis in indicated micro-area



1		2		3		4		5		6	
Element	Weight %	Element	Weight %	Element	Weight %	Element	Weight %	Element	Weight %	Element	Weight %
Al	37.9	Fe	39.4	C	88.2	Mg	51.6	C	98.9	Fe	82.5
C	35.2	C	20.4	Fe	10.6	O	34.1	Si	1.1	S	6.9
Mg	17.2	Ca	15.2	Si	1.2	Ca	7.7			Ca	3.4
Ca	5.0	S	11.6			Al	4.7			Si	2.7
S	4.7	O	5.1			Si	1.9			Mg	2.4
		Al	4.6							Al	2.1
		Mg	2.5								
		Si	1.2								

Fig. 11. Cross-sections of the sample of cast iron drop on Magnesia Ceramics | 10 Graphite substrate after wettability test; a) and b) LM, non-etched, microscope magnification 20 and 100x; c) and d) SEM with EDS microanalysis in area of contact between metal and ceramics

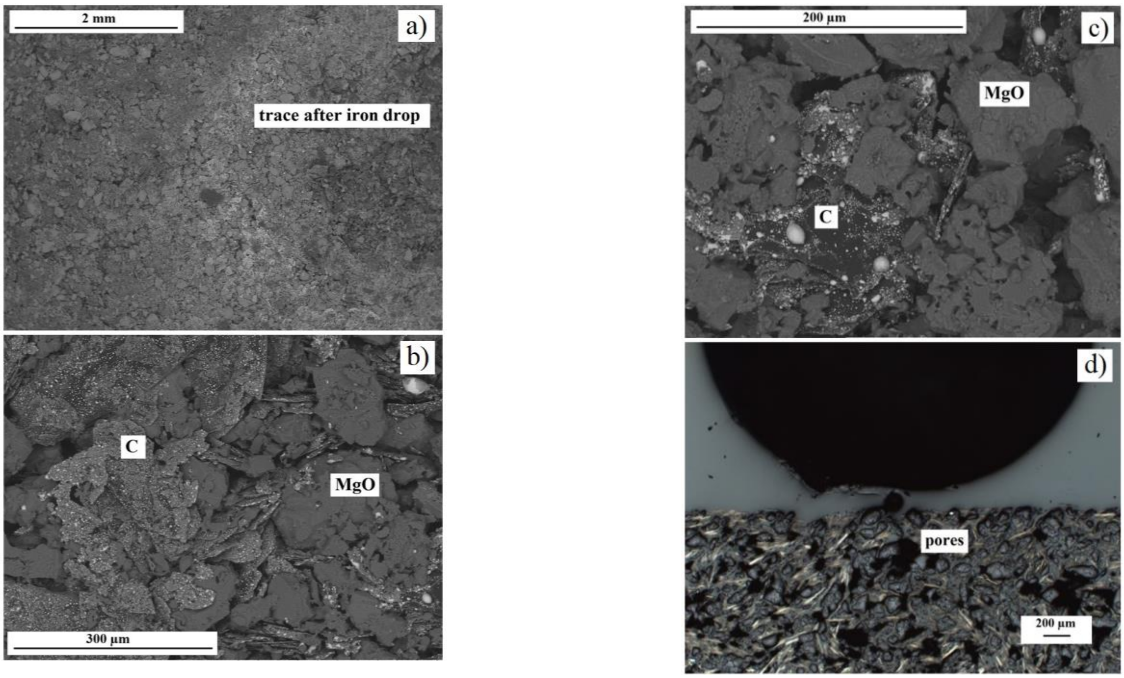
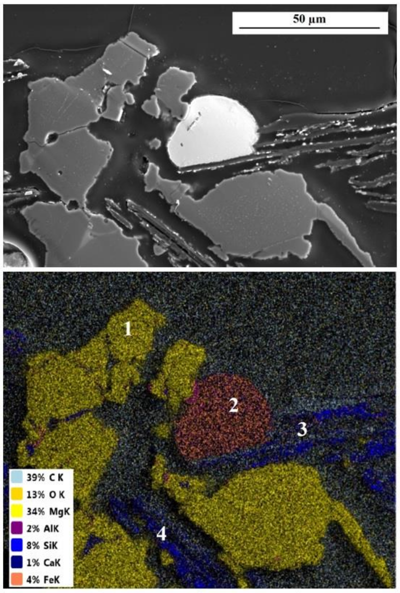


Fig. 12. Sample of Magnesia Ceramics | 30 Graphite substrate, SEM (a-c) and cross-section of cast iron drop with Magnesia Ceramics | 30 Graphite substrate, LM, non-etched, microscope magnification 50x (d) after wettability test

Microscopic observations of this sample after the cutting perpendicularly from the center, in the contact area of the metal and ceramics, indicate the presence of products composed of various elements such as Al, C, Mg, Ca, S, Fe, O, Si in the investigated micro-area. It is worth paying attention to the EDS map (Fig.11d), where the presence of Si, which appears in the structure of the ceramic material (marked in blue colour), is accumulated on the graphite flakes. The point analysis confirms this relationship between carbon and silicon as well as iron (Fig. 11, points 3 and 5).

In Figs. 12-13 the results of microscopic observations for the sample of the cast iron drop on the substrate based on magnesia ceramics with the addition of 30 part per weight of flake graphite were presented. After the wetting test, the drop separated from the surface of the substrate, leaving a circular trail with fine separations/droplets on the surface of the ceramic material (Fig. 12a). At higher magnification (Fig. 12b,c) irregular, matt grains of porous MgO magnesia ceramics are observed with a „clean" surface - without fine droplets. However, these tiny separations have accumulated on the surface of the graphite flakes. Additionally, the EDS analysis in Fig. 13. confirms this dependence; in points 3 and 4 (blue marked) apart from the main element C, the presence of Si and O was confirmed. Fig. 12d shows the structure of the ceramic substrate: MgO grains with graphite flakes and numerous voids, and pores.



1		2		3		4	
Element	Weight %	Element	Weight %	Element	Weight %	Element	Weight %
Mg	66.5	Fe	94.2	C	99.4	C	88.6
O	33.5	Si	5.8	Si	0.6	O	7.4
						Si	3.9

Fig. 13. Cross-section of cast iron drop with Magnesia Ceramics | 30 Graphite substrate after wettability test, SEM with EDS microanalysis in area of contact between metal and ceramics

In the case of the sample consisting of the cast iron drop on the substrate with magnesia ceramics, the formation of fine droplets was not observed, as opposed to the systems with the substrate with the addition of natural graphite. Numerous, fine separations accumulate on the graphite flakes and consist mainly of Si as well as Fe and O. On the other hand, the rough MgO grains have a gray, matt surface, without fine droplets.

4. Conclusions

High-temperature wetting tests with microscopic observations of experimental systems: nodular cast iron in contact with magnesia ceramics and natural graphite, present the following conclusions:

1. Under the test conditions, at a temperature of 1450°C and a time 15 minutes, experimental systems show non-wetting behaviour. The average contact angle for the system with cast iron drop on magnesia ceramics was 140°, on magnesia ceramics with 10 parts per weight of graphite was 137° and on magnesia ceramics with 30 parts per weight of graphite - 139°.
2. The addition of graphite did not improve the wettability of tested systems.
3. The conducted observations indicate the mechanical nature of the bonding - liquid metal penetrates into the pores of the rough ceramics of the substrate.
4. 4. In the case of the sample consisting of the cast iron drop on the substrate with magnesia ceramics, the formation of fine separations was not observed, unlike the systems with the substrate with the addition of natural graphite. Numerous, fine droplets accumulated on the graphite flakes and consist mainly of Si as well as Fe and O. Probably the adhesive connection and the physical attraction of elements derived from cast iron drop with the flake graphite appeared. On the other hand, the rough MgO grains had a gray, matt surface, without fine droplets.
5. The differences between the left and right angles at the same time points of the test as well as the slight decrease of the contact angle with time could be affected by the influence of the surface quality of ceramic substrates.
6. In the iron drop microstructure after the wetting test, flake graphite was observed, although the initial microstructure of this material contained nodular graphite. The fading of the spheroidization effect occurred, as a result of heating the sample to the temperature of 1450°C during the wetting tests and disappearing the modification effect due to remelting. In addition, magnesium evaporates in this temperature and its final content is insufficient to form nodular graphite.

Acknowledgements

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