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ANALYSIS OF THE INTERFACE BETWEEN THE MATRIX AND THE REINFORCEMENT PHASE IN CERAMIC-REINFORCED MMCs

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Abstract: The objective of the present paper is to stady the one main problem in the production of MMC – the wettability of the reinforcement particles by the metal matrix. The investigated composites are "invitro" type, with unformed and unchanging reinforcement phase in the process of obtaining and they were obtained using a technology, based on the capillary effect, that is different from the conventional methods known so far. Ceramic particles SiO_2, Al_2O_3 and SiC were used as a reinforcement phase has a complex microrelief that contributes to the consolidation of the mechanical bond, which they form with the metal matrix. In the method used to obtain MMC the melt is forcibly infiltrated into the capillary spaces between the ceramic particles of the reinforcement phase, such as helps to overcome the surface tension of the melt and provides good wetting of the particles of the reinforcement phase. A metallographic analysis was performed to investigate the wetting of the reinforcement particles by the metal matrix. The obtained results indicate that the method of poduction of MMCs, based on the capillary effect, provides stable mechanical bonding between the matrix and the reinforsment phase, owing to forced infiltration of the melt into the capillary spaces, which helps to overcome the surface tension of the melt and provides good wetting of the reinforcement phase particles.

Key words: MMCs, capillary forming, ceramic reinforcement phase, wetting, mechanical bond

1. INTRODUCTION

Metal matrix composites (MMCs) reinforced with hard ceramic particles have received considerable interest because they can preopose relative ease of fabrication, low cost and better mechanical and wear properties. Composites reinforced with ceramic particles such as SiO_2, Al_2O_3 and SiC, are widely used to make tools, matrices, wear-resistant and corrosion resistant products.

Some researchers divide metal matrix composites (MMCs) systems into three classes. The first class includes composites obtained from non-reactive and mutually insoluble components; the second class involves certain solubility of the components without the occurrence of a chemical reaction; the third class comprises a system obtained from reacting components. Six types of bond underlie this classification: mechanical bonding; wetting and dissolving bonding; reaction bonding; exchange-reaction bonding; oxidation bonding; mixed bonding [1, 2]. Mechanical bonding, which is the object of the present study, occurs when the reinforcing element has a rough surface. The purely mechanical bonding suggests the absence of any causes for chemical interaction. It can be formed by mechanical adhesion or due to friction occurring between the reinforcing phase and the matrix. Particles such as SiO_2 and Al_2O_3 have a complex microrelief that contributes to the consolidation of the mechanical bond. The formation of a strong mechanical bond requires a close contact achieved through adhesion between the liquid matrix and the particles of the reinforcing phase. Initially, the properties of the MMCs and their retention in operation are determined by the adhesion interaction of the reinforcement phase with the matrix [1]. The homogeneity of composite materials plays a crucial role in shaping their ultimate properties that determine their potential application in the industry. To the group of the most important factors which create the interface properties belong the conditions of the manufacturing process such as temperature, pressure and atmosphere, also the quantity, shape, dimensions, and distribution homogeneity of the reinforcement phase in the melal matrix volume [3, 4]. The local stresses in the composites reach their maximum levels directly in the interface region, where microcracks are formed and the beginning of the destruction of the material occurs. Therefore, the surface of the ceramic particles (SiO_2, Al_2O_3 and SiC), used for the production of MMCs, needs to be thoroughly cleaned of contaminants prior to their contact with the liquid phase [1, 2]. In addition, in order to effect a physical contact on the entire surface of the reinforcing phase particles, it is necessary to take into account the surface tension, density and good wetting of the reinforcing phase by the melt. The surface tension is the basis of the processes that

take place at the interface between the liquid phase and the surface of the solid reinforcing phase.
Taking into account the above mentioned, the studied MMCs of the "invitro" type were obtained using a technology, based on the capillary effect, that is different from the conventional methods known so far. This method is based on the "capillary forming" method, which is patented [5]. The main point is that the MMCs are obtained as a result of the forced infiltration of the melt (matrix) in the capillary spaces between the elements of the reinforcing phase, by vacuuming the mold. In this way a very good adhesion is achieved, as the forced infiltration of the melt into the capillary spaces, helps to overcome the surface tension of the melt and provides good wetting of the particles of the reinforcement phase [6-8].

2. MATERIALS AND METHODS

The main objective of the present work is to analyze the interphase bonding in MMCs with ceramic reinforcing phase type "in vitro" (with unchanging in the process of building a composite reinforcing phase), obtained by an unconventional method based on the capillary effect.

2.1 Materials used for obtained MMCs

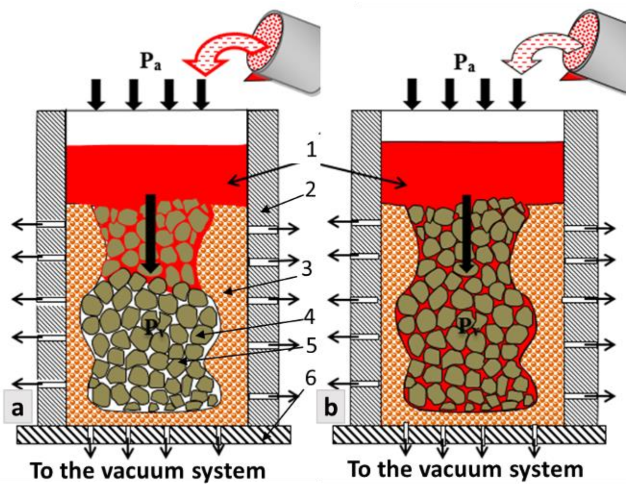
The materials and alloys employed for reinforcement phase and matrix and some of their primary characteristics are shown in Table 1.

Table 1. The employed materials and alloys

Materials		Main characteristics			
		density γ [g/sm^3]	hardness	mel-ting point [^{o}C]	size [µm]
Metal matrix material	Cu	8.86	35HB	1083	-
	aluminum alloy AlSi12	2.7	50-60 HB	577	-
	copper alloy CuZn38Pb2	8.5	70-80 HB	900	-
	AISI 304 stainless steel	7.9	201HB	1400	-
Rein-forcement phase material	quartz particles SiO_2	2.32	7 Mohs scale	1713	100-150
	quartz particles SiO_2	2.32	7 Mohs scale	1713	150-250
	corundum Al_2O_3	4.05	9 Mohs scale	2044	50-100
	corundum Al_2O_3	4.05	9 Mohs scale	2044	250-350
	Carborundum SiC	3.21	9 Mohs scale	2830	180

2.2 Essence of the method based on the capillary effect

One of the advantages of the method is that the production of single blanks is economically viable, since implementing the conventional methods of foundry mould production (ceramic, or with gypsum and cement in their composition). In the specific case, a moulding mixture of quartz sand and cement was used to make the moulds. Also according to the classical method of 'in vitro' MMCs, a mechanism is applied for the forceful introduction of the reinforcement phase in the prepared melt followed by homogenizing of the composite structure. In this case the process of mixture of the melted matrix and the reinforcement phase takes place following the principle of 'Capillary forming'. First, the reinforcement phase (ceramic particles) is put in the mould, then the metal matrix in melt form is infiltrated forcefully in the space among the ceramic particles by vacuum (Figure 1) [7, 8].



1- melt (matrix); 2- a perforated mouldingflask; 3- a mould; 4- reinforcement phase particles; 5- the capillary spaces; 6- a basic plate

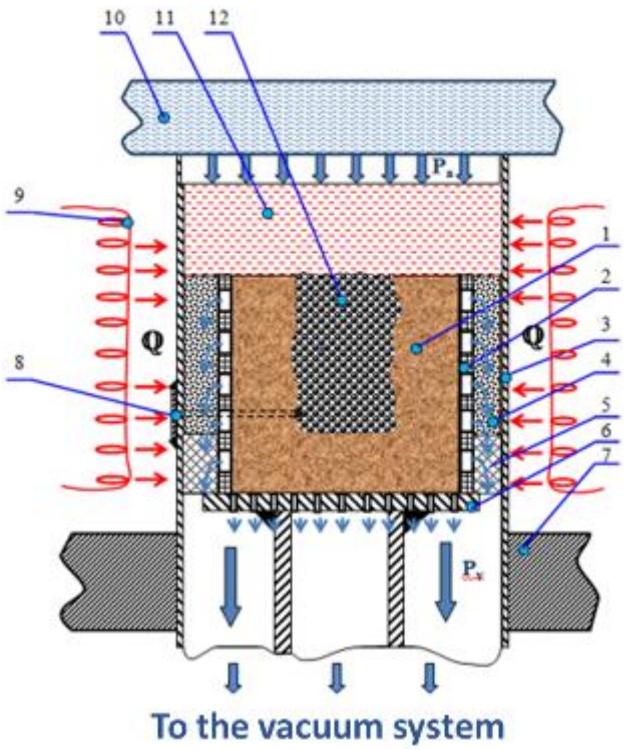
Fig. 1. Infiltration of the melt (the matrix) in the capillary spaces between the elements of the reinforcement phase: a- at the beginning of the pouring; b- after completed infiltration

The main element of this scheme is the chamber developed, where the moulding flask vacuum takes place in three directions. This effect is achieved by perforating the moulding flask frame 2. Thus the pressure in the mould becomes equal along the side surfaces and the lower surface which acts as extra compaction of both the melt and the constituents of the composite.

2.3 Experimental procedure

In order to realize the given task, a laboratory device was developed, which principle scheme is shown in Figure 2.
The experiment involved cleaning of the ceramic

particles from dirt (dust, grease) carried out in an ultrasonic bath using alcohol as a cleaning medium. Then they are put in the mould 1. The mould with ceramic reinforcement phase) is put in the laboratory device for MMCs obtaining and is heated up by heaters 9 to the melting point temperature of the metal matrix.



1- a mould; 2- a perforated flask; 3- a steel shell; 4- a thermo-insulation seal; 5- a fireproof insert; 6- a fireproof stand; 7- a fireproof plate; 8- a thermocouples; 9- a heaters; 10- a fireproof insert; 11- melt; 12- melt (matrix) and reinforcement phase particles.

Fig. 2. A general view of the MMCs production laboratory device

After reaching the required temperature the melt (from metal matrix) is added wich self-hermetizes the system. As a result, the melt is infiltrated forcefully in the capillary spaces among the reinforcement phase by vacuum with a pressure:

$$\Delta p = p_a - p_v \text{ [kPa]}, \quad (1)$$

where: p_a – the atmospheric pressure; p_v – the pressure in the capillary spaces among the reinforcement phase;

The atmospheric pressure acts over the melt, but under the melt the pressure is lower since the space in this area is connected with the vacuum system. To overcome the surface tension of the melt and ensure good wetting of the particles of the reinforcement phase, the use of surfactant borax flux was also tested in some experiments [7, 8].

The method described has been applied for conducting a number of experiments designed to obtain MMCs with different metal matrices and different ceramic particles used as a reinforcement phase. The combinations of a metal matrix and a reinforcement phase in the specified and tested composites are presented in Table 2.

Table 2. The combinations of a metal matrix and a reinforcement phase in the tested composites

Test Procedure №	**Metal matrix materials**	**Reinforcement phase materials (size, µm)**
1	Cu	corundum Al_2O_3 (50- 100)
2	aluminum alloy AlSi12	corundum Al_2O_3 (50- 100)
3	aluminum alloy AlSi12	quartz particles SiO_2 (100- 150)
4	copper alloy CuZn38Pb2	quartz particles SiO_2 (150- 250)
5	AISI 304 stainless steel	corundum Al_2O_3 (250- 350)
6	Cu	Carborundum SiC (180)

The experimental samples obtained were used to produce test objects for macro and microstructural analysis. Macrostructure was examined in different zones of the samples in terms of height and cross section with a binocular microscope. The micro-structural analysis was carried out on prepared metallographic sections using an optical metallographic microscope [9].

3. RESULTS AND ANALYZING

The results of the experimental studies conducted largely verify the possibility to obtain a MMC of the invitro type from non-reactive and mutually insoluble components by applying the basic principles of the capillary effect (capillary forming). In addition, the MMCs obtained features firm mechanical bonding. MMCs samples of the invitro type were obtained from different combinations between a metal matrix and a reinforcement phase (and different reinforcing ceramic particle sizes and matrices with both low and high melting point as well). The experimental study has also identified some typical technological characteristics accompanying the composite forming process.

Test Procedure 1 (Metal matrix materials Cu and reinforcement phase Al_2O_3 (50- 100 µm)) involves the addition of a borax flux surfactant (as a surface-active agent). The microstructures in Figure 3 indicate that the results obtained are not satisfactory. The dark areas that can be observed signal defects mainly formed by the flux used, which remained between the metal component of the composite obtained and the reinforcement phase. Such defects are not compensated for and eliminated in the vacuum process (Figure 3(b)). Due to the vacuum, the flux cannot be pushed out of the area where the composite is formed. Thus, it remains between the metal matrix and the reinforcement phase. However, wetting of the corundum copper particles is observed, which is an indicator of a strong mechanical bond between the matrix and the reinforcement phase. Taking into account the unsatisfactory results of this experiment, a surfactant (flux) was not used in the subsequent experiments.

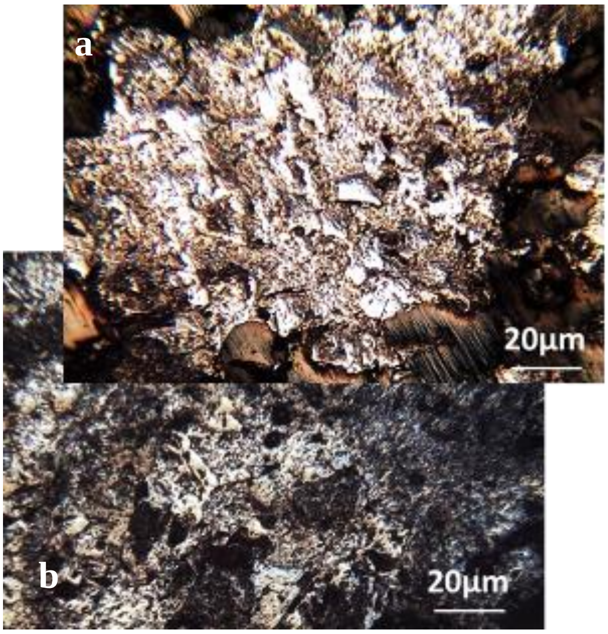


Fig. 3. Microstructures of Test Procedure 1: a- after etching x100; b- before etching x100

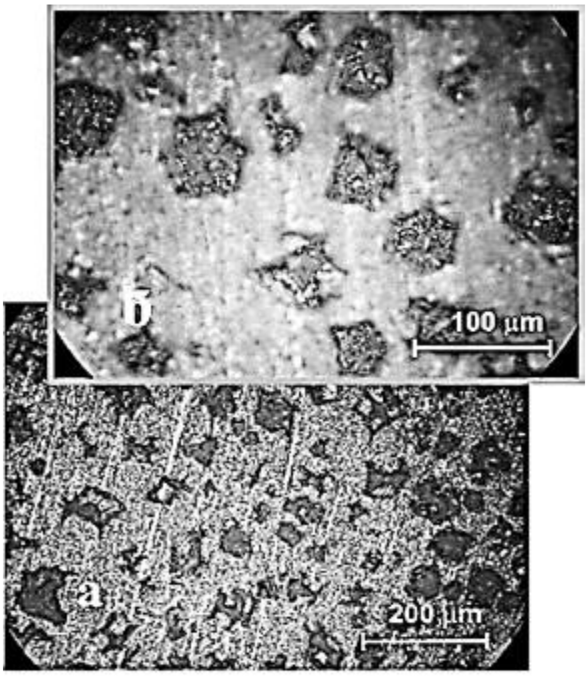


Fig. 4. Microstructures of Test Procedure 2: a- after etching x100; b- after etching x200

The microstructural studies of the test samples obtained under Test Procedure 2 (Metal matrix materials AlSi12 alloy and reinforcement phase Al_2O_3 (50-100 µm)) (Figure 4) and Test Procedure 3 (Metal matrix materials AlSi12 alloy and reinforcement phase SiO_2 (100-150 µm)) (Figure 5) confirm the predicted satisfactory results of strong mechanical bonding.

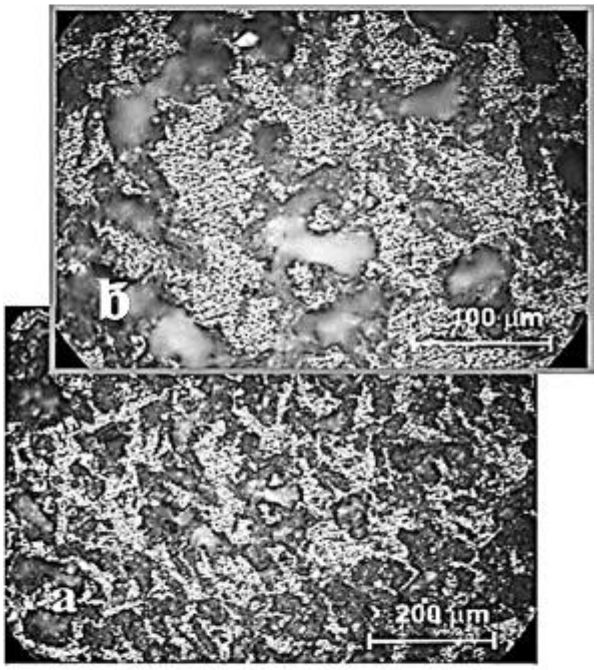


Fig. 5. Microstructures of Test Procedure 3: a- after etching x100; b- after etching x200

The experiments indicate complete filling of the capillary cavities around the reinforcing particles with the liquid metal, the particles showing a very good wetting by the melt. This is proven by the absence of any gas "pockets", separation strips or other defects of this type in the interface between the particles of the reinforcement phase and the metal matrix.

The next two variants, Test Procedure 4 (Metal matrix materials CuZn38Pb2 alloy and reinforcement phase SiO_2 (150- 250 µm)) and Test Procedure 5 (Metal matrix materials AISI 304 stainless steel and reinforcement phase Al_2O_3 (250- 350 µm)), feature larger sizes of the reinforcement phase, i.e. the boundary between the reinforcing particles and the metal matrix is of a larger area.

The microstructure in Figure 6 and Figure 7(a) indicates even distribution of the reinforcing particles throughout the volume of the composite, a dense structure, and very good infiltration of the melt into the capillary spaces formed between the particles of the reinforcement phase. The good wetting of the reinforcing phase by the melt and the stable mechanical bonding formed are clearly observable in Figure 6(b) Moreover, no defects are detected in the interface region between the ceramic particles and the matrix.

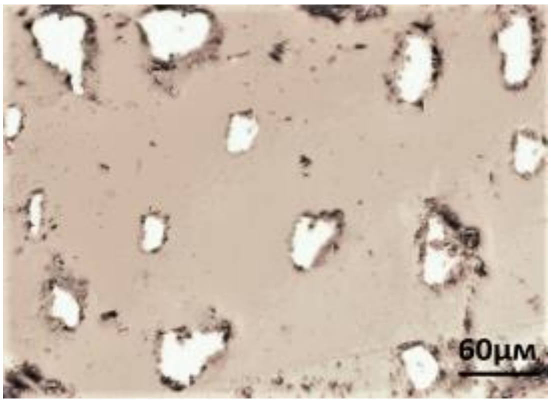


Fig. 6. Microstructures of test Procedure 4 before etching x150

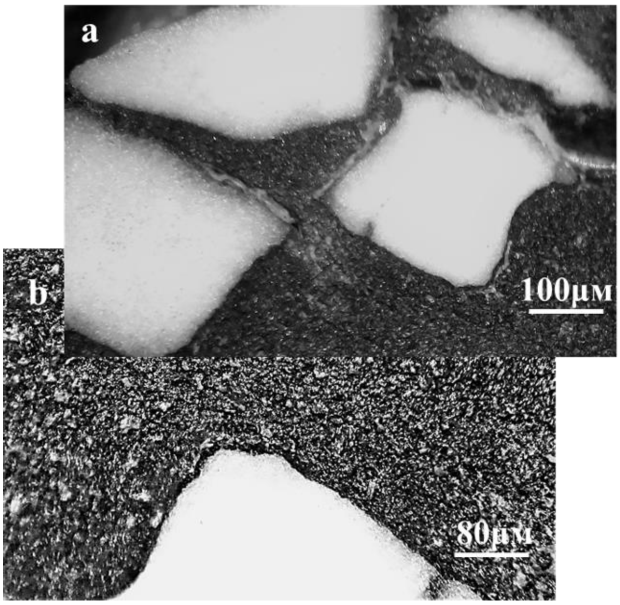


Fig. 7. Microstructures of test Procedure 5; a- before etching x50; b- - before etching x100

The obtained results show that the bigger size of the ceramic particles used does not affect the rate of the wetting, and respectively the mechanical bonding between the elements of the MMCs tested. The same results are observed with MMCs with significantly smaller particle sizes of the reinforcement phase. This is due to the capillary effect scheme applied, where the forced infiltration of the melt into the capillary spaces, helps to overcome the surface tension of the melt and provides good wetting of the reinforcement phase particles.

Unfortunately, with Test Procedure 6 (Metal matrix materials Cu and reinforcement phase SiC (180 µm)), the results did not comply with the expectations. In this case, the wetting of the reinforcing phase by the metal matrix was not satisfactory and a large volume of unfilled capillary spaces was observed. This is obvious and clearly seen in Figure 8(a). Accordingly, this resulted in forming unstable bonding between the elements of the reinforcement phase, which led to low strength of the cast composite and disruption of its structural integrity (Figure 8(b)).

The ceramic particle surface normally is covered with a gas layer. May be this gas layer is the main reason for the poor wettability of SiC such as prevents molten matrix material from coming into contact with the surface of individual particles. When we have more SiC particle concentration in a melt, the gas layers can form a barrier, which can be leading to total rejection of particles from the melt [10]. This is a problem that will be the object of further research.

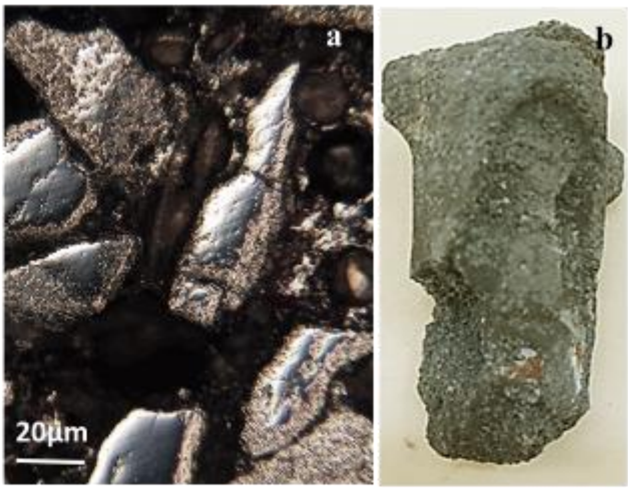


Fig. 8. a- microstructures of test Procedure 6 before etching x500; b- the obtained complex relief MMC by Procedure 6

The stable mechanical bond between the ceramic particles and the metal matrix is indicated by the macro-fractographic analysis of some MMCs samples, upon their fracture under impact bending load. Figure 9 illustrates the type of fracture of Test Procedure 2 (Metal matrix materials AlSi12 alloy and reinforcement phase Al_2O_3 (50-100 µm)) and Test Procedure 5 (Metal matrix materials AISI 304 stainless steel and reinforcement phase Al_2O_3 (250-350 µm)).

It can be assumed that the sample is characterized by good structural homogeneity, i.e. even distribution of ceramic particles in the metal matrix. In general, as a result of the reinforcement phase redistribution during the melt infiltration, no (or no noticeable) areas of reduced reinforcement phase content are detected. In addition, upon fracture, no erosion of ceramic particles from the metal matrix is observed, which indicates resilient mechanical bonding.

Some of the MMCs obtained by this method are shown on Figure 10 and Figure 11. The MMCs shown at Figure 10 are cylindrical in shape, but the MMCs on Figure 11 are with complex relief and produced in expendable moulds, constructed by liquid self-hardening mixture (based on cement and gypsum) not as in the conventional methods in metal moulds.

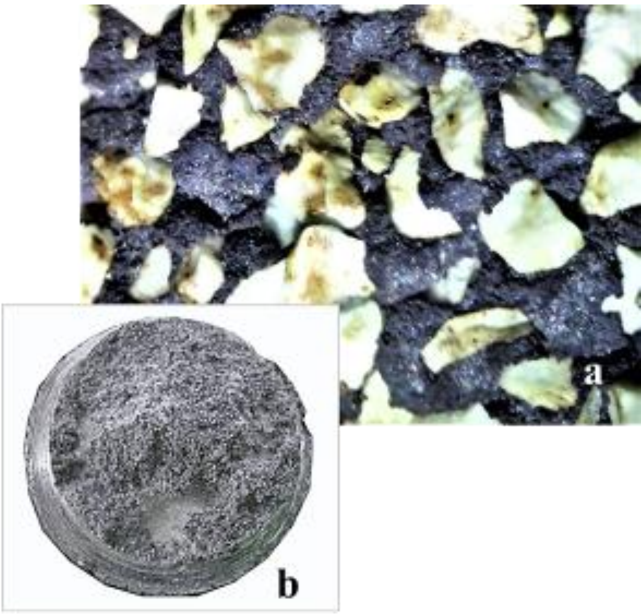


Fig. 9. Macro-fractographic analyse of “a”- Test Procedure 2 and “b”Test Procedure 5 x25

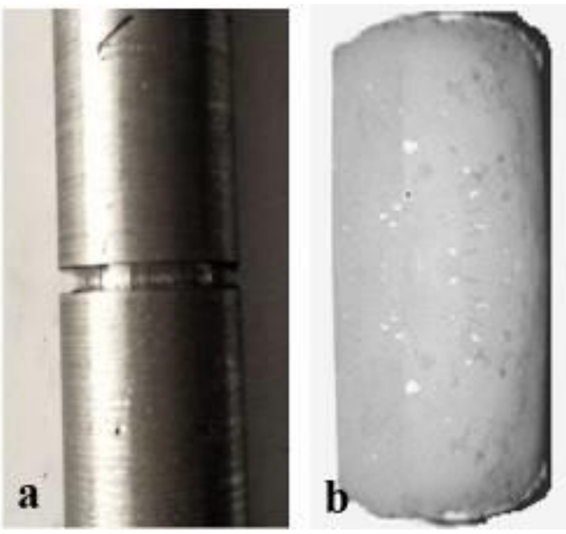


Fig. 10. Picture of the obtained MMCs by: a- Test Procedure 2; b- Test Procedure 5

Fig. 11. Picture of the obtained MMCs by: a- Test Procedure 1; b- Test Procedure 3; c- Test Procedure 4

4. CONCLUSIONS

After the experiments conducted, based on the results obtained we can conclude that the method under consideration for MMCs poduction, based on the capillary effect, provides stable mechanical bonding between the matrix and the reinforsment phase, as the forced infiltration of the melt into the capillary spaces, helps to overcome the surface tension of the melt and provides good wetting of the reinforcement phase particles. And another advantage is that the production of single blanks is economically viable, since the MMCs areproduced in expendable moulds, constructed by liquid self-hardening mixture (based on cement and gypsum) not as in the conventional methods in metal moulds. Also the obtained results show that the size of the ceramic particles used does not affect the rate of the wetting, and respectively the mechanical bonding between the elements of the MMCs tested. This is due to the capillary effect scheme applied, where the forced infiltration of the melt into the capillary spaces, helps to overcome the surface tension of the melt and provides good wetting of the reinforcement phase particles. The other main factor- the melting temperature of the various alloys forming the matrix does not have an impact on the degree of wetting and the quality of the mechanical bonding obtained. Unfortunately the use of surfactants in the MMC formation does not yield good results. Defects are observed from the flux used, which remained between the metal matrix component and the reinforcement phase of the composite. The rezults of obtained MMCs with SiC rainforcement phase a not good either. This is a problem that will be the object of further research.

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