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ABSTRACT

CERCETĂRI PRIVIND OBŢINEREA UNOR COMPOZITE DE UTILITATE TEHNOLOGICĂ CU MATRICE METALICĂ DIN ALIAJE DE ALUMINIU ȘI PARTICULE GREU FUZIBILE

RESEARCH REGARDING THE OBTAINING OF SOME TECHNOLOGICAL UTILITY COMPOSITES WITH ALUMINUM ALLOYS BASE METAL MATRIX AND REFRACTARY PARTICLES

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Cuvinte cheie/ Keywords

Material compozit	Composite material
Matrice metalică	Metallic matrix
Particule granulare	Granular particles
Feroaliaje	Ferroalloys
Rol tehnologic	Technological role
Metoda Stir Casting Vortex	Stir Casting Vortex method
Metoda de amestecare mecanică prin vibrații	Method of mechanical mixing by vibrations
Metoda de dezoxidare prin precipitare	Deoxidation method by precipitation
Tensiunea superficială	Surface tension
Metoda picăturii pe plăcuță	Sessile drop method
Unghi de contact	Contact angle
Vâscozitatea dinamică	Dynamic viscosity
Distribuție uniformă	Uniform distribution
Reactivitate chimică la interfață	Chemical reactivity at interface
Înglobare	Embedding
Analiză macrostructurală	Macrostructural analysis

Introduction

Composite materials composed from an aluminium alloy as matrix and a dispersed phase represented by refractory particles of FeSi45, FeTi30 and SiC, have been designed for the technological purpose of deoxidation by precipitation of steels melts, combining synergistic the positive effects of the two component elements, represented by alloy A6061 and granular particles.

The doctoral thesis has the purpose of obtaining and characterization of composite materials with metallic matrix and disperse refractory particles, using a liquid phase method known as Stir Casting Vortex (SCV) and a new mechanical mixing method, respectively mechanical mixing with the aid of vibrations.

Emphasis is laid on minimizing some problems such as chemical reactions between matrix and dispersed phase and achieving a distribution as uniform as possible of granular materials FeSi45, FeTi30 and SiC in metallic matrix.

An important part of this thesis consists inthe design and achievement of a special equipment which has allowed obtaining composite materials with metallic matrix and researching the influence of the process parameters, as well as research of their physical-chemical properties.

Metallurgical practice from steels mills does not provide sorting on granulometric classes but only the excluding of the use from the steel development of granular particles with substandard dimensions, which thus become waste and pollute the environment.

Grinding feroalloys FeSi45, FeTi30 appears after the successive manipulations on route: production – packing – loading – transport – unloading warehouse – transport section (raw materials) – transport platform.

The present standard SR ISO 5445/1995 imposes an inferior limit for granulation of 3.5 mm particle diameter for ferroalloy use on steel making in the deoxidation by precipitation stage. As a result of being solid materials with low granulation, the following effects may occur:

- economic, in the sense of rising costs;
- ecological, in the sense of polluting industrial spaces.

In extractive industry there have appeared several methods of recovery of such materials by briquetting and compacting, technologies which require specialized equipment with high energy consumption without guaranteeing the material compactness. Furthermore, the treated steel contamination in view of deoxidation through organic binders used especially in briquetting process is possible.

The research objective is that of achieving experimental research not mentioned in literature and namely to find a way by which granular ferroalloy (FeSi45, FeTi30) with substandard dimensions can be valorized, by an efficient method which does not produce chemical changes in the chemical composition. Also, research has been conducted for the use of silicon carbide SiC, in granular state, at steel deoxidation and recarburizing with the aid of composites containing SiC under the form of embedded particles into an metallic matrix.

Aluminium and some aluminium alloys are used currently for deoxidation steels, therefore, alloy A6061 was considered a good matrix for embedding waste of ferroalloys and silicon carbide. Thus, we obtained composite materials with metallic matrix from aluminium alloy A6061 and dispersed granular phase of FeSi45, FeTi30 and SiC.

Obtaining new materials was determined by the necessity of responding to some requirements of valorification of ferroalloys and silicon carbide as particles with medium diameters lower of 3.5 mm.

Newly created composite materials present technological utility, being suitable for use in steel making. Both components of composite materials, aluminium alloys and dispersed phase, are used into technological stage of deoxidation by precipitation, combined deoxidation being used in this case. This method of deoxidation has a synergistic effect, unlike deoxidation in sequence, because the two deoxidizing materials are present at the same time and place. Composite materials with technological utility are made of a metallic matrix of aluminium alloy A6061 and particles in various forms and dimensions of ferroalloys FeSi 45, FeTi30 and SiC.

Granular particles (FeSi45, FeTi30 and SiC) have been subjected to granulometric ranking, their average diameter being determined for the study of the granular particles behaviour at contact with matrix of aluminium alloy according to medium diameter of particles (d_{mp}) and to the possibilities of obtaining of some complex deoxidants, as well as obtaining of a material with double purpose deoxidation-recarburation, respectively A6061/SiC.

From all spectra of dimensions, three representative granular sizes: d_{mp} = 0.04 mm, d_{mp} = 0.4 mm, d_{mp} = 0.8 mm have been chosen for the present study. Granular ferroalloys invery small dimensions cannot be used as such in the steel making process because they cannot penetrate the slag.

Embedding granular particles into liquid phase of aluminium alloy which after solidification will form metallic matrix, makes that interface processes (melt – granular particles) to be decisive in obtaining composite materials.

The research on the interface phenomena have highlighted the purpose of superficial tension, interphasic tension and contact angle on wetting and of component adhesion phase. Also, there are highlighted the formation of new phases such as intermetallic compounds or chemical as a result of diffusion processes and chemical interaction between aluminium and certain elements falling within the chemical composition of refractory particles, temperature condition, time and blending mode imposed.

The method used for the determination of the superficial tension in the systems researched was "sessile drop", and a piece of equipmentwas designed for this purpose.

Dynamic viscosity is an important property in the systems liquid-granular particles because it describes the rheology of analysed heterogeneous systems.

For pursuing experimental research regarding obtaining of composite materials two method of mechanical mixing have been studied: the Stir Casting Vortex method and the mechanical mixing by vibrations.

Experimental conditions allowed for the obtaining of some composite materials with metallic matrix of aluminium alloy and refractory granular particles of FeSi45, FeTi30 and SiC.

Composite materials can be obtained by using various technologies, chief among which processing in liquid state, respectively the technologies by mixing Stir Casting Vortex and by vibration, are considered as having the greatest potential in point of production capacities and cost efficiency for different applications such as: applications in the technological field, in steels making and for the machines constructions field, etc.

The main factors which control the properties of metallic composites, obtained using elaboration technology, include: the distribution as uniform as possible of the granular particles, the wetting process of granular materials toward metallic matrix, chemical reactivity at interface particle/matrix and the content of porosities resulted from the obtaining process. Embedding of some granular particles of refractory materials in liquid matrix represented by aluminium alloy A6061 in a large quantity is difficult because of the poor wetting of particles by the melt.

Rising temperature of aluminium alloy liquid [1], oxidation particle in granular state, adding into melted alloy of some active superficial elements such as magnesium and lithium [2] and blending of melt matrix for an adequate time, are some of the ways of improvement wetting of granular particles on one hand, and on the other hand the mixing and the retaining in matrix of particles to be as easy as possible.

Ferroalloys FeSi45, FeTi30 and SiC as well as aluminium alloys are used as reinforcement materials and respectively metallic matrix, because of the compatibility between these materials, as well as for the potential properties, when are combined.

For finding the optimal conditions necessary for realizing composite materials with metallic matrix and refractory particles further research has been conducted in the following directions: contact angle measurements, determination of surface tension, measuring dynamic viscosity, determining the thermal properties, chemical analysis, macro and microstructural analysis, determination of micro hardness and phase analysis of samples from composite materials studied. Experimental results have shown that, by proposed technologies, composite materials with metallic matrix for special purposeswere obtained in a profitable way. The proposed elaboration methods allow solving the wetting problems associated with these obtaining technologies. Macro and microstructural analyses show that the particles distribution in the metallic matrix is relatively good.

For accomplishing the purposes of this thesis, equipment and apparatus available in the laboratories of “Dunărea de Jos” University of Galați have been used. The equipment has been designed and realized by the author of the doctoral thesis in the laboratory of elaboration from Faculty of Engineering, as well as the apparatus from ICECHIM S.A. Bucharest for thermal analysis.

The research studies in the field approached in the doctoral thesis, the methods description, designing and achievement of equipment for obtaining composite materials with metallic matrix, the study of liquid phases properties (superficial tension, dynamic viscosity) from the development of composite materials, presentation of the investigation methods of materials, results and general conclusions from this paper are organized into 6 chapters as follows:

- Chapter 1 presents the current state of research regarding the used materials and methods of obtaining of composites with metallic matrix. Also, some of the major criteria underlying decision of producing composite materials are presented. A description of granular materials introduced in metallic matrix is provided, the current technologies used in obtaining composite materials as well as the research undertaken in similar systems with those studied within the framework of the thesis.
- Chapter 2 analyses and characterises the materials used in the process of obtaining composites, from the point of view of physical-mechanical properties, macro and microstructural properties (optical microscopy and electronic microscopy), as well as chemical analysis and of phases of composite materials.
- In chapter 3 we present the experimental research for obtaining composite materials with metallic matrix from aluminium alloy A6061 and granular particles FeSi45, FeTi30 and SiC, by Stir Casting Vortex method (SCV).

With consideration to the principles of SCV method, the author of the thesis has designed and made the plant used for obtaining composite materials from the systems A6061/FeSi45, A6061/FeTi30 and A6061/SiC. The composites obtained have been investigated and their chemical composition was determined, being analysed from the macro and microstructural point of view. The yield embedding of granular particles in metallic matrix has also been calculated.

- Chapter 4 presents the results of the experimental research results regarding the method mechanical mixing with vibrations of composites with metallic matrix and granular particles. The composites obtained from metallic matrix (aluminium alloy A6061) and granular particles FeSi 45, FeTi30 and SiC are characterised. The characteristics and the working principle of elaboration plant are, the plant is designed and produced by the author of the doctoral thesis.
- Composite materials, elaborated according to the experimental plan have been analysed in order to determine the embedding yield of granular particles, to evaluate the chemical composition, macro and microstructure, and the thermal characterization of composites, determination of microhardness and structural analysis by x-ray diffraction.
- Chapter 5 displays the results of the experimental research and the research mode regarding surface tension determination and the dynamic viscosity of composites in liquid state, corresponding to the systems A6061/FeSi45, A6061/FeTi30 and A6061/SiC.

The equipment was designed and produced by the author for the determination of the contact angle between liquid alloy and solid phase allowing the calculus of surface tension, and for the determination of dynamic viscosity of melted aluminium alloy, with and without granular particles used to composites obtained, was realized another equipment.

Chapter 6 presents the general conclusions, the original contributions and future directions for research.

CHAPTER 1
CURRENT STATUS OF RESEARCH REGARDING THE OBTAINING COMPOSITE MATERIALS WITH METALLIC MATRIX AND GRANULAR REFRACTARY

1.1. Composite materials with metallic matrix

Composite materials are a combination from at least two different materials with a separation interface of the two constituents. The accomplishment of these composite materials for a given application is based on rational selection of obtaining technology, of matrix and reinforcements.

Composite materials are also designed for other purposes than those classically presented, namely new technological materials combining intrinsic properties of constitutive parts, respectively, aluminium alloy and ferroalloys particles FeSi45, FeTi30 and SiC, for obtaining of some complex deoxidants used on elaboration steels.

1.2. Selection criteria of composite materials with metallic matrix

The purpose of designing composite materials with metallic matrix is that of combining desirable properties of metals and reinforcements, one of them being to serve as transport media of granular particles of ferroalloys FeSi45, FeTi30 and SiC in melting steel in technological steps of deoxidation by precipitation of steels.

1.2.3. Uses of composite materials

If composite materials used for technological applications are obtained with the purpose of high reusability of granular particles of ferroalloys FeSi45, FeTi30 and SiC, it is important that intermetallic compounds in high concentration do not occur at the interface, nor do defects such as pores.

1.3. Selection criteria of material for metallic matrix

For composite materials with metallic matrix different metals and alloys may be used. For composite materials with technological utility, metallic matrix can be an aluminium alloy. In this case, important are its surface properties (surface tension and contact angle) for favouring of an yield of embedding as high as possible.

1.4. Selection criteria of reinforcements

Technological purpose of reinforcements is to facilitate their transport into an metallic melt, if the composite material are used for deoxidation steels or recarburation, or to take external forces.

In the present paper other particles such as ferroalloys FeSi45 and FeTi30 were tested in technological purpose. Studying these granular materials regarding surface properties (superficial tension and contact angle) helps explaining some processes which determine yields of embedding in metallic matrix.

1.5. Obtaining methods of composite materials with metallic matrix

Obtaining in liquid state of composite materials, when granular particles remain in contact with melted alloy of matrix for a long time and locally can result in a reaction between the two. Stir Casting Vortex method is not a suitable technology for materials like fibre or filaments because mixing produces their rupture [34].

For obtaining composite materials with granular particles with nano dimensions, research has been performed with the help of technologies which uses mechanical mixing with ultrasounds to form the "ultrasonic cavitation" effect [43].

1.5.3. Obtaining methods in liquid state

With Stir Casting Vortex method, granular particles are embedded in melted matrix using different technologies followed by mechanical mixing or pressing and casting, resulting a composite material. In this process, a strong bond between matrix and reinforcement is achieved at high obtaining temperatures.

In the case of Stir Casting Vortex method [50], described in figure 1.2., reinforcement is introduced in the created vortex of liquid metallic material by mechanical mixing. Reinforcement is uniformly distributed in melt, then the composite material resulted can be casted.

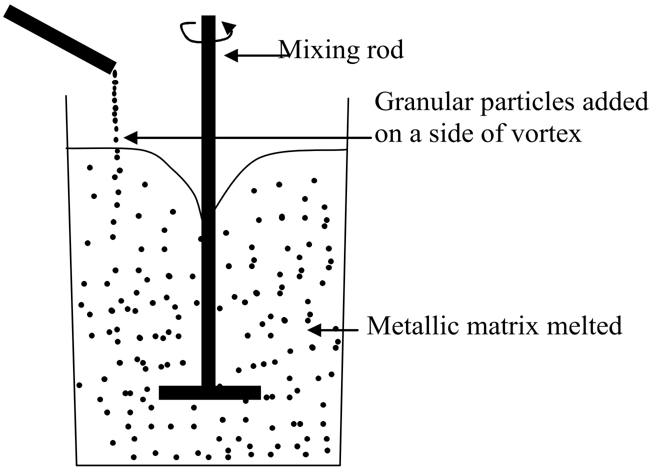


Figure 1.2. Stir Casting Vortex method [50]

For obtaining composite materials by Stir Casting Vortex method, one should be aware of the following factors which influence manufacture: field of temperature (above lichidus temperature or under lichidus temperature); introducing some quantities of granular particles in metallic melts; preparing granular particles (preheating or superficial oxidation); obtaining environment pressurized/vacuum or in inert environment; preparing surface of metallic ingot; using of different forms of blades for mechanical mixing; establishing of rotation speed for achieving superior yields of embedding of granular particles.

CHAPTER 2
MATERIALS, METHODS AND EXPERIMENTAL PROCEDURES

In the present chapter, the physical, chemical and mechanical properties of metallic matrix, aluminium alloy A6061 are extensively analyzed, together with the granular particles of ferroalloys of FeSi45, FeTi30 and SiC. The extensive description of used materials' properties has the purpose of knowing and designing the technologies of obtaining in liquid state of composite materials by mechanical mixing, by Stir Casting Vortex method and by the method of mechanical mixing by vibrations.

2.1. Materials used in the frame of experiments

The materials used in the process of obtaining composite materials with metallic matrix and refractory particles are:

- Aluminium alloy, A6061 [185] with chemical composition presented in tab. 2.1., and
- Refractory particles of FeSi45, FeTi30 and SiC.

2.1.1. Characterization of aluminium alloy used for the metallic matrix of the studied composites

In table 2.1. some of specific properties of alloy A6061 were shown. These properties reflect some of the conditions required for obtaining composite materials of type A6061/FeSi45, A6061/FeTi30 and A6061/SiC.

Table 2.1. Chemical composition of aluminium alloy A6061 [185]

Aluminium alloy	Chemical composition (%)								
	Si	Mg	Cr	Cu	Fe	Mn	Ti	Zn	Al
A6061	0.4 – 0.8	0.8 – 1.2	0.04 – 0.35	0.15 – 0.4	Max 0.7	Max 0.15	Max 0.15	Max 0.25	95.8 – 98.6

2.1.2. Characterization of granular materials used in the obtaining process of composite materials

Refractory materials in granular state, of ferroalloy are waste, generally resulting on the route obtaining – transport – manipulations – use. These materials (FeSi45, FeTi30 and SiC) are recognized [186], [187], [188], [189], [190], [191], [192] as being hard by friable (they easily break). We can specify that, with rising of element of alloying in concentration, the friability is reduced on all types of ferroalloys used in the present research [192].

2.1.2.1. Physical properties determination of materials in granular form

a. The establishment of solid particle form

The form of granules is generally determinated, by the grinding process following succesive manipulations of granular materials of ferroalloys and SiC. Granular particles may have different forms, such as: spherical, elongated lamellar, lenticular, needle-like, polyhedral or foam, dendritic and others, as it can be seen in figure 2.2.

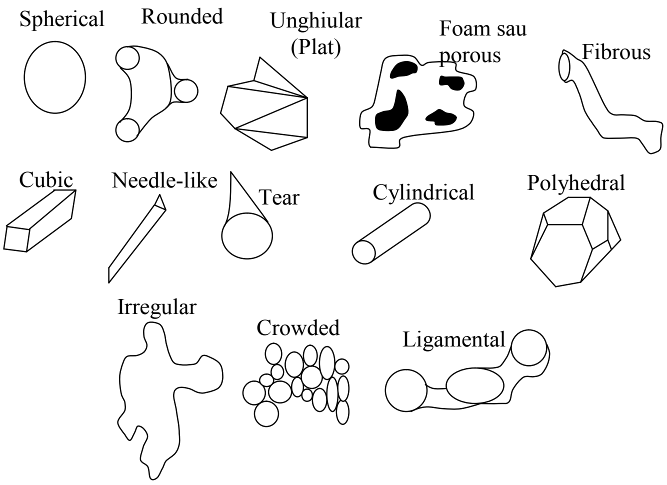
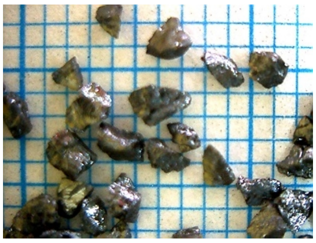
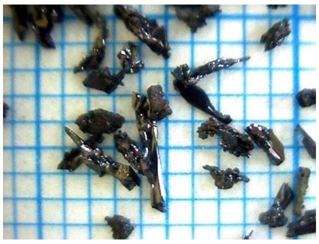
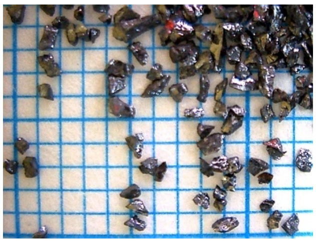
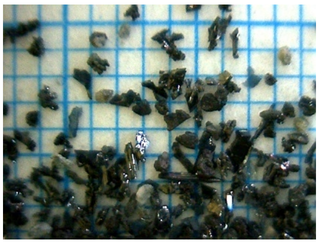
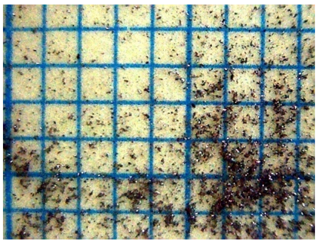
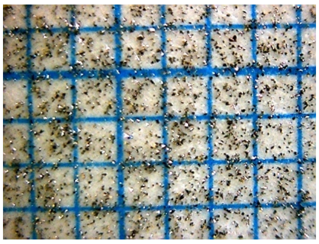
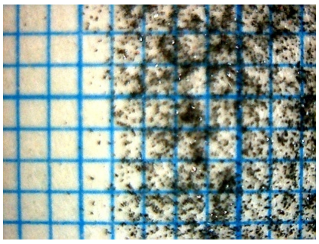


Figure 2.2. Granular particles under different forms

Tabel 2.4. Images of particles of FeSi45, FeTi30 and SiC on a millimeter grid with division of 1 mm

No.	Granulation	Granular materials types		
	mm	FeSi45	FeTi30	SiC
1	d_{mp} = 0,80			
2	d_{mp} = 0,40			
3	d_{mp} = 0,04			

By analysing the macrographs presented in table 2.4, we may observe that, regarding the shape of the particles from refractory particles used for obtaining composites with matrix of aluminium alloy, respectively those of FeSi45 and FeTi30 are mainly polygonal and spherical due to the fragile breaking character during the grinding process, and those of SiC are needles-like form.

b. Determination of particle size and the particle size fraction

Within the frame of experiments made, particle size, respectively granular distribution, were determined by sieving through a set of chosen site in accordance with SR EN

24497:1994, with known mesh size, then fractions were weighed separately and the procentual mass was calculated. In figure 2.3 is shown granulometric distribution obtained by sieving for granular particles of FeSi45.

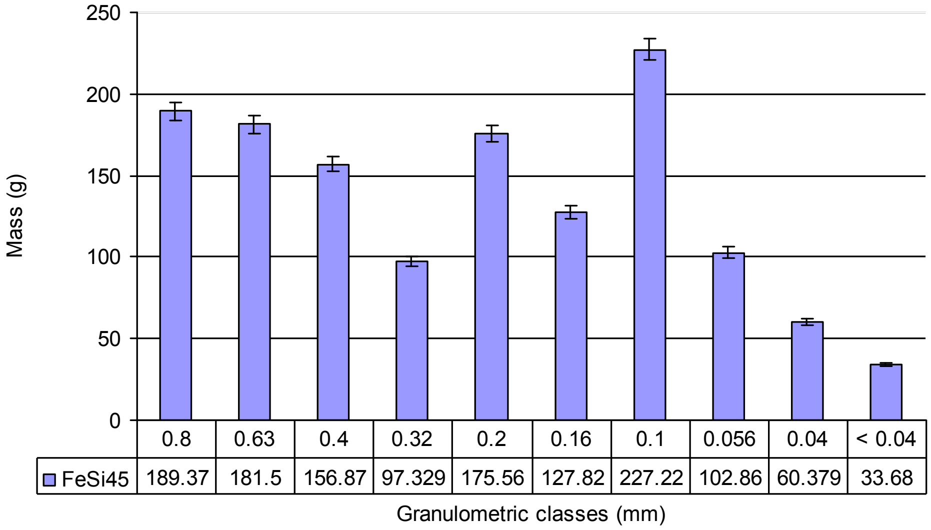


Figure 2.3. Granulometric distribution of granular particles of FeSi45 obtained by sieving

By analysing the granulometric distribution of granular particle of FeSi45 from figure 2.3, we may observe that particles with d_{mp} = 0.8 mm and d_{mp} = 0.4 mm are important fractions from the analysed material, which justifies the necessity of knowing their behavior at obtaining composite materials.

In figure 2.4 we present granulometric distribution for granular particle of FeTi30.

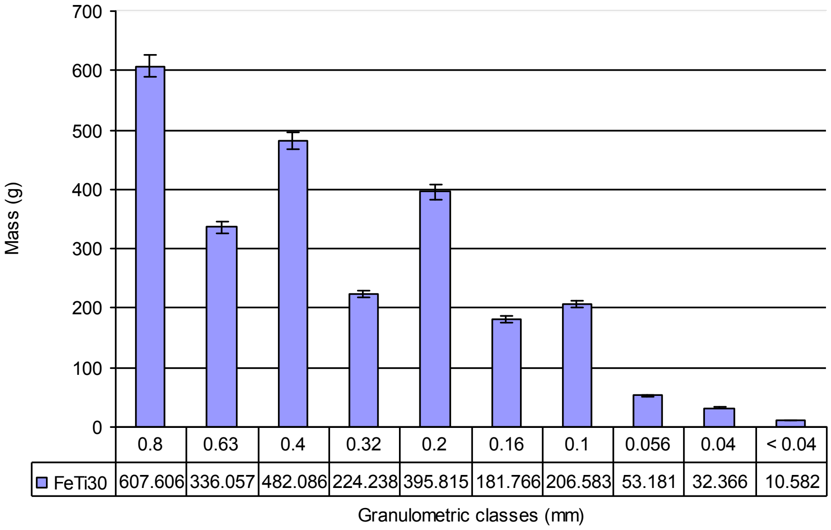
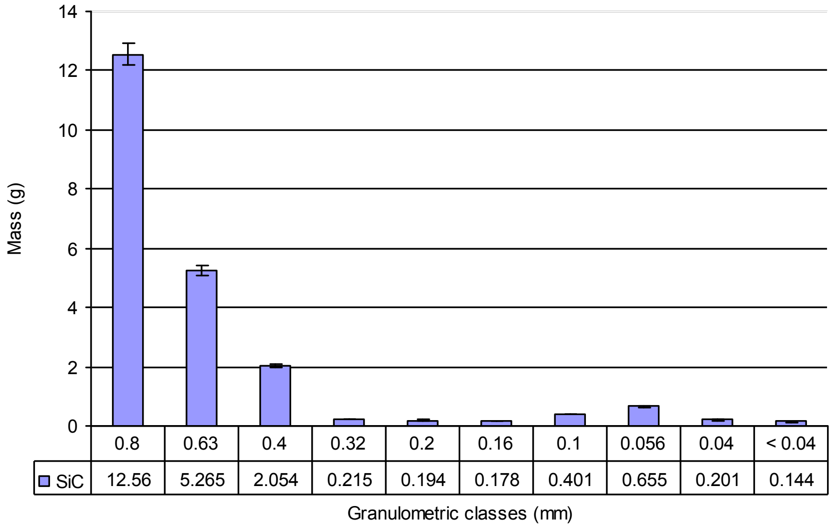


Figure 2.4. Granulometric distribution of granular particles of FeTi30

By analysing granulometric distribution of granular particle of FeTi30 from figure 2.4, we may observe that their friability property determines the creation of particle which belongs to more granulometric classes. It results that there are not preferences for a specific dimension. These consequences are due to the mechanical impact energy applied to large chunks of particles.

In figure 2.5 we present granulometric distribution for granular particles of SiC.



	0.8	0.63	0.4	0.32	0.2	0.16	0.1	0.056	0.04	< 0.04
SiC	12.56	5.265	2.054	0.215	0.194	0.178	0.401	0.655	0.201	0.144

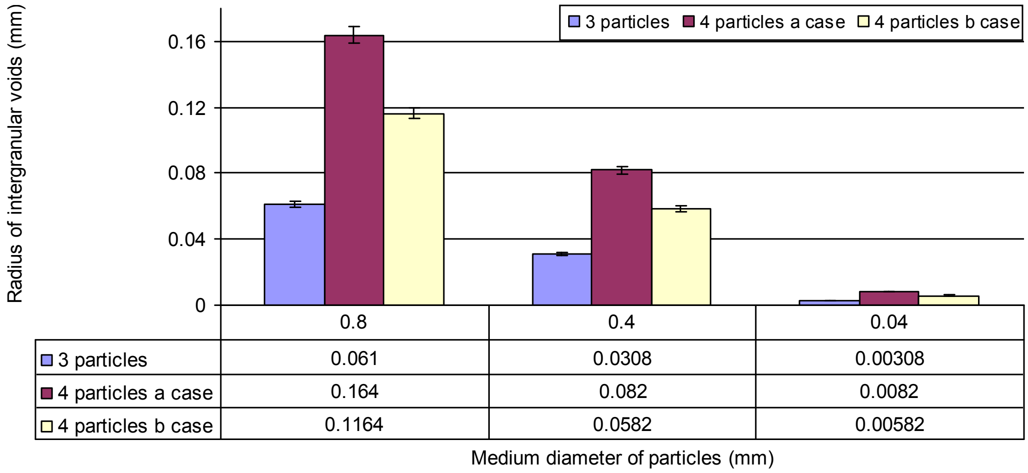
Figure 2.5. Granulometric distribution of granular particles of SiC

By analysing the granulometric distribution of granular particles of SiC from figure 2.5 we may observe that there is a diversity of dimensions, as a result of a locally condition consequence of mechanical grinding (chunks with reduced dimensions are dislocated from the chunks of coarse material).

c. The influence of permeability of particles in granular state concerning infiltration of alloys in liquid state

Permeability is defined as the property of a fluid to infiltrate a particle layer. Of great importance is the physical dimension of the intergranular space, because the bigger the intergranular space is, the easier for the liquid to infiltrate the layer.

Knowing the interparticle diameter space (for the case of spherical shape), figure 2.15, represents a very useful evaluation because it can determine with a good approximation the minimum volume of liquid alloy intended to fill these voids and we can determine the minimum pressure imposed because the gaps between the particles are filled with the alloy.



	0.8	0.4	0.04
3 particles	0.061	0.0308	0.00308
4 particles a case	0.164	0.082	0.0082
4 particles b case	0.1164	0.0582	0.00582

Figura 2.15. Variation size intergranular voids depending on the particle size

We can determine the existence of a linear dependence or by other nature between voids - particles. Intergranular space is the initial void, in static condition without application of mechanical vibration, which aluminium alloy in liquid state fills it partially toward total.

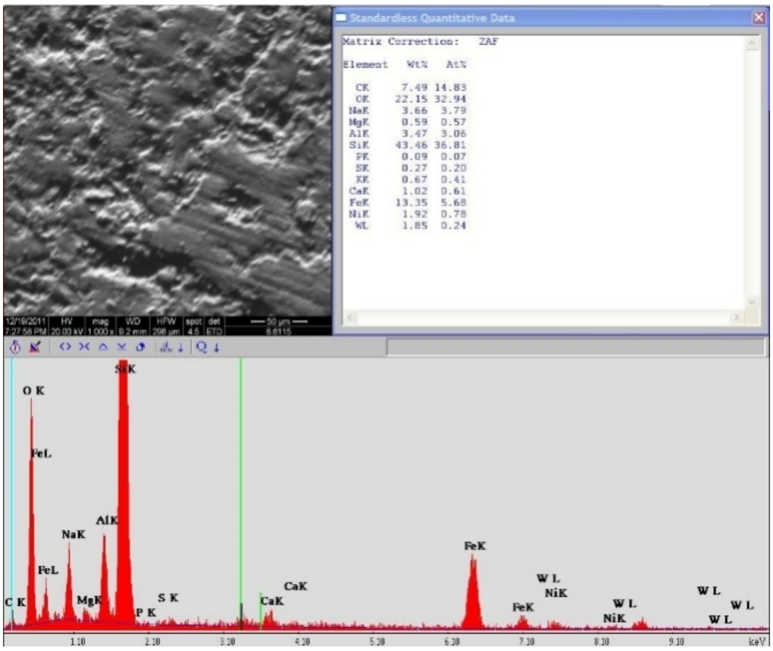
2.1.2.2. Chemical composition determination

The methods for chemical composition determination have been x-ray fluorescence spectrometry (XRF) for which was used spectrometer Innov-x System from Engineering Faculty and electron microprobe EDX from electron microscope SEM Fei Quanta 200 from Sciences and Environment Faculty of “Dunărea de Jos” University of Galați. The obtained results, by applying these two methods, for the types of particles researched, are presented in table 2.5 comparatively with standard data.

Table 2.5. Chemical composition of granular particles

No.	Type of granular particles	Chemical composition of particles (%)		
		Method EDX	Method XRF	Standard
1	FeSi45	Si – 43.46; Fe – 13.35; O – 22.15; C – 7.49; Al – 3.47; Na – 3.66; W – 1.85; P – 0.09; S – 0.27	Si – 43.6; Fe - 55.53; Mn – 0.54; Cr – 0.33	SR ISO 5445/1995 Si 40 – 47; Mn_{max}- 1; Cr_{max} – 0.6; P_{max} – 0.04; S_{max} – 0.04; C_{max} – 0.5; Al_{max} – 2;
2	FeTi30	Ti – 19.90; Fe – 24.4; O – 27.79; C – 7.07; Al – 10.25; Si – 5.15; P – 0.42; S – 0.65	Ti 34.87; Fe – 53.67; Si -6.24; Al – 4.55; Mn – 0.67	SR ISO 5445/1995 Ti – 35; C_{max} – 0.15; P_{max} – 0.08; S_{max} – 0.05; Cu – 2;
3	SiC	C – 30; Si – 69.99	-	SR ISO 5064:1997 C – 30; Si – 69.99

Figure 2.23 presents a microarea by electron microscopy (SEM) on which EDX analysis was pursued, as well as the result of this analysis for particles of FeSi45.



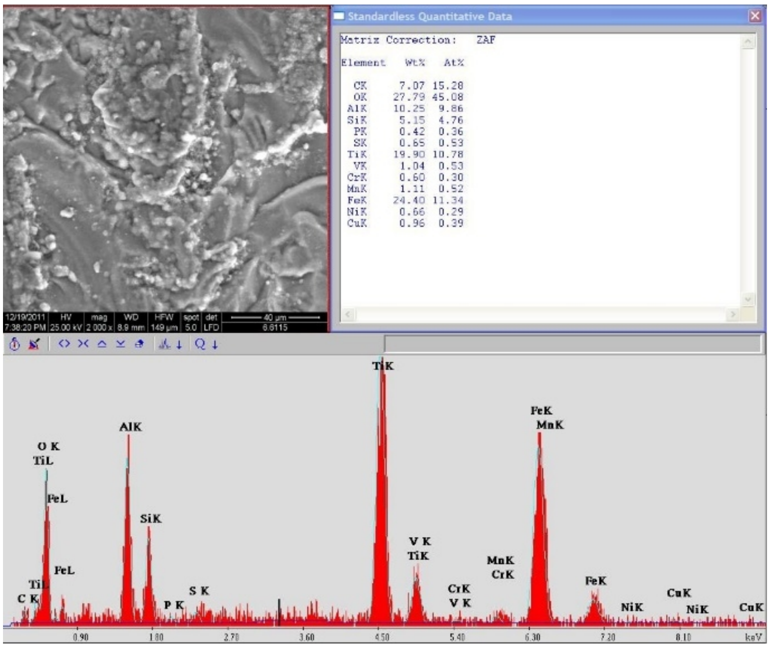
```
Matrix Correction:   ZAF

Element    Wt%   At%

   CK      7.49 14.83
   OK     22.15 32.94
  NaK      3.66  3.79
  MgK      0.59  0.57
  AlK      3.47  3.06
  SiK     43.46 36.81
   PK      0.09  0.07
   SK      0.27  0.20
   KK      0.67  0.41
  CaK      1.02  0.61
  FeK     13.35  5.68
  NiK      1.92  0.78
   WL      1.85  0.24
```

Figure 2.23. Spectra regarding the chemical composition determined by EDX analysis of particles FeSi45

In figure 2.24 we present the result of SEM and EDX analysis for a sample of FeTi30.



```
Matrix Correction:   ZAF

Element    Wt%   At%

   CK     7.07 15.28
   OK    27.79 45.08
  AlK    10.25  9.86
  SiK     5.15  4.76
   PK     0.42  0.36
   SK     0.65  0.53
  TiK    19.90 10.78
   VK     1.04  0.53
  CrK     0.60  0.30
  MnK     1.11  0.52
  FeK    24.40 11.34
  NiK     0.66  0.29
  CuK     0.96  0.39
```

Figure 2.24. Spectra regarding the chemical composition determined by EDX analysis of particles FeTi30

Following EDX analysis for chemical composition determination, we may observe that particles in granular shape of ferroalloys contain high concentration carbon (C approximatively 7%) and the surface is highly oxidated (high content of oxygen, approximatively 28%).

2.1.2.3. Technological properties determination

Technological properties of granular particles are:

a) Friability;
b) Apparent density of the reinforcement;
c) Fluidity.

a) Friability of granular materials

The intrinsic property of friability is specific to solid materials undergoing mechanical properties of mixing, rolling, mechanical friction that materialized by loss in weight and formation of loss which became waste and if it is not harness represents loss for manufacturer and/or user.

Manufacturers sell ferroalloys on granulometric classes: 15 – 20 mm; 35 – 40 mm and 50 – 100 mm. Industrial practice has shown that for ferroalloys with lower granulation, the loss resulted is larger and for those with larger chunks, the loss resulted is more reduced – table 2.6.

Table 2.6. Losses by crushing chunks of ferroalloys

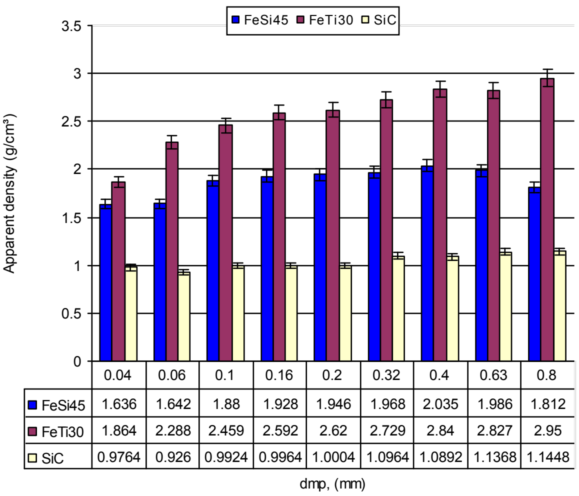
No.	Friability	Granulation of granular materials , [mm]	
		15 - 20	35 – 40
1	Losses by crushing on route production – transport for FeSi45, FeTi30	< ~ 8 %	< ~ 2%

According to the international standard, friability tests are provided for materials from metallurgical processing field, which highlights the materials' capacity to crumble on a scale from 1 to 6.

International standard ASTM, Euronorm, SR ISO includes, for every material undergoing process manipulation – transport, a guide which the final user (steel plant) may consult.

b. The apparent density of the filler;

Apparent density ρ_a, in g/cm^3, is calculated by making the ratio between mass M of paticles from container, in g, and volume container, of 25 cm^3. The arithmetic average of the three determinations is calculated.



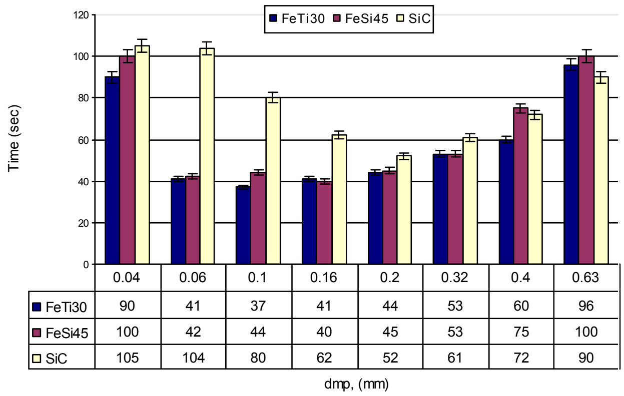
	0.04	0.06	0.1	0.16	0.2	0.32	0.4	0.63	0.8
■ FeSi45	1.636	1.642	1.88	1.928	1.946	1.968	2.035	1.986	1.812
■ FeTi30	1.864	2.288	2.459	2.592	2.62	2.729	2.84	2.827	2.95
□ SiC	0.9764	0.926	0.9924	0.9964	1.0004	1.0964	1.0892	1.1368	1.1448

Figure 2.26. The variation of apparent density depending on the size of granular particles FeSi45, FeTi30 and SiC

Apparent density shows a proportional increasing with medium diameter of particles increase. As it can be seen in figure 2.26, the highest density had the particles of FeTi30 with d_{mp} = 0.8 mm, because the highest density was FeTi30 = 6.2 g/cm^3 and the lowest density was that of the granular particles of SiC with d_{mp} = 0.04 mm.

c. Fluidity of solid particles in granulare state

Fluidity of granular particles is assessed by the flow capacity with the aid of a calibrated funnel (SR ISO 4490:2000) [299]. Fluidity is characterized by time, in seconds, in which a certain quantity of particles passes through a calibrated orifice, by a certain size of a standard funnel (Hall device).



	0.04	0.06	0.1	0.16	0.2	0.32	0.4	0.63
■ FeTi30	90	41	37	41	44	53	60	96
■ FeSi45	100	42	44	40	45	53	75	100
□ SiC	105	104	80	62	52	61	72	90

Figura 2.29. Variation of flow time of granular particles of FeSi45, FeTi30 and SiC

For granular particles of FeSi45 and FeTi30 a progressive increase can be seen in the discharge time due to the polygonal type or spherical shape of the particles. For granular particles of SiC, we can observe semnificative difference between medium diameters because elongated forms or accicular induce a random behavior.

Characterization techniques of composite materials in liquid or solid state

For the characterization of properties of composite materials the following methods have been employed:

- Thermal properties determinations (DSC analysis with calorimeter DSC823e Mettler Toledo);
- Micro hardness determination with microhardness testing device Vickers PMT3;
- Determination of morphological aspects by optic and electron microscopy (SEM) and chemical analysis of composite materials (EDX)brand FEI QUANTA 200;
- Determination of structural properties (XRD) with diffractometer DRON 3;
- Measuring dynamic viscosity (rotative method with Brookfield viscometer);
- Measuring superficial tension and contact angle (sessile drop method);

2.3. Partial conclusions

1. The chemical composition of solid materials FeSi45, FeTi30 and SiC was determined in order to evaluate the influence of some elements from chemical composition of these granular materials on metallic matrix (aluminium alloy A6061) on obtaining temperature, which may influence the embedding of particles inthis metallic matrix, as well as their distribution into solidified material.

2. According to the current standard regarding friability of materials FeSi45, FeTi30 and SiC, these have been assigned in the high friability class with the corresponding loss.

3. From the diversity of shapes of granular particles, we can conclude that ferroalloys FeSi45, FeTi30 are predominantly polygons and spherical, and SiC present an a needle like shape.

4. From all determined classes, of interest for the present research theme have been those corresponding to averagediameter of particle 0.8 mm, 0.4 mm and 0.04 mm for FeSi45, FeTi30 and SiC.

5. It was carried out a classification of particles according to free spaces between granules and their permeability, according to influence on infiltration capacity of liquid alloy through these intergranular spaces.

6. Determination of the thikness of particle layer is necessary for the prediction of the possibility of infiltration of aluminium alloy. It resulted that the greater the layer of granular particles and the smaller the size, the harder is to achieve a satisfactory yield of embedding (above 50%).

7. The surface and volume of particle, number of particles per gram and the lenght of the channel filled with liquid alloy were determined with the purpose of appreciating yield of infiltration of aluminium alloy between particles.

8. For the ideal case of spherical form of a particle, the diameter of interparticulate void was determined for three different situations: arrangement with three particles – by two methods, arrangement with four tangents particles and arrangement with four distanced particles.

9. The apparent density of filling, filling compactity and filling porosity were also determined, taking into account the nature of particles and the diameter of granular particles, and it resulted that the best infiltration of melted alloy is carried out among SiC particles because they have the lowest filling porosity at d_{mp} = 0.8 mm. All these properties can be explained by plated and needle – like shape of particles.

CHAPTER 3

EXPERIMETAL RESEARCH FOR OBTAINING COMPOSITE MATERIALS WITH METALLIC MATRIX OF ALUMINIUM ALLOY (A6061) AND GRANULAR PARTICLES OF FeSi45,FeTi30 AND SiC, BY STIR CASTING VORTEX METHOD

Within the frame of experimental research, starting from the current technologies of obtaining composite materials, an installation of mechanical mixing called Stir Casting Vortex was designed and manufactured, with the purpose to achieve high yields of embedding of refractory granular particles in systems A6061/FeSi45, A6061/FeTi30 and A6061/SiC.

Granular particles used as dispersed phase in composite materials have different dimensions, from the finest with d_{mp} = 0.04 mm to those with d_{mp} =0.8 mm.

3.1. Motivation and experimental plan

In the experimental plan for obtaining composite materials presented in table 3.1, are the main characteristics of samples of composite materials and the operating parameters applied in the process of obtaining composites from analysed systems.

Aluminium alloy A6061, during heating time up to 700^{o}C, leads to the formation of a quantity of slag as a result of melt oxidation, but the respective slag was placed in the upper part where it was solidified once with composite materials resulted. It has been assesed that an approximately even quantity of slag resulted on all samples because the obtaining for all samples took place in the same conditions of temperature, time, elaboration equipment and quantity of aluminium alloy from elaboration batch.

Calculation of batch has taken into consideration a total quantity of materials of 200 g from which 33% (mass percentage), respectively 50g have been granular particles.

It was taken into account the quantity of non-embedded particles in sample of composite materials for determination of yield of embedding η:

$$\eta = \frac{m_{imp} - m_{nmp}}{m_{imp}} \cdot 100 \quad , (\%) \qquad (3.1.)$$

where: η – yield embedding, (%); m_{imp} – initial mass of particles, (g); m_{nmp} – non-embedded mass particles, (g)

Table 3.1. Experimental program for obtaining composite materials

Composite materials	Cod sample	Composition load				Yield of embbeding η (%)	Temperature T (^{o}C)	Time t (min.)	Rotation speed v (rot/min)
		Mass of aluminium A6061 m (g)	Initial mass of particles m_{pi} (g)	Medium diameter of particles d_{mp} (mm)	Non-embedded particles mass m_{nmp} (g)				
A6061/FeSi45	A_1	150	50	0.8	-	-	700	10	420
	A_2	150	50	0.63	-	-	700	10	420
	A_3	150	50	0.40	-	-	700	10	420
	A_4	150	50	0.32	-	-	700	10	420
	A_5	150	50	0.20	-	-	700	10	420
	A_6	150	50	0.16	-	-	700	10	420
	A_7	150	50	0.10	-	-	700	10	420
	A_8	150	50	0.056	-	-	700	10	420
	A_9	150	50	0.04	-	-	700	10	420
	A_{10}	150	50	<0.04	-	-	700	10	420

continuation 1

A6061/FeTi30	B_1	150	25	0.8	-	-	700	15	170
	B_2	150	50	0.8	-	-	700	30	170
	B_3	150	50	0.4	-	-	700	30	170
	B_4	150	50	0.04	-	-	700	30	170
A6061/SiC	C_1	150	50	0.8	-	-	700	15	150
	C_2	150	50	0.4	-	-	700	15	150

3.2. Experimental installation for obtaining composite materials with metallic matrix of aluminium alloy and dispersed phases

Mechanical mixing method called Stir Casting Vortex was applied using an installation designed and manufactured by the author of the thesis, presented in figure 3.1, which has allowed the introduction of reinforcements (refractory particles) such as ferroalloys (FeSi45 and FeTi30) and silicon carbide supplied in the vortex area created in liquid melt.

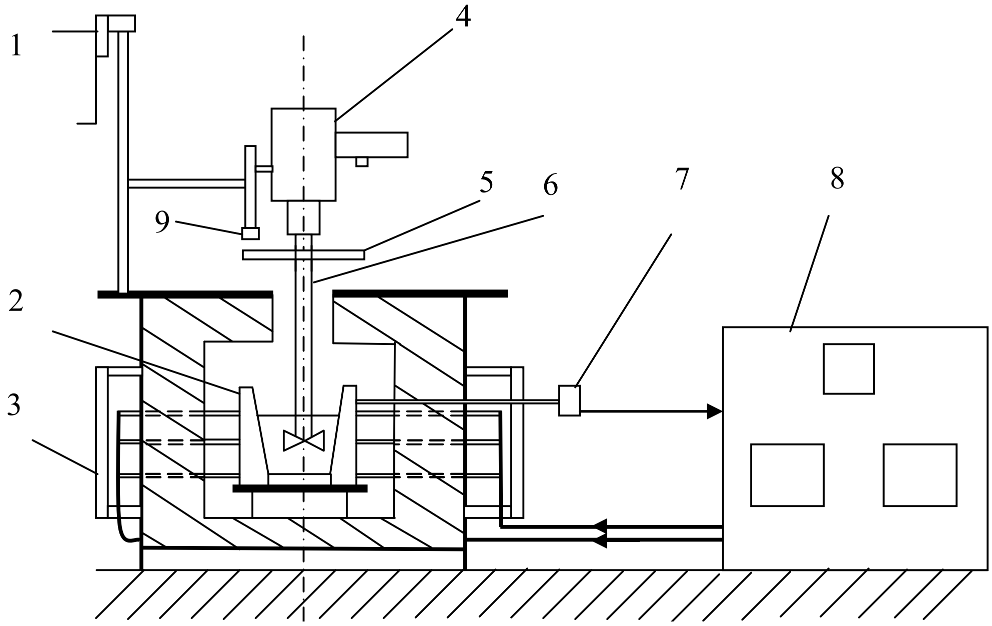


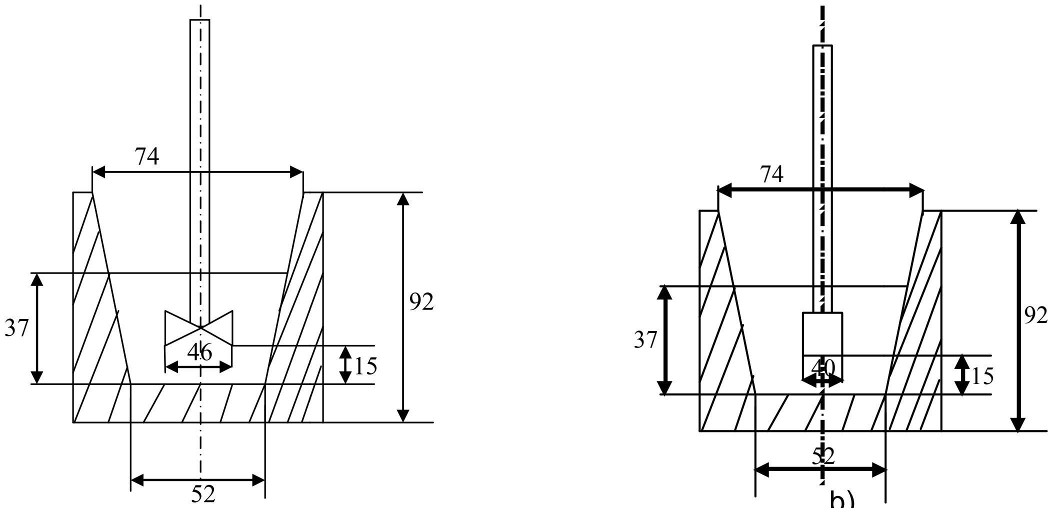
Figure 3.1. Mechanical mixing installation scheme, Stir Casting Vortex method [291]

1 – handling system of mixing device of metal bath; 2 – metallic crucible; 3 – heating furnace; 4 – shaking device; 5 - disc equipped with magnetic sensor; 6 – port propeller shaft; 7 – temperature measuring system (thermocouple Cr – Al); 8 – power source equipped with temperature regulator; 9 –speed measuring device

Technical data about installation of elaboration with mechanical mixing method Stir Casting Vortex are:

Agitation system: P_{max} = 1100 W
n = 0 …800 rpm
Electrical heating: heating elements represented by forced bars of SiC
R = 4 Ω
Power = 0 ...3,6 kW
Automatic temperature control
- PID – 1RT96;
- temperature sensor - sondă K (Cromel - Alumel);
- temperature range 0 ... 900°C,
- System for measuring rotational speed of agitator: microcontroller with Hall sensor

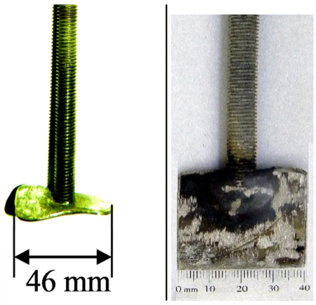
- Batch: volume max. 1.05 dm^3, mass max. 0.7 kg.



a) b)

Figure 3.3.a), b) Dimensions of metallic crucible and stirrer

In the frame of research, two types of blades with forms and different sizeshave been tested (see figure 3.3). În figure 3.4 is showed the attack angle measurement of propeller. Also, the speed measuring device with magnetic sensor is presented in figure 3.6.



a) b)

Figure 3.4. Mixing blade: a) propeller type (stirer), b)simple paddle

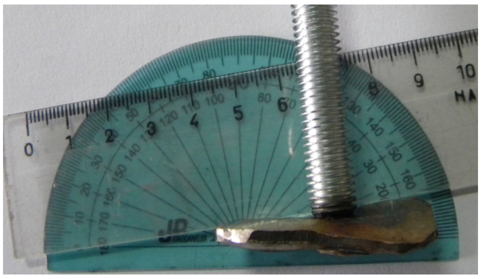


Figure3.5. Measuring device of angle attack of propeller

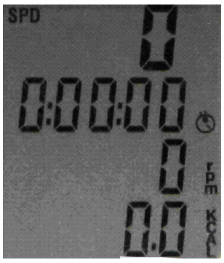


Figure 3.6. Measuring device with magnetic sensor of speed

Operation mode of experimental installation Stir Casting Vortex is schematically presented in figure 3.7.

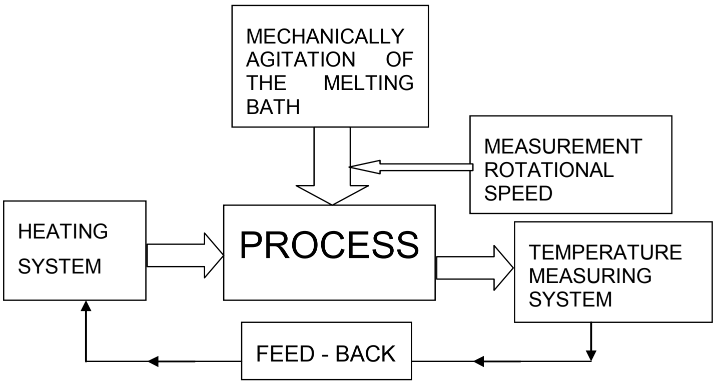


Figure 3.7. Block diagram of experimental installation Stir Casting Vortex [291]

3.3. Obtaining composite material from system A6061/FeSi45

Designing and manufacturing experiments followed to highlight the incorporation in metallic matrix of aluminium alloy A6061 of refractory particles, especially ferroalloys with medium diameter contained in different granulometric classes. Thus, there were varied dimensions of ferroalloys particles FeSi45, FeTi30 and SiC, maintaining other constant parameters: temperature, mixing time, rotation speed, blade shape and chemical composition of aluminium alloy.

Table 3.2. Conditions of obtaining composite materials from system A6061/FeSi45

Cod sample	Composition load				Yield of embbeding η (%)	Temperature T (^oC)	Time t (min.)	Rotation speed v (rot/min)
	A6061	FeSi 45						
	Mass of aluminium A6061 m (g)	Initial mass of particles m_{pi} (g)	Medium diameter of particles d_{mp} (mm)	Non-embedded particles mass m_{nmp} (g)				
A_1	150	50	0.8	30	40	700	10	420
A_2	150	50	0.63	30.5	39	700	10	420
A_3	150	50	0.40	31	38	700	10	420
A_4	150	50	0.32	31.25	37.5	700	10	420
A_5	150	50	0.20	32	36	700	10	420
A_6	150	50	0.16	32.15	35.7	700	10	420
A_7	150	50	0.10	32.75	34.5	700	10	420
A_8	150	50	0.056	33.5	33	700	10	420
A_9	150	50	0.04	33.6	32.8	700	10	420
A_{10}	150	50	<0.04	34.50	31	700	10	420

Experimental research presented in literature has shown that at composite materials obtained by mechanical mixing Stir Casting Vortex embedding regime has been suitable.

3.4. Characterization of composite material from system A6061/FeSi45 obtained by Stir Casting Vortex

For the characterization of composite materials with granular particles of FeSi45 obtained by mechanical characterization by Stir Casting Vortex method the following investigation methods are used:

Chemical analysis EDX and XRF, which highlighted chemical composition modification at metallic matrix interface and granular particles. This type of analysis was performed selectively on representative samples;

The metallographic method, which highlighted aspects from sample field such as: particle dispersion, the influence of mixing method on process of obtaining composite materials and pore which are forming.

Analysis of structure by electron microscopy (SEM) to highlight microstructure of studied composites.

3.4.1. Chemical analysis made on sample surface of composite material A6061/FeSi45 by EDX method

For the composite material sample the result of chemical composition analysis made by electron microscope (SEM), EDX method is presented, in figure 3.12 and in table 3.3.

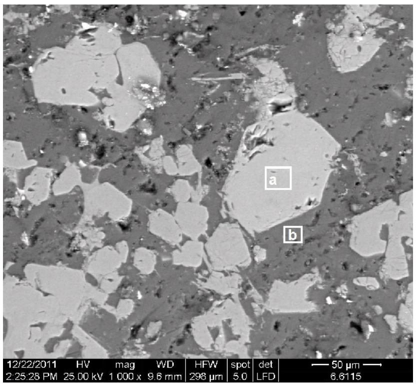
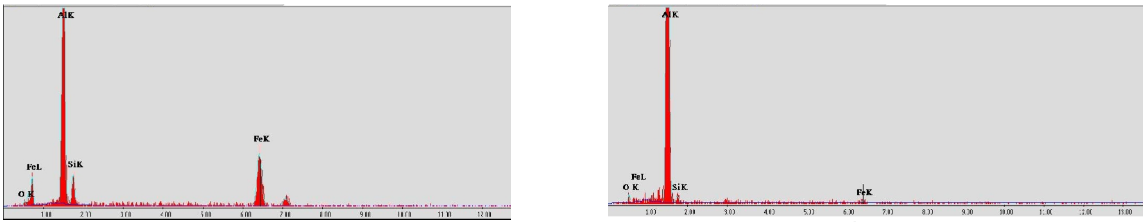


Figure 3.13. SEM image of composite material A6061/FeSi45 with d_{mp} = 0.4 mm



a b

Figure 3.14. Spectra a, b which give results for elemental analysis EDX, for composite material A6061/FeSi45

Table 3.4. Chemical composition of composite material A6061/FeSi45

No.	Analysed area	Chemical composition (%)			
		Al	Fe	Si	O
1	Particle (a)	60.88	26.57	10.6	1.95
2	Metallic matrix (b)	90.36	1.43	3.55	4.65

From figure 3.13, 3.14 and table 3.4 we may observe the following: the chemical composition from the particle highlights the presence of aluminium in 60.88% concentration (mass percent). Mechanical mixing by Stir Casting Vortex method, at temperature (700°C) and 10 min mixing time. has favoured the total wetting of granular particles by the aluminium alloy and by bidirectionate diffusion mechanism it can explain the raised concentration of aluminium in particle area. Also, we may observe the presence of oxygen in 1.95% concentration (mass percent), a low concentration resulting in a layer of aluminium oxide with thickness reduced on the edge of the particle.

3.4.3. Macrostructural analysis of composite material samples A6061/FeSi45 obtained by Stir Casting Vortex method

Macrostructural analysis aims to highlight on macro scale aspects related to particles distribution in metallic matrix, different shapes of particles, highlighting inherent defects of obtaining process in this case pores, diffusion as formation mechanism of linkage between matrix – particles.

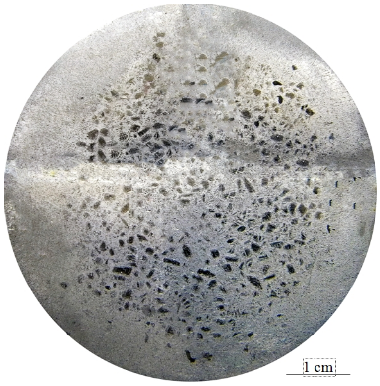


Figure 3.15. Macrostructure probes A_1 of composite materials A6061/FeSi45 with d_{mp} = 0.8 mm

In figure 3.15 a transverse section through sample A1 (A6061/FeSi45 with particles with d_{mp} = 0.8 mm) is presented. Particle mass was embedded by Stir Casting Vortex method, the section was prepared by mechanical cutting, grinding and polishing. We may observe the non-uniform distribution of particle on the transversal section. Mixing particles is difficult to achieve on the edge because of high viscosity of metallic melt and wall effect on mixing.

In the central area we may observe a uniform distribution of particles and an orientation of particles on eddy direction, on the bases of minimum resistance at mechanical mixing of liquid and due to the fact that particles are pushed towards the centre of sample by the solidification front. In the case of dendritic structure, the particles are trapped between secondary or tertiary branches. Also, in the central area we may observe a slight decrease in particles density due to the centrifugal effect.

3.4.4. Microstructural analysis of composite material samples A6061/FeSi45 by optical microscopy

By microstructural analysis samples from composites A6061/FeSi45 were sampled, and they were prepared by grinding and polishing in sample preparation laboratory of “Dunărea de Jos” University of Galați. There were highlighted polygonal grain of grey color of solid solution α (of Si in Al) which can be at interfaces, possible intermetallic compounds of $FeAl_3$ and particles of FeSi45 which can have a polygonal or spherical aspect and crystals acicular which may be of Si or eutectic (α + Si).

Microstructural analysis shows that, although particles have initial certain dimensions some of them have been crumbled and reduced down to appropriate sizes.

In figure 3.16 – 3.21 there were highlighted microstructures and micrography composite material A6061/FeSi45 in which granular particles of FeSi45 with d_{mp} = 0.04 mm, 0.4 mm and 0.8 mm have been embedded in metallic matrix of aluminium alloy.

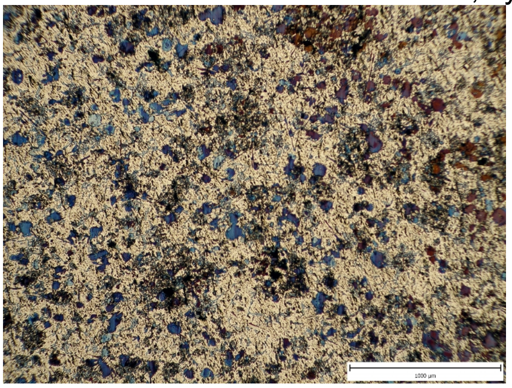


Figure 3.16. Microstructure of composite materials A6061/FeSi45, Stir Casting Vortex method (10 min) and d_{mp} = 0.04 mm (attack 10% HF)

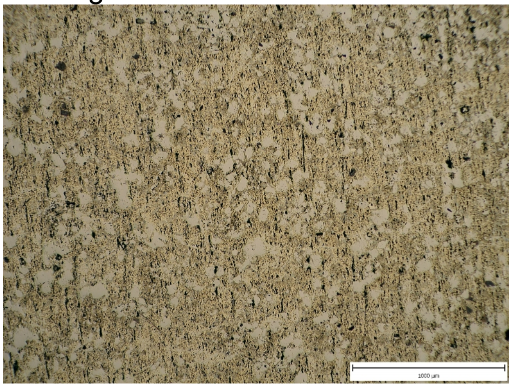


Figure 3.17. Micrograph of composite material A6061/FS45, Stir Casting Vortex method (10 min) and d_{mp} = 0.04 mm (without attack)

Fine granular particles of FeSi45 with d_{mp} = 0.04 mm embedded in metallic matrix of composite materials obtained by Stir Casting Vortex method present the following aspects: homogenous distribution on the entire surface, polygon-shaped particle presence, homogeneity of granules sizes, lack of local clusters and a reduced degree of pores on surface, as evidence of fast homogeneization.

3.5. Obtaining of composite materials from system A6061/FeTi30

Research regarding the obtaining and characterization of composite A6061/FeTi30 are similar with those for A6061/FeSi45. Composite material was obtained using exterior mechanical energy.

Load composition and experimental regimes on obtaining composite material from system A6061/FeTi30 are presented in table 3.6.

Table 3.6. Conditions of obtaining of composite material A6061/FeTi30

	Composition load							
	A6061	FeTi30						
Cod sample	Mass of aluminium A6061 m (g)	Initial mass of particles m_{pi} (g)	Medium diameter of particles d_{mp} (mm)	Non-embedded particles mass m_{nmp} (g)	Yield of embbeding η (%)	Temperature T (^oC)	Time t (min.)	Rotation speed v (rot/min)
B_1	150	25	0.8	20	20	700	15	170
B_2	150	50	0.8	31.5	37	700	30	170
B_3	150	50	0.4	32.5	35	700	30	170
B_4	150	50	0.04	35.5	29	700	30	170

Following the process of obtaining by Stir Casting Vortex method on all working regimes, the composite material was formed. For granular particle FeTi30, with d_{mp} = 0.8 mm and maintaining time of elaboration for incorporation of particle was 15 minute, respectively 30 minute, and a significant modification of chemical composition was observed. Mixing time increases contact time between particle and aluminium alloy, and thus increases the surface where the diffusion process of aluminium in particles may take place.

3.6. Analysis of composite materials from system A6061/FeTi30

For the analysis of composite material the following investigation methods were used:

- Chemical analysis EDX and XRF, for inhomogeneity highlight of chemical composition.

- Microscopical analysis, for highlighting of different aspects in sample field: homogenous distribution, appearance of pores, dispersion of particles, significant influence of obtaining method of composite materials.
- Microscopic analysis SEM, for highlight chemical compound newly formed.

3.6.1. Chemical analysis of samples surface of composite material A6061/FeTi30 by EDX method

For sample of composite material from figure 3.24 (table 3.7), analysis of chemical composition was pursued.

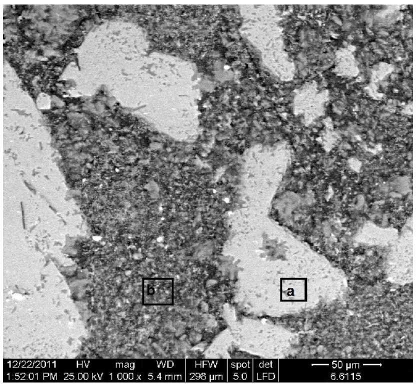
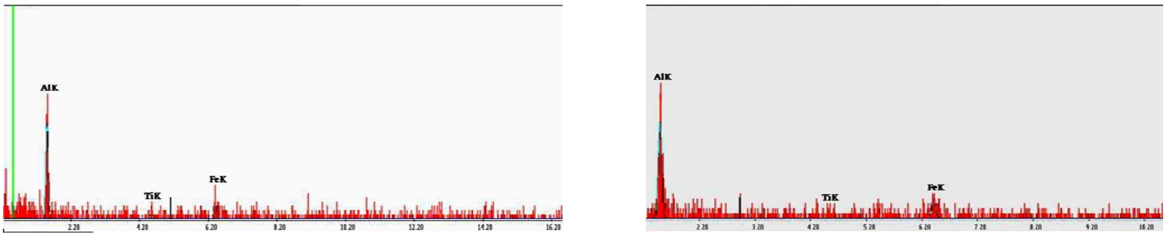


Figure 3.25. SEM imagine of composite material A6061/FeTi30 with d_{mp} = 0.8 mm



a b

Figure 3.26. Spectra a, b which shows EDX analysis, in two points, for composite material A6061/FeTi30

Table 3.8. Chemical analysis of area analysed of A6061/FeTi30

No.	Analysed area	Chemical composition (%)		
		Al	Fe	Ti
1	Particle (a)	61.75	30.13	8.13
2	Metallic matrix (b)	63.96	29.81	6.23

From figure 3.25, 3.26 and table 3.8 we may observe variation of chemical composition for chemical elements aluminium, iron and titanium, presented in the area analysed on the particle and from matrix area.

In the particle area, for aluminium we can observe a high concentration, of 61.75% (mass percent) due to the process of mechanical mixing by Stir Casting Vortex method, which has created conditions for total wetting and for bidirectional diffusion at the edge.

Also, we can observe close values of aluminium concentration, either due to analysis method – made on reduced area, or because obtaining time was sufficient for diffusion to be produced in depth (close values of aluminium on matrix and on particle).

3.6.4. Microstructural analysis of sample of composite material A6061/FeTi30 by electron microscopy SEM

Electron microscope SEM Fei Quanta 200 was used for the analysis. Determination highlighted some details of sample surface which may be observed by optical microscopy. The analysed surface was prepared by grinding and polishing. A special attention was granted to sample preparation in order to avoid pulling out particles and formation of scratches on surface sample.

Surface preparation was most carefully performed for avoiding to destroy details of characteristic structure of matrix of aluminium alloy with high deformability and hard dispersate particles with shapes and dimensions characteristics.

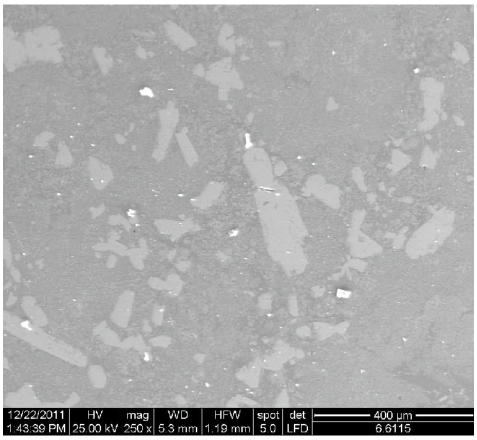


Figure 3.29. SEM imagine of composite material A6061/FeTi30, Stir Casting Vortex method, t = 30 min and d_{mp} = 0.8 mm

In figure 3.29 we may observe the distribution of granular particles FeTi30. Even if in figure were found in a small quantity, we cannot observe formation of clusters of particles and pores.

3.7. Obtaining composite material from system A6061/SiC

Obtaining composite materials from system A6061/SiC was made by embedding a known mass of granular particles in the metallic matrix. With the help of an external force of mechanical nature was induced in liquid melt and particles have been embedded in that.

The experimental program for obtaining the composite material A6061/SiC comprises data and conditions is shown in table 3.10.

Table 3.10. Obtaining conditions of composite material A6061/SiC

Code sample	Composition load				Yield of embbeding η (%)	Temperature T (^oC)	Time t (min.)	Rotation speed v (rot/min)
	A6061	SiC						
	Mass of aluminium A6061 m (g)	Initial mass of particles m_{pi} (g)	Medium diameter of particles d_{mp} (mm)	Non-embedded particles mass m_{nmp} (g)				
C_1	150	50	0.8	32.5	35	700	15	150
C_2	150	50	0.4	34.5	31	700	15	150

Yield of embedding of granular particles with d_{mp} = 0.8 mm was 35% and for particles with d_{mp} = 0.4 mm to 31%.

Low yields are consequence of a complex elaboration process in which several overlapped factors are conjugated: geometrical factors, roughness of the wall (wall effect), blade form of mechanical mixer, distance from the blade to the wall, distance from the bottom of crucible to the wall (bottom effect); thermodynamic factors depending on temperature which varies during processing time from the wall towards the centre, in the upper plane of slag by increasing the contact area due to the generation of vortex; based on the temperature difference; generation of convective currents within the melt; the effect of the atmosphere (moisture) on the aluminium alloy; preheating effect; shape and type of edges; the surface roughness of the particles to contact with a superheated melt.

3.8. Analysis of composite materials from system A6061/SiC

For the analysis of composite material the following investigation methods were used:

- Chemical analysis EDX, to highlight chemical composition inhomogeneity;
- Chemical elements distribution using x-ray spectrometry method (EDX);
- Microscopic analysis which highlights the following details in the field of sample: homogeneous distribution, pores appearence , dispersion of particles, significant influence of method of obtaining composite material;
- Microscopical analysis (SEM) for detailed analysis of areas of newly formed chemical compounds.

3.8.1. Chemical analysis of surface sample A6061/SiC by x-ray spectrometry (EDX)

Chemical analysis was carried out in the matrix metal area, interface area and particle area (table 3.11). In figure 3.33 (a, b, c) are presented spectra which show chemical composition variation in the three area investigated of composite material A6061/SiC.

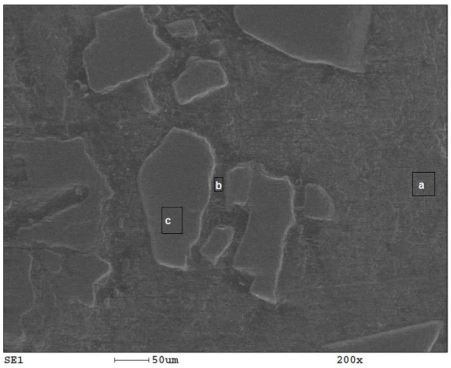
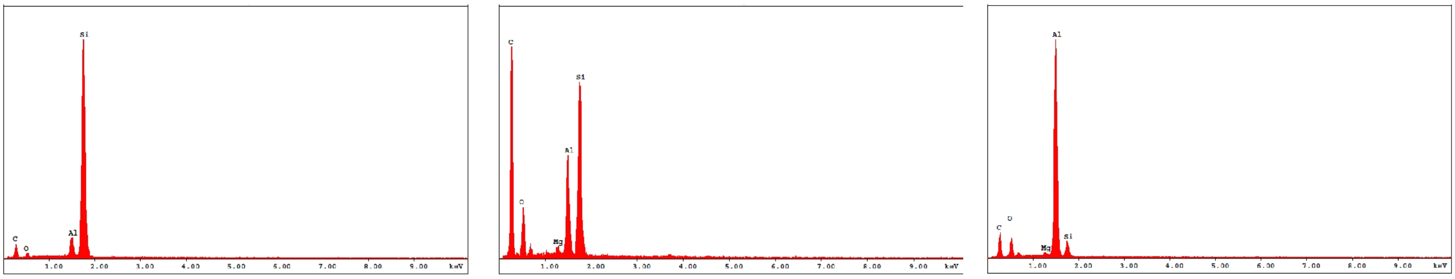


Figure 3.32. SEM imagine of composite material A6061/SiC with $d_{mp} = 0.8$ mm



a b c

Figure 3.33. Spectra a, b, c regarding EDX analysis in three points for composite material A6061/SiC

Table 3.11. Chemical composition in three different areas of composite material A6061/SiC

No.	Analysed region	Chemical composition (%)				
		Al	Si	C	O	Mg
1	Metal matrix A6061 (a)	43.71	4.38	40.83	10.42	0.56
2	Interface (b)	6.48	13.96	68.15	10.96	0.45
3	Particles SiC (c)	4.38	52.17	40.40	2.84	0.21

From figure 3.33 we may observe the following: chemical composition on particle revealed the presence of aluminium in 4.38 wt% concentration, content possible due to conditions of elaboration by Stir Casting Vortex method (700^{o}C) and mechanical mixing time (15 min.) when the possibility of bidirectional transfer is created and there is also the possibility of formation of compound Al_4C_3 as a result of diffusion process of C from particles of SiC, the molten aluminum wets the particles, thus favouring their embedding.

Compound Al_4C_3 can be found in area from interface when they are provided conditions for its formation, respectively temperature and maintaining time.

3.8.2. Chemical elements distribution using x-ray spectrometry (EDX)

Chemical elements distribution was achieved using features of electron microscope Fei Quanta 200, with the help of a program by which a map based on color tones was drawn. Thus, it was able to highlight homogeneity of the presence of chemical elements on analysed surface.

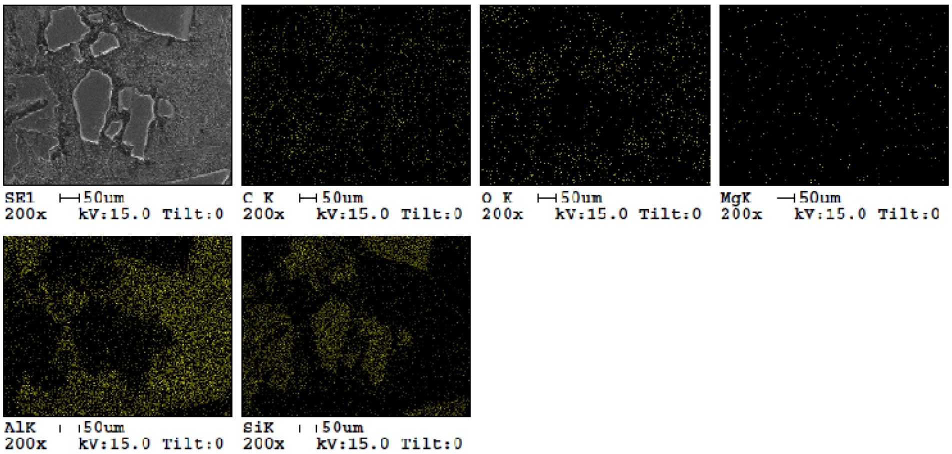


Figure 3.34. Chemical elements distribution for composite material A6061/SiC

Composite material show area where aluminium is found in high concentration and area where chemical elements of particles of SiC are preponderant, accordinaly to figure 3.34.

Silicon is present both in the metallic matrix (in 0.8 % concentration according to chemical composition of A6061) as well as in particulate (70% according to chemical composition of SiC), and chemical elements distribution revealed silicon in all analysed area with a high concentration on the particle.

Oxygen seems to have a uniform distribution in metallic matrix of aluminium alloy, following the presence of Al_2O_3, and less on the particle when oxygen can be found under the SiO_2 form.

3.8.3. Macrostructural analysis of composite material for sample A6061/SiC

Macrostructural analysis is a common method for the study of composite materials for surface with submilimeter dimensions of particles obtained by mechanical mixing. Figure 3.35 presents a vertical section through sample of composite material A6061/SiC.

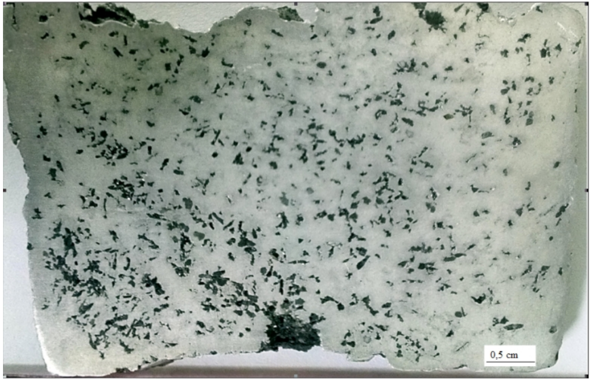


Figure 3.35. Macrostructural sample of composite material A6061/SiC, with d_{mp} = 0.8 mm

Macrostructure from figure 3.35 presents an overview of distribution of SiC particles with d_{mp} = 0.8 mm, in mass aluminium alloy due to shape and speed propeller. Continuous mechanical mixing causes the formation of areas with a high number ofparticles,areas with lower concentration and also areas with a more even distribution. Also, pores can be observed in the bottom parts – a defect resulted of bottom effect where mechanical mixing process was minimized.

3.8.5. Microstructural analysis of sample surfaces of composite material A6061/SiC by electron microscopy (SEM)

Microstructural analysis was pursued on sample of composite material obtained by Stir Casting Vortex method according to the experimental program, and figure 3.38 presents the image of the analysed sample.

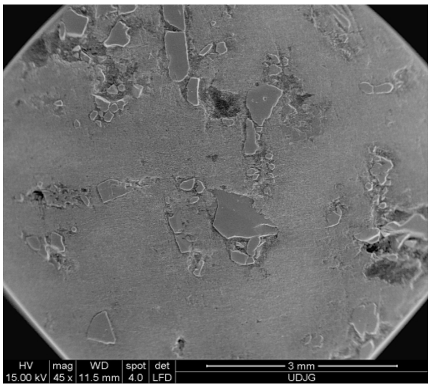


Figure 3.38. SEM imagine of sample composite material A6061/SiC, for d_{mp} = 0.8 mm

Analysing sample microstructure from figure 3.38 we can make the following observation: the distribution of particles is non-uniform and agglomerations of particles are highlighted. With the help of scale printed on the image the dimensions of particles were estimated, these varying from values <0.2mm and up to 0.8 mm. In addition to determining the dimensions, this method is essential for observing the shapes of particles which can modify in the time of mixing, especially because of mechanical stirring when particles can crumble or can agglomerate, as apparent in figure 3.38.

3.9. Partial conclusions regarding obtaining composite materials A6061/FeSi45, A6061/FeTi30 and A6061/SiC

1. Obtaining method of composite material with technological role, by embedding of some granular particles in an metallic matrix by Stir Casting Vortex method, is a promising method, with reduced costs, which does not involve important investement. This method can also represent an alternative solution to other costly methods of ecology of production spaces and storage.
2. An installation for obtaining composite materials by Stir Casting Vortex method was designed and manufactured. The method is is based on mechanical mixing of aluminium alloy melted and introduction portioned of refractory granular particles in vortex created by a stirrer. The shape of propeller and rotational speed significantly favours yield of embedding by creating vortex in central area of crucible, which is the place where maximum effect of mechanical energy is ensured.
3. Experimental research was made by testing two types of blades respectively blades type propeller with 10^{o} angle and simple paddle. Each of the two presents specific advantages.
4. For an appropriate embedding, three times of elaboration were tested: 10 min for A6061/FeSi45, 30 min for A6061/FeTi30 and 15 min for A6061/SiC. Obtaining temperature was set at $700^{o}C$ with an overheating approximately $40^{o}C$ with melting temperature of aluminium alloy. In the case of rotation speed, three rotation speeds, 150, 170 and 420 rpm, were tested, and it was observed that the highest rotation speed, 420 rpm, creates the most suitable conditions for a good embedding.
5. In conditions of temperature, time, speed of rotation and propeller shape (fixed) were obtained yield of embedding for particles with medium diameters: 0.8 mm, 0.4 mm și 0.04 mm between $30 \div 40$ % for A6061/FeSi45, between $20 \div 39$% for A6061/FeTi30 and between $32 \div 35$ % for A6061/SiC. These low yields of embedding are the result of a complex obtaining process in which the following factors are overlapped: variation of obtaining temperature from the wall of crucible towards mixer,increasing the contact surface by creating vortex and increasing of slag quantity, difference of temperature matrix/particle due to low thermal conductivity, shape and type of edges, the surface roughness of the particles; geometrical factors, roughness of the walls (wall effect) and blade form of mechanical mixer.
6. Determination of chemical composition by XRF method revealed a significant variation of chemical composition in function of analysed area and of number of granular particle in the condition of an area of 10 mm.
7. Determination of chemical composition by EDX method has shown that the greeater the amount of granular particles, the more changes occur in the chemical composition; being in the area of the intermediary values of matrix and granular particles.
8. Determination of chemical composition by EDX method on low areas on the particles, at interface particle/matrix and in metallic matrix area shows a variation of chemical composition due to physico-chemical processes which take place at obtaining composites by mechanical mixing and localized diffusion around particles.
9. On macrostructural level, there was observed a dispersion of particles on surface composite material, correlated with the type of the mixer immersed in the melt, observing the effect of centrifugal. Thus, are emphasized areas with a high number of particles, areas with lower concentration and also areas with a more even distribution.
10. Microstructures of samples of composite material, obtained by Stir Casting Vortex method, present aspects which depend on particle distribution, on their condition, due to obtaining conditions, or their agglomeration. On their surface a high concentration of adsorbed gases is found, which can reduce the positive effect of complex deoxidation by rising hydrogen content in steel. For composite material to be useful and effective in deoxidation process, metallic matrix/granular particles is not desirable to form new intermediate phase at interface.

11. Obtaining process of composite materials by Stir Casting Vortex method must be conducted to ensure a rapid mixing to avoid pronounced segregation on height of solidified composite material.

CHAPTER 4

EXPERIMENTAL DETERMINATION REGARDING OBTAINING AND CHARACTERIZATION OF COMPOSITES WITH MATRIX OF ALUMINIUM ALLOY (A6061) AND GRANULAR PARTICLES, BY MECHANICAL MIXING WITH VIBRATIONS

This chapter presents the results of experimental determinations regarding obtaining and characterization of composite materials with metallic matrix with the help of a new method of mechanical mixing, namely by vibrations, as well as the characterization of this materials from the point of view of chemical composition, structural and technological properties.

To this effect, the author has designed and manufactured an installation and technology for obtaining by mechanical mixing with the aid of vibrations of alloy melt with particles stacked in layers.

Elaboration technology was used for obtaining composite materials with metallic matrix from systems A6061/FeSi45, A6061/FeTi30 and A6061/SiC and were embedded particles with d_{mp} = 0.8 mm, 0.4 mm, 0.04 mm.

4.1. Motivation and experimental plan

Experimental research aimed at studying the embedding of a quantity as high of refractory granular materials of FeSi45, FeTi30 and SiC in matrix of aluminium alloy. Thus, a percentage ratio matrix/particles of 23% was established quantity embedded by method of obtaining with the help of vibrations.

Experimental conditions were fix, respectively working temperature at 730°C and 10 minutes elaboration time, as determined by preliminary tests. Smaller amounts of time do not favorably influence homogeneity and higher durations lead to appearence of mass transport by diffusion and appearence of chemical compounds, an unwanted practical situation because it can distribute at the edge of particles, thus preventing a good embedding of particles in metallic matrix.

Mechanical vibrations induce in compound system of aluminium alloy A6061 in liquid state and granular particles stacked in layers a controlled vertical displacement (by amplitude and frequency of vibrations) which creates the conditions for embedding.

The method used for mechanical mixing was based on a spatial arrangement of sandwich type. This arrangement is composed of three overlapped layers (figure 4.1). The top layer and bottom are of aluminium alloy and the third from the middle consists in granular particles of FeSi45, FeTi30 and SiC.

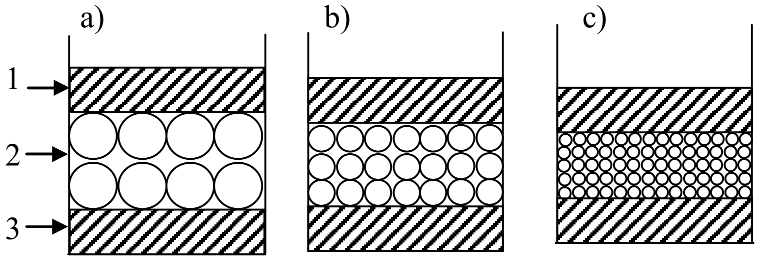


Figure 4.1. (a,b,c) Successively arranged layers (sandwich)
1 – compact layer of A6061; 2 – layer of refractory particles, respectively FeSi45, FeTi30 and SiC; 3 – compact layer of A6061

Metallic melt was superheated up to 730°C temperature, followed by mechanically mixing of molten alloy with the solid particles by vibrations of the support table.

Infiltration aluminium alloy among solid granular particles was favoured by vibration process of crucible with heterogeneous composite. Amplitude and frequency have been established according to experimental conditions.

At the beginning, the layer of particles is compact and aluminium alloy is infiltrated by intergranular spaces. The layer of particles from the interface solid-liquid is wetted and the surface is washed by oxides. During mechanical vibrations, solid particles collide on height against each other and void occurs – interstitial spaces. The diameters of intergranular spaces can have a minimum dimension, equivalent with the value of vibration amplitude of installation mass, or can have a greater value. In this intergranular spaces liquid aluminium alloy can be infiltrated and lead to homogenization of the mixture.

4.2. Experimental installation for obtaining composite materials with metallic matrix of aluminium alloy and granular particles of FeSi45, FeTi30 și SiC

Obtaining composite material by this method implies melting of the aluminum in the presence of particles, followed by an intense mechanical mixing of load from crucible by vibration.

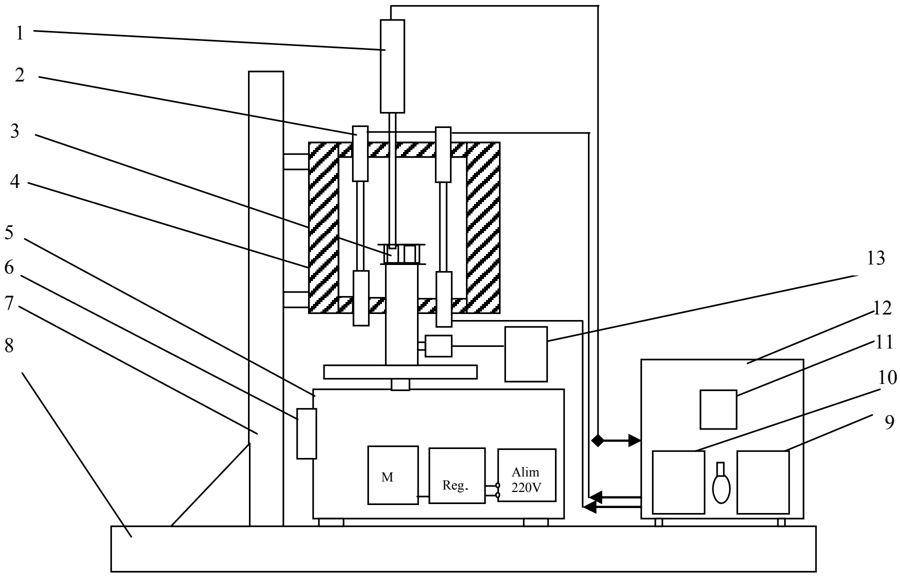


Figure 4.4. Simplified scheme of obtaining installation of composite materials with refractary particles using vibrations

1 – temperature measurement system (thermocouple); 2 – heating elements; 3 – crucible steel; 4 – electric furnace; 5 – vibrating equipment; 6 – mechanical system for adjustement of amplitude and frequency; 7 – support column; 8 – motherboard; 9 – ampere indicator; 10 – voltage indicator; 11 –temperature regulator; 12 – power source provided with temperature; 13 – vibration measuring device (amplitude and frequency) X – viber [206]

Technical data about installation of elaboration with mechanical mixing vibration method:

- Electric heating is made with heating elements represented by forced bars of SiC, with the following characteristics: R = 4 Ω; Putere = 0 ...3.6 kW
- Temperature control system: PID – 1RT96; temperature sensor – K (chromel – alumel); range 0 … 900 °C
- Equipment for generation of vibrations:
  - Total weight on the table: 20 kg;
  - Frequency of vibrations: 0 – 80 Hz;
  - Amplitude of vibrations: 0 – 5 mm;

  - System for measuring amplitude and frequency X-viber VMI 199-28.

- Batch: mass max. = 40 g

In figure 4.6 is shown block diagram for installation for obtaining composite materials.

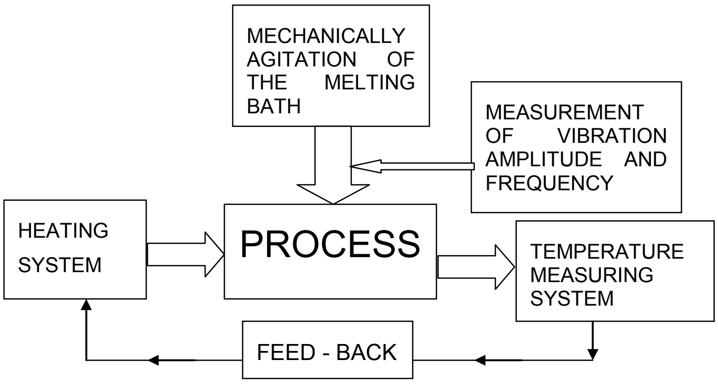


Figure 4.6. Block diagram of experimental stand based on mechanical mixing through vibration during obtaining [206]

4.3. Obtaining composite materials from system A6061/FeSi45

Aluminium alloy A6061 in heating time at 730°C has also led a to the formation of a quantity of slag as a result of melt oxidation, but the respective slag was placed in the upper part where it was solidified once with composite materials resulted.

It was determined yield of embedding η with formula 3.1.

Calculation of batch has taken into consideration a total quantity of materials of 32.5 g from which 23% (mass percentage), respectively 7.5 g have been granular particles.

Table 4.2. Variation of yield of embedding of particles of FeSi45 with d_{mp} = 0.8 mm in alloy A6061, in function of working parameters accordinaly of mixing method with vibration

Cod sample	Non-embedded mass particles (g)	FeSi45	Amplitude (m x10^{-3})	Frequency (Hz)	Speed V_E (mm/s)	Temperature (°C)	Time (min)
		Yield of embedding η (%)					
A_1	7.5	0	zero vibrations	zero vibrations	0	730	10
A_2	4.62	38.4	0.02	70	7.07	730	10
A_3	3.91	47.82	0.12	70	16.06	730	10
A_4	0.18	97.52	0.45	62	59.28	730	10
A_5	0.43	94.25	0.95	63	125.2	730	10
A_6	0.28	96.25	1.1	72	159.57	730	10
A_7	0.3	96	3.2	35	245	730	10

Maximum quantity of disperse phase embedded in metallic matrix of aluminium alloy is 97.52 % for an amplitude of 0.45 mm and a frequency of 62 Hz, figure 4.11.

From tabel 4.2 and figure 4.11, a high yield of embedding is observed, beginning from an amplitude of vibration of 0.45 mm, which suggests that intergranular void is favorable of a good infiltration of alloy which is formed relatively quickly, determining a good homogenization of alloy with granular particles.

At the same time with rising of amplitude is observed maintaining of values high of yield of embedding (94.25%, 96,25%, 96%) in metallic matrix, values which support and confirme the fact that amplitude is a fundamental parameter at embedding of a quantity as high of refractory particles of FeSi45. These phenomena are explained by the fact that the layers of particles are less compact and apparent porosity is larger.

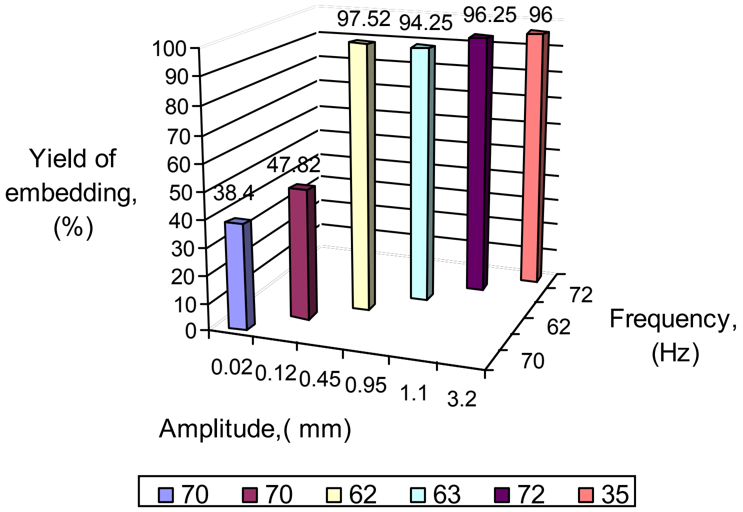


Figure 4.11. Variation of yield of embedding of particles of FeSi45 with d_{mp} = 0.8 mm, depending on frequency and amplitude

It may be concluded that, this is the field of amplitude and frequency on which embedding particles occurs.

For elaboration method with vibration was established the field of amplitudes of vibrations and of frequency for embbeding corresponding particles of FeSi45 with d_{mp} = 0.4 mm.

Table 4.3 Variation of yield of embedding of particles of FeSi45 with d_{mp} = 0.4 mm in alloy A6061, in function of working parameters accordinaly of mixing method with vibration

Cod sample	Non-embedded mass particles (g)	FeSi45	Amplitude (m x10^{-3})	Frequency (Hz)	Speed V_E (mm/s)	Temperature (°C)	Time (min)
		Yield of embedding η (%)					
B_1	0.2	97.32	0.45	62	59.28	730	10
B_2	1.47	80.38	0.7	61	83.85	730	10
B_3	0.25	96.59	0.95	63	125.2	730	10
B_4	0.24	96.72	1.1	72	159.57	730	10
B_5	0.15	97.95	3.2	35	245	730	30

Maximum quantity of particles embedded in metallic matrix of aluminium alloy (η = 97.95%), is at 3.2 mm and 35 Hz amplitude, and the graphic representation is given in figure 4.12.

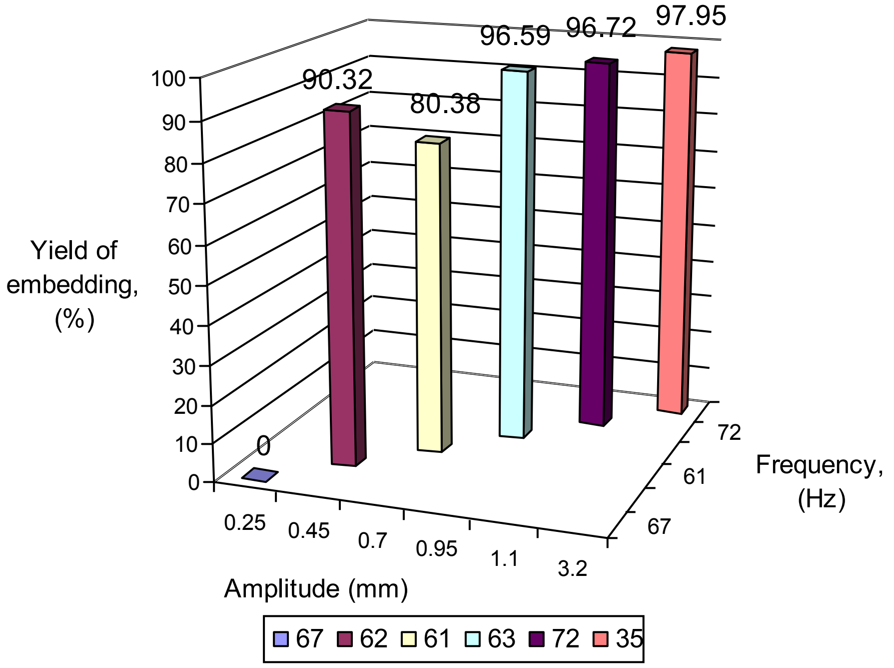


Figure 4.11. Variation of yield of embedding of particles of FeSi45 with d_{mp} = 0.4 mm, depending on frequency and amplitude

From table 4.3 and figure 4.12 it results that the field of amplitude and frequency whose yield of embedding has high values is between 0.7 mm – 3.2 mm.

This may indicate that intergranular void will assure a good infiltration of melted alloy from a greater difference of amplitude.

Saturation of liquid melt of aluminium alloy with solid particles by initial formation of bridges between particles and subsequently their growth, makes that amplitude present an important role.

High yield of embedding begins from a lower amplitude in case of particles with medium diameter of 0.8 mm, respectively from values of amplitude of 0.7 mm and 70 Hz, which confirms the importance of inter-granular void initially reduced, but with a high potential of favoring infiltration of aliquid alloy.

Experiments where amplitude is even more reduced – 0.45 mm and frequency 62 Hz show that the domain in which particles with d_{mp} is 0.4 mm may by embedded in metallic matrix can start from low amplitudes, simultaneously may be observed proportionality between amplitude and dimension of particle.

Table 4.4 Variation of yield of embedding of particles of FeSi45 with d_{mp} = 0.04 mm in alloy A6061, in function of working parameters accordinaly of mixing method with vibration

Cod sample	Non-embedded mass particles (g)	FeSi45 Yield of embedding η (%)	Amplitude (m x10^{-3})	Frequency (Hz)	Speed V_E (mm/s)	Temperature (°C)	Time (min)
C_1	4.11	45.2	0.55	62.6	62.14	730	10
C_2	4.23	43.52	0.9	62.5	119.63	730	10
C_3	6.76	9.85	1.15	56	137.5	730	10
C_4	5.67	24.28	2.1	46.9	221.2	730	10
C_5	6.92	7.7	2.7	44.5	289.63	730	10
C_6	1.50	79.9	3.2	35	245	730	10
C_7	6.52	12.95	3.6	31.9	240.66	730	10

From table 4.4 and graph fromfigure 4.13 is observed a much lower embedding compared to other granulometric classes of FeSi45, respectively up to a maximum of 80% yield of embedding. This phenomenon may be explained by the great height of particle layer which must covered by the liquid alloy.

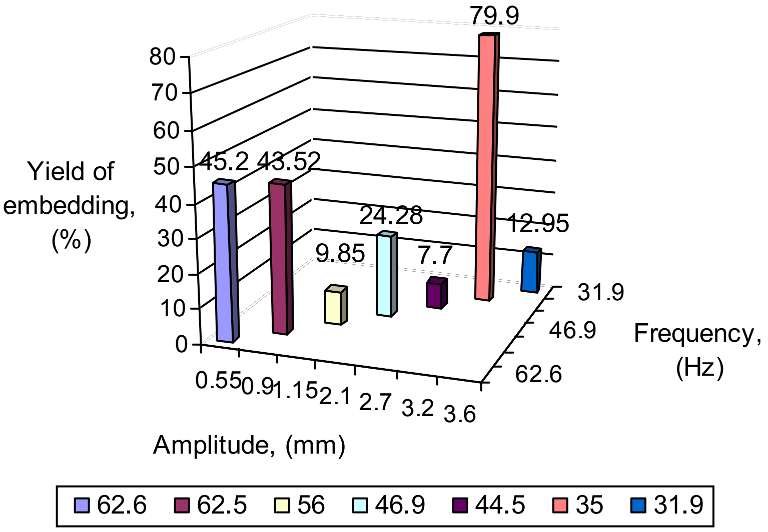


Figure 4.13. Variation of yield of embedding of particles of FeSi45 with d_{mp} = 0.04 mm, depending on frequency and amplitude

Also, a high number of particles from elaboration batches lowers the quantity of embedded particles, due to the fact that d_{mp} = 0.04 mm is very small, and yield of embedding drops very much. This aspect highlights the importance of the particles diameters which can influence yield of embedding. The higher values of yield being 79.9%, on maximum amplitude and frequency of vibration applied. Simultaneously yield of embedding of 45.2%, for amplitude relatively reduced of 0.55 mm and 62.6 Hz, reveals the possibility that those particles too may be embedded and used in composite materials with a technological role.

4.4. Characterization of composite materials from system A6061/FeSi45

For the characterization of composite materials with particles of FeSi45 obtained by mechanical mixing with vibrations the following investigations methods were used:

- Chemical analysis EDX, which highlights modification of chemical composition at interface metallic matrix – granular particles;
- Chemical elements distribution,using x-ray spectrometry method (EDX);
- Metallographic method, which highlights in sample field aspects such as: particles dispersion, formation of pores, size and shape of particles, quantity of embedded particles.
- Electron microscopy analysis (SEM), in order to highlight microstructure of studied composites.

4.4.1. Determination of chemical composition of samples surface of composite material A6061/FeSi45, by EDX method

For sampling of composite material presented in figure 4.14 analysis of chemical composition with electron microscope (SEM), EDX method was performed. Analysis results are presented in figures 4.14, 4.15 and 4.16.

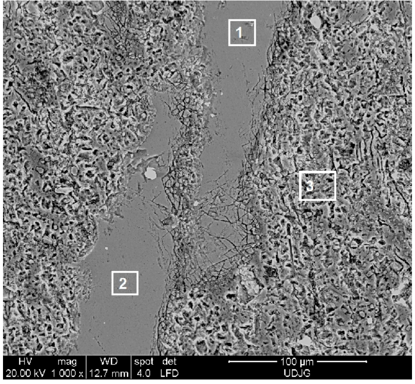
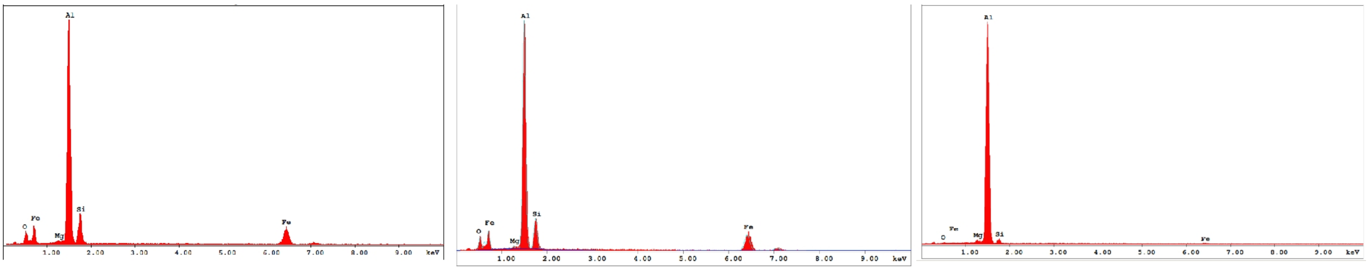
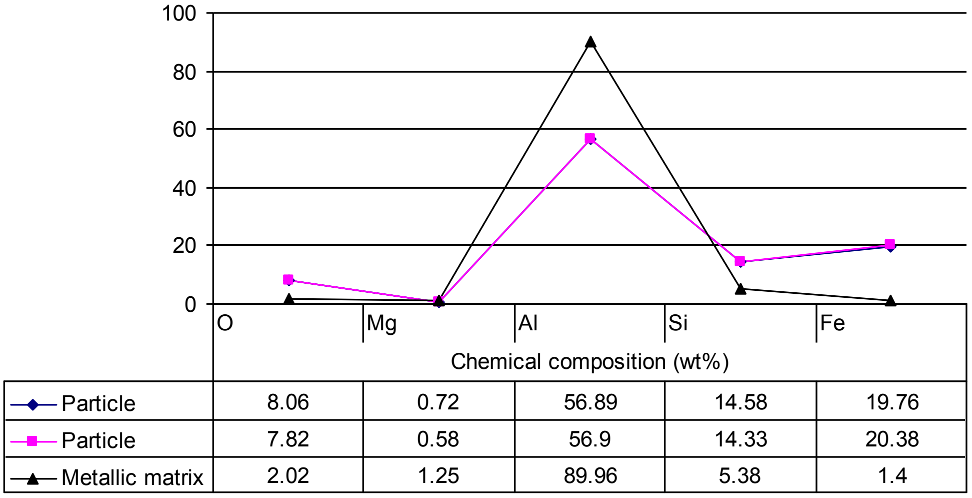


Figure 4.14 SEM Imagine for composite material A6061/FeSi45



1 2 3

Figure 4.15. Spectra 1, 2, 3 regarding elemental chemical composition for composite material A6061/FeSi45



	O	Mg	Al	Si	Fe
◆ Particle	8.06	0.72	56.89	14.58	19.76
■ Particle	7.82	0.58	56.9	14.33	20.38
▲ Metallic matrix	2.02	1.25	89.96	5.38	1.4

Figure 4.16. Chemical composition determined by EDX analysis of composite A6061/FeSi45

Data presented in figures 4.14 – 4.16 highlight the following aspects: chemical composition determined on particle shows that aluminium concentration is of 56.89 wt%, at 730°C temperature for a 10 minute mixing time. Aluminium concentration in metallic matrix was of 89.96 wt%.

Oxygen in concentration of 8 wt% on particles shows an oxidated surface which can made worse yield of embedding.

Iron in 1.4 wt% concentration highlights only mechanical mixing specific to obtaining process by vibrations with a low diffusion and localized at interface particle/matrix.

4.4.2. Chemical elements distribution using x-ray spectrometry (EDX)

Chemical elements distributed on surface of metalographic sample have been highlighted by using electron microprobe attached on electron microscope using a program for drawing maps by using various colors for chemical elements.

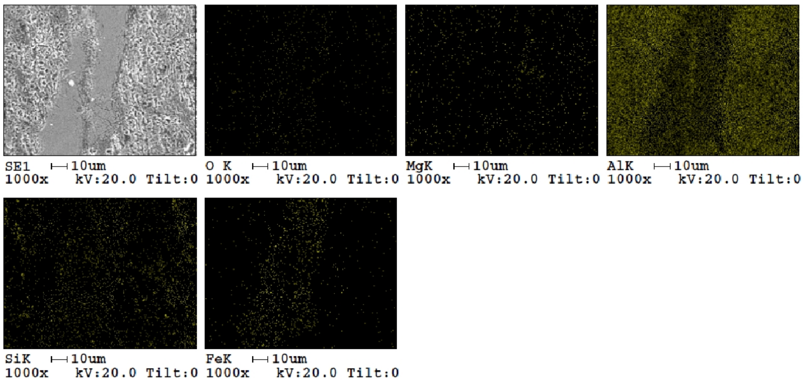


Figure 4.17. Chemical elements distribution for composite material A6061/FeSi45

Composite material presents areas where aluminium has a high concentration and areas where elements iron and silicon corresponding FeSi45 are predominant, as shown in figure 4.17.

It is found that silicon is present both in the particle area as well as in the metallic matrix area, an inhomogeneous distribution and a higher distribution on particle being observed.

Iron is found in particle area and in a very small amount in matrix area, showing that obtaining process has not been accompanied by the formation of a new phase which contains iron, compound $FeAl_3$, being only a localized diffusion at interface.

Oxygen is present both on the particle and in matrix area, observing that it has a higher concentration in particle area, possibly due its oxidation.

4.4.3. Macrostructural analysis of samples of composite material of A6061/FeSi45 obtained by vibrations method

Macrostructural analysis is a common method for the study of surfaces of composite material obtained by liquid phase by casting, with particles of micrometric dimensions. This analysis is required because important aspects regarding distribution and appearence of casting defects (pores) may be highlighted.

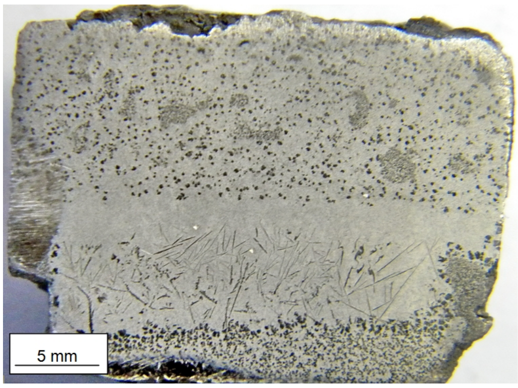


Figure 4.21. Macrostructure of sample B_1 of composite from system A6061/FeSi45 obained by vibration method, $t = 10$ min and $d_{mp} = 0.4$ mm

Figure 4.21 presents a vertical section by sample B_1 of composite material A6061/FeSi45 elaborated by vibration method. In this macrostructure the following aspects are observed: non-uniformity of sample on height and a local uniformity which can be noted horizontally. On height, to the lower part, a layer of particle separately of liquid metal is observed which was seated on the base of crucible, 7 mm in height.

4.4.4. Microstructural analysis of sample of composite material A6061/FeSi45 by optical microscopy

For microstructural analysis, samples were taken from composite material A6061/FeSi45 and were prepared by grinding and polishing in metallographic sample preparation lab from Faculty of Engineering. Microstructural analysis of surfaces highlights details as: dispersion, shape and size of granular particles embedded, presence of defects – pores on analysed surfaces, initiation of diffusion process at interface.

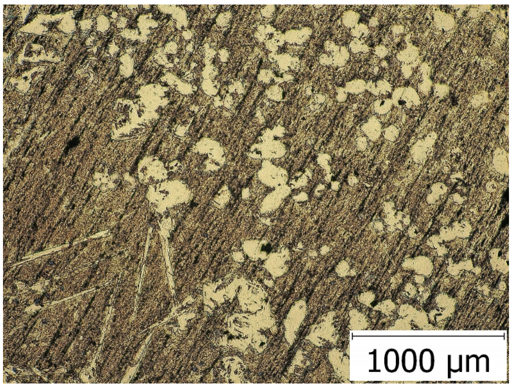


Figure 4.24. Microstructure of composite A6061/FeSi45 obtained by vibration method t = 10 min and d_{mp} = 0.04mm (attack 10%HF)

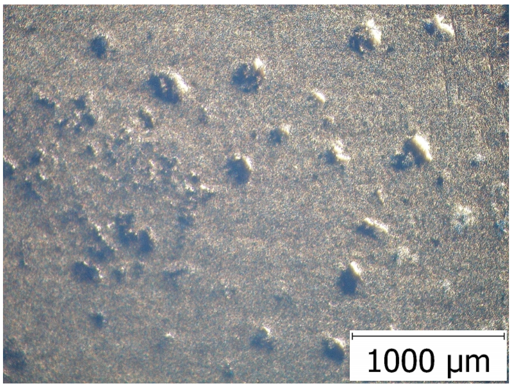


Figure 4.25. Micrography of composite A6061/FeSi45 obtained by vibration method, t = 10 min and d_{mp} = 0.04 mm (without attack)

Microstructures from figures 4.25 and 4.26 highlight fine particles embedded in metallic matrix of composite materials obtained by vibration and presenting the following aspects: homogenous distribution in alloy mass, presence of particles with polygonal shapes; inhomogeneity of granule size, absence of local clusters, good embedding and a reduced number of pores due to of a rapid homogenization and ensuring conditions for evacuation of significant amounts of gas situated between inter-granular voids.

4.4.5. Microstructural analysis of samples of composite material A6061/FeSi45 by electron microscopy (SEM)

Microstructural analysis of sample of composite with particle of FeSi45 embedded in matrix of aluminium alloy obtained by mechanical mixing method with vibrations was achieved with electron microscope SEM Fei Quanta 200. The analysis highlighted some details of surfaces which cannot be observed with help of optical microscopy. Surfaces was prepared by grinding and polishing.

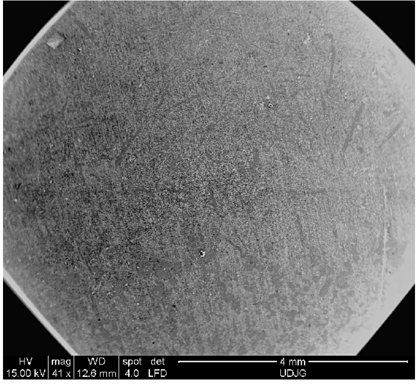


Figure 4.30. SEM imagine of sample C_4, composite material A6061/FeSi45 obtained by vibration method, time 10 min and d_{mp} = 0.4 mm (whitout attack)

In microstructure from figure 4.30 a high density of particles FeSi45 in metallic matrix of aluminium alloy is observed. Also, shapes of particles vary from polygonal to rounded. Variation of dimensions of particles depends on the granulometric fraction used. On sample surface presence of pores is reduced. Mass transfer by diffusion is reduced due to low elaboration duration of composite material and as a result of working temperature of 730°C. On interface metallic matrix and particle was highlighted by EDX analysis contents of aluminium which justifies diffusion process of aluminium from alloy at interface particles and atoms of iron in melt alloy going at formation of compound $FeAl_3$.

4.5. Obtaining composite materials from the system A6061/FeTi30

Obtaining and characterization of composite material A6061/FeTi30 is similary with those of composite material A6061/FeSi45 elaborated with the same method.

Table 4.5 Variation of yield of embedding of particles of FeTi30 with d_{mp} = 0.8 mm in alloy A6061, in function of working parameters accordinaly of mixing method with vibration

Cod sample	Non-embedded mass particles (g)	FeTi30	Amplitude (m x10^{-3})	Frequency (Hz)	Speed V_E (mm/s)	Temperature (°C)	Time (min)
		Yield of embedding η (%)					
D_1	7.5	0	Fără vibrații	Fără vibrații	0	730	10
D_2	0	100	0.45	62	59.28	730	10
D_3	0	100	0.95	63	125.2	730	10
D_4	0.3	96	1.1	72	159	730	10
D_5	0.33	95.6	2.1	46.9	221	730	10
D_6	0.3	95.99	3.2	35	245	730	10

Using data from table 4.5 were plotted graph in figure 4.35.

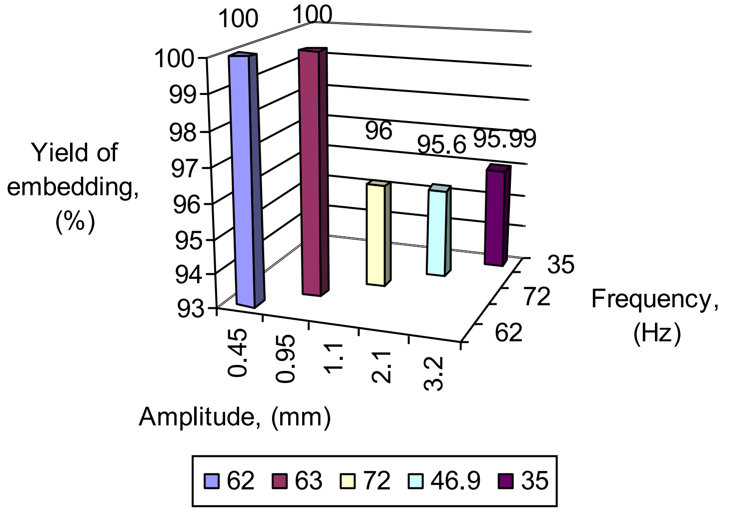


Figure 4.35. Variation of yield of embedding of particles of FeTi30 with d_{mp} = 0.8 mm, depending on frequency and amplitude

Analysing data from table 4.5 and graph from figure 4.35 there appears to be a domain amplitudes between 0.45 and 3.2 mm in which embedding is close to 100% due to fulfilling the conditions of amplitude and frequency of vibrations.

Maximum embedding starts from low amplitude 0.45 mm and frequency 62 Hz and a ratio amplitude/diameter of particle is approximately 0.5. By analysing the spectrum of amplitude (0.45 – 3.2 mm) and frequency (35 ÷ 62 Hz) of vibrations in correlation with particle diameter – 0.8 mm the yield of embedding high can be observed.

Table 4.6 Variation of yield of embedding of particles of FeTi30 with d_{mp} = 0.4 mm in alloy A6061, in function of working parameters accordinaly of mixing method with vibration

Cod sample	Non-embedded mass particles (g)	FeTi30	Amplitude (m x10^{-3})	Frequency (Hz)	Speed V_E (mm/s)	Temperature (°C)	Time (min)
		Yield of embedding η (%)					
E_1	2.38	68.24	0.25	67	41.7	730	10
E_2	3.82	48.94	0.45	62	59.28	730	10
E_3	9.47	53.65	0.5	70	76.61	730	10
E_4	0	100	0.7	61	83.85	730	10
E_5	0	100	0.95	63	125.2	730	10
E_6	0.1	98.61	3.2	35	245	730	10

In table 4.6 and figure 4.36 are presented yields of embedding obtained for composite material A6061/FeTi30 with d_{mp} = 0.4 mm. By analysing the graph in figure 4.36, one observes a diversity of amplitudes and frequencies at which embedding is produced. Domain with an high yield of embedding of particles of FeTi30 is on domain 0.7 ÷ 3.2 mm of amplitude of vibration.

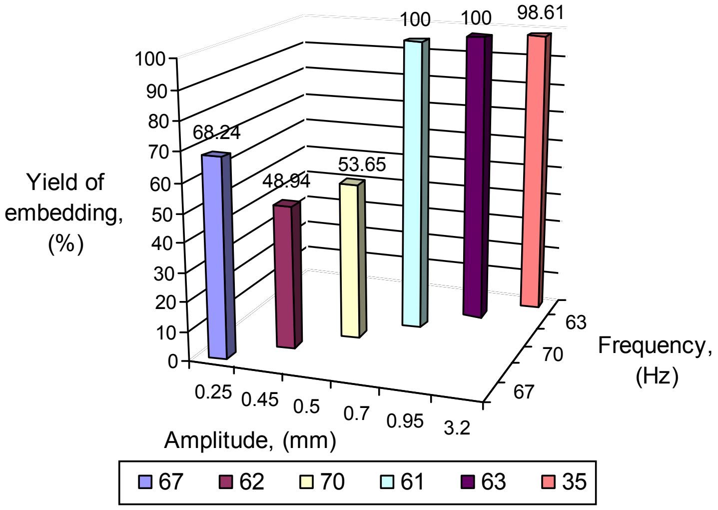


Figure 4.36. Variation of yield of embedding of particles of FeTi30 with d_{mp} = 0.4 mm, depending on frequency and amplitude

From the graph from figure 4.36 is observed a wide range of amplitudes and frequency on which embedding is produced.

From performed experiments on domain with high yield embedding begin from amplitude (0.7 ÷ 3.2 mm) and frequency (63 ÷ 70 Hz).

The process of embedding of particle FeTi30 starts from a lower amplitude of 0.25 mm and frequency of 62 Hz, resulting that the influence of lower diameter of intergranular void is significant for a relatively satisfactory yield 68.24% when d_{mp} = 0.4 mm.

Table 4.7 Variation of yield of embedding of particles of FeTi30 with d_{mp} = 0.04 mm in alloy A6061, in function of working parameters accordinaly of mixing method with vibration

Cod sample	Non-embedded mass particles (g)	FeTi30	Amplitude (m x10^{-3})	Frequency (Hz)	Speed V_E (mm/s)	Temperature (^oC)	Time (min)
		Yield of embedding η (%)					
F_1	3.39	54.75	0.55	62.6	62.14	730	10
F_2	7.09	5.42	0.9	62.5	119.63	730	10
F_3	7.36	1.74	1.15	56	137.5	730	10
F_4	7.24	3.4	2.1	46.9	221.2	730	10
F_5	7.17	4.31	2.7	44.5	289.63	730	10
F_6	2.09	72.03	3.2	35	245	730	10

Using data from table 4.7 has been plotted graph in figure 4.37 and are presented yields of embedding obtained for composite material A6061/FeTi30 with d_{mp} = 0.04 mm.

By analysing the graph shown in figure 4.37, it can be observed that we obtained low rates of embedding in the 0.9 – 2.7 mm field of amplitude. This is due to the following factors: thickness of the layer, high number of particles, diversity of particles forms, high wetting angle, oxide layer formed at the surface of liquid.

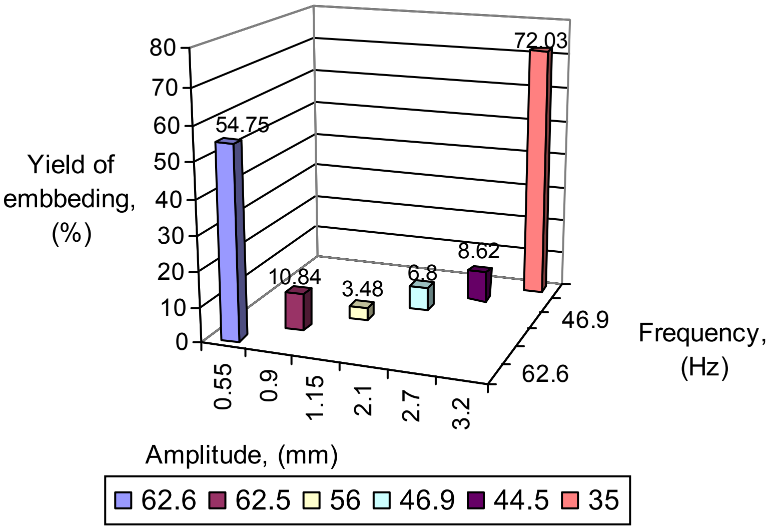


Figure 4.37. Variation of yield of embedding of particles of FeTi30 with d_{mp} = 0.04 mm, depending on frequency and amplitude

Because the average diameter of particles is relatively reduced,of 0.04 mm, a thick layer of particles can react as a compact porous material. Thus, dividing the layer entails the creation of different conditions so that they can be embedded into a large quantity (amplitude and frequency much grater than the analysed cases). Above 3.2 mm it can be observed the growth embedding as a result of high amplitude.

4.6. Characterization of composite materials from system A6061/FeTi30

For characterization of composite material were used the following investigation methods:

- Chemical analysis with the aid of electron microsond, EDX method, which highlight modification of chemical composition at interface between metallic matrix and solid particles in granular form.
- Chemical elements distribution using x-ray spectrometry.
- Metalographic method, which highlight in sample field aspects such as: particle dispersion, pores appearence, distribution, quantity of embedded particles. It was made ground glass joint metalographic to highlight the emergence of new phases due to diffusion of aluminium from melt at interface particle-alloy of aluminium and of elements iron and titanium from particle in alloy melt, when at temperature 730°C may form compounds $FeAl_3$ and $TiAl_3$, the influence of cooling process on composite material, appearence of specific structureson rapid or slow cooling;
- Electron microscope analysis (SEM), to highlight morphology of composite material.

4.6.1. Chemical analysis on sample surface for composite material A6061/FeTi30 by EDX method

In the course of obtaining composite material in liquid state between metallic matrix and particles in granular state localized chemical reactions can take place at the edge of particles following the diffusion of atoms of aluminium from metallic bath at particle surface and by diffusion of iron and titanium from particle of FeTi30. At temperature 730°C and particulary on greater temperatures can appear $FeAl_3$ and $TiAl_3$ that are distributed around the particles and which, as a result of the vibrations end up in the metal matrix.

The data regarding chemical and structural analysis are represented in figures 4.38 ÷ 4.40, which can show this chemical modification by x-ray spectrometry (EDX).

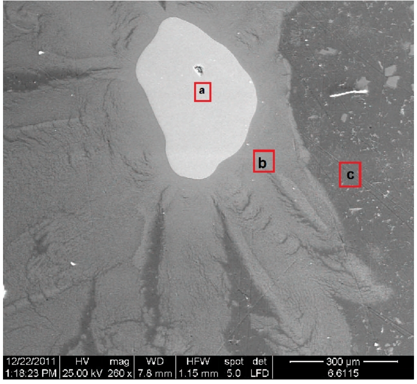
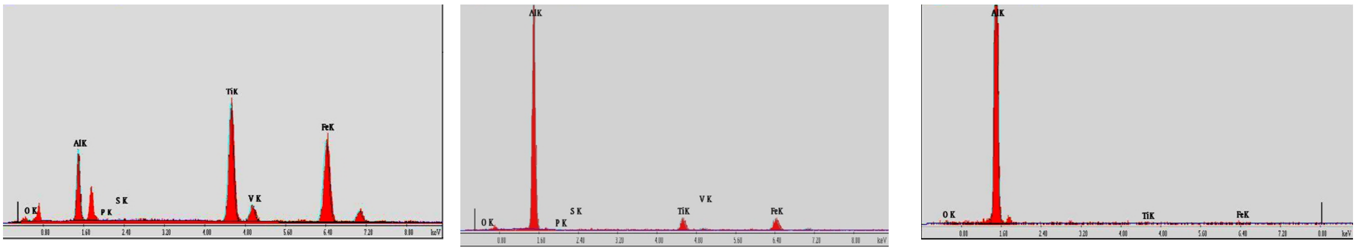
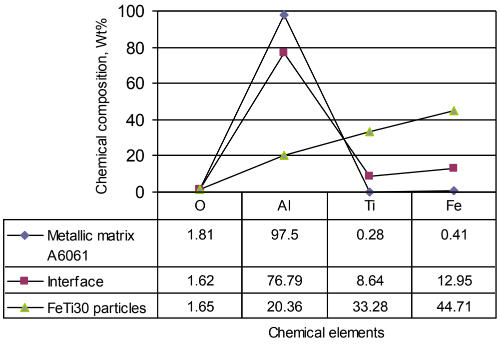


Figure 4.38. SEM imagine for composite material A6061/FeTi30



a b c

Figure 4.39. Spectra a, b and c regarding elemental chemical composition in three points on sample surface of composite material A6061/FeTi30



	O	Al	Ti	Fe
Metallic matrix A6061	1.81	97.5	0.28	0.41
Interface	1.62	76.79	8.64	12.95
FeTi30 particles	1.65	20.36	33.28	44.71

Figure 4.40. Chemical composition determined by EDX analysis of composite A6061/FeTi30

The data presented in figure 4.38 ÷ 4.40 reveal the following: chemical composition determined on the particle highlight aluminium in concentration 20.36 wt%, due to obtaining conditions by mechanical vibration method, at 730^{o}C temperature and 10 min. mixing time.

At interface, the aluminium content is 76.79 wt%, a value between that of metallic matrix, namely 97 wt%, and the one determined on particle 20.36 wt%. These values of aluminium content determined on the three area are due to mass transfer of aluminium from matrix toward the other two areas and because of oxides washed and partial wetting of particle by liquid alloy which also determine a superficial dissolution.

4.6.2. Chemical elements distribution using x-ray spectrometry method (EDX)

Chemical elements distribution were determined by x-ray spectrometry and is highlighted in the maps presented in figure 4.41, where on different colors is highlight presence and quantity of chemical elements.

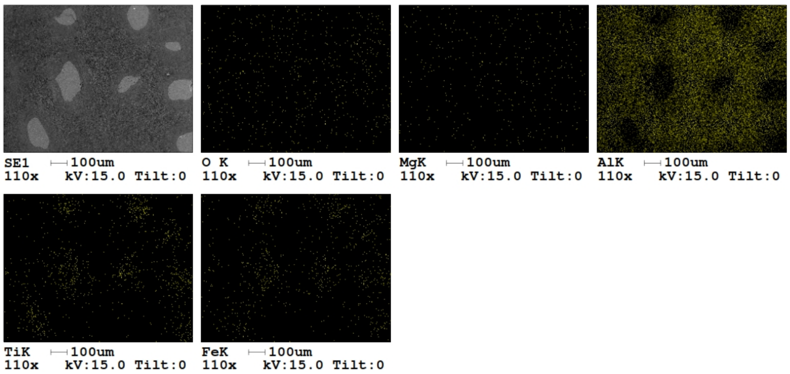


Figure 4.41. Chemical elements distribution for composite material A6061/FeTi30

Aluminium from metallic matrix is found in high concentration and in area of particles is present but in reduced concentration according to figure 4.41.

Titanium is present only in the area afferent of particles, thus showing that elaboration process of composites took place in a reduced time and diffusion was limited.

Iron, also, is found only in areas where are presented particles and very low in metallic matrix, as a result of reduced time of obtaining process.

Oxygen is found in a reduced concentration both on particle as well as in metallic matrix, positive aspect because oxidation process worse yield of embedding.

4.6.3. Macrostructural analysis of sample of composite material from system A6061/FeTi30

Macrostructural analysis is a common method for the study of composite materials surfaces, with particle of micrometric dimensions.

This analysis is compulsory as it can highlight relevant aspects, such as the uniform distribution and casting defects – porosity.

Figure 4.42. Macrostructure of sample D_2, composite A6061/FeTi30 obtained by vibration method, time = 10 min and d_{mp} = 0.8 mm

In figure 4.42, in right area it can be observed that a rapid cooling has taken place as a result of an existing (embedding) quantity of particles of FeTi30 in the metallic matrix. The uniform dispersion of particles horizontally and vertically can be observed especially in the right area of the analysed sample.

4.6.5. Microstructural analysis of sample of composite material from system A6061/FeTi30 by electron microscopy (SEM)

Microstructural analysis was carried aut on sample of composite material by vibration method. In figures 4.50 – 4.53 we present SEM images of composite material with granular particles with d_{mp} = 0.8 mm, 0.4 mm and 0.04 mm.

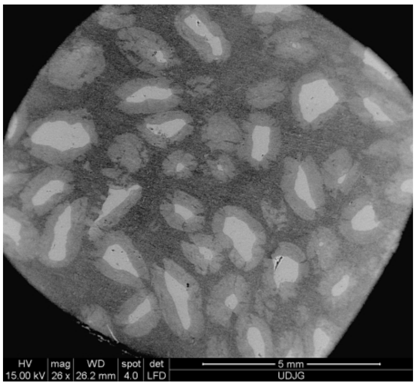


Figure 4.50. SEM image of composite A6061/FeTi30 with particles with d_{mp} = 0.8 mm

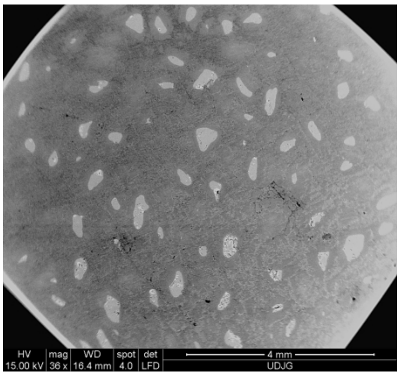


Figure 4.51. SEM image of composite A6061/FeTi30 with particles with d_{mp} = 0.4 mm

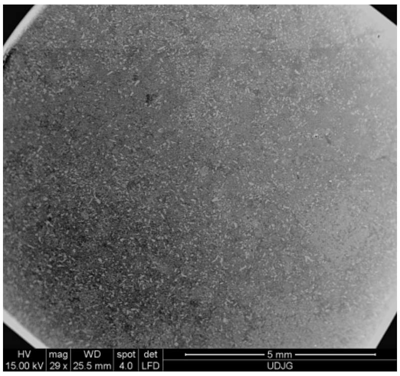


Figura 4.52. SEM image of composite A6061/FeTi30 with particles with d_{mp} = 0.04 mm

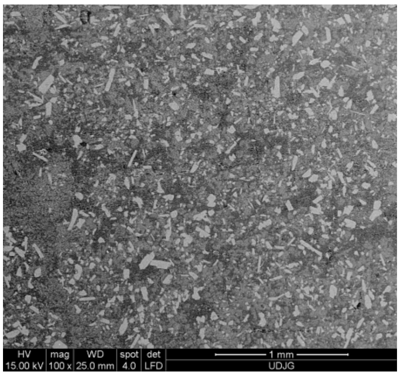


Figura 4.53. SEM image of composite A6061/FeTi30 with particles with d_{mp} = 0.04 mm

Analysing microstructural sample from figures 4.50 ÷ 4.53 it can be seen that the particle distribution in metallic matrix is more uniform that in the case of larger particles,and also the lack of agglomerates because of the positive effect of amplitude and vibration frequency, so that optimum conditions are created for many of particles to move vertically in the metal matrix. An important role in this respect it is the time of vibration and choosing the right moment of cooling the melt.

4.7. Obtaining composite materials from system A6061/SiC

The analysis of manufacturing methods of composite material A6061/SiC and its characterization is similar to those of composite A6061/FeSI45 and A6061/FeTi30. Thus, the previously presented methodologyis abided by, namely formation of structure type sandwich and the associated operating mode.

Table 4.8. Variation of yield of embedding of particle of SiC with d_{mp} = 0.8 mm in alloy A6061, in function of working parameters coresponding mechanical mixing method by vibration

Name sample	Non-embedded mass particles (g)	SiC	Amplitude (m x10^{-3})	Frequency (Hz)	Speed V_E (mm/s)	Temperature (°C)	Time (min)
		Yield of embedding η (%)					
G_1	7.5	0	Without vibrations	Without vibrations	0	730	10
G_2	3.54	52.74	0.45	62	59.28	730	10
G_3	0.02	99.64	0.95	63	125.2	730	10
G_4	0.1	97.08	3.2	35	245	730	10

Analysing data from table 4.8 and graph from figure 4.54, we observe that the value domain of vibration amplitude which is favourable to embedding in metallic melt of SiC particles is between 0.95 mm and 3.2 mm. From the graph in figure 4.54 it can be observed that the yield of embedding is above 50% from a low amplitude of 0.45 mm and 62 Hz frequency.

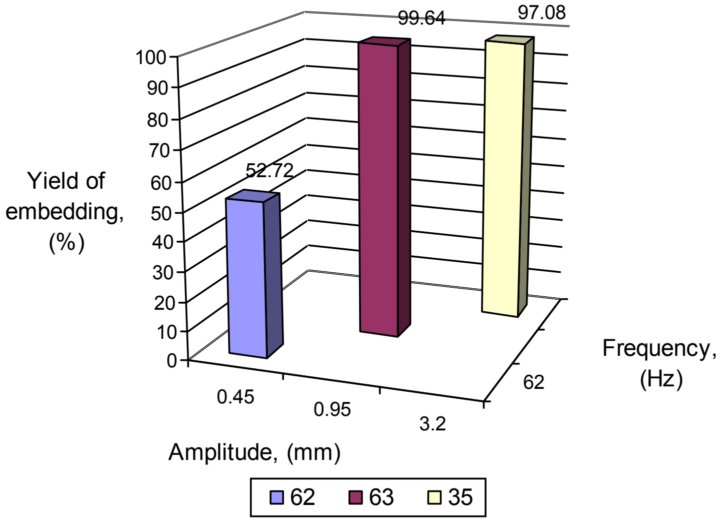


Figure 4.54. Variation of yield of embedding of particles of SiC with d_{mp} = 0.8 mm, depending on frequency and amplitude

Ratio of amplitude vibration/ particle diameter is of 0.5% and this shows that newly created intergranular voids and reduction of wetting angle favour obtaining composite materials.

Tabel 4.9. Variation of yield of embedding of particle of SiC with d_{mp} = 0.4 mm in alloy A6061, in function of working parameters coresponding mechanical mixing method by vibration

Name sample	Non-embedded mass particles (g)	SiC	Amplitude (m x10^{-3})	Frequency (Hz)	Speed V_E (mm/s)	Temperature (°C)	Time (min)
		Yield of embedding η (%)					
H_1	5.76	23.15	0.25	67	41.7	730	10
H_2	0.11	98.49	0.45	62	59.28	730	10
H_3	1.69	77.46	0.5	70	76.61	730	10
H_4	0.06	99.12	0.7	61	83.85	730	10
H_5	0.10	98.63	0.95	63	125.2	730	10
H_6	4.35	41.9	3.2	35	245	730	10

By analysing table 4.9 and graph from figure 4.55 we can observe that results are diverse in amplitude domain 0.45 – 3.2 mm: from 0.45 mm amplitude, yield of embedding is of 98.49 % and at 3.2 mm amplitude yield of embedding is of 41.9%.

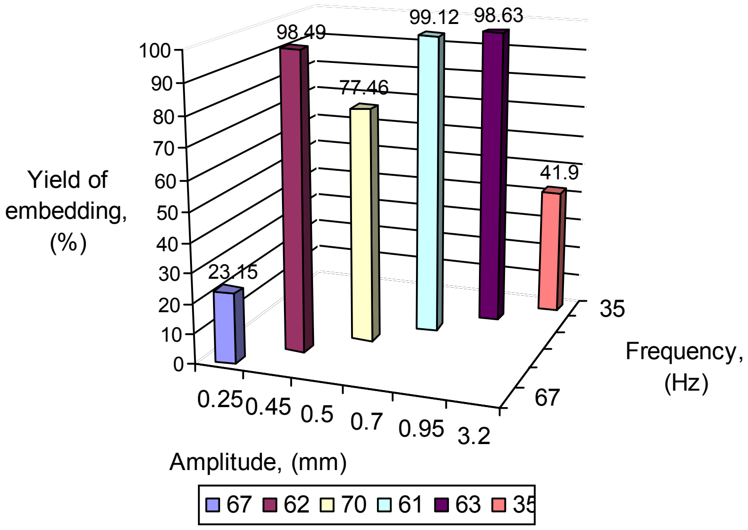


Figure 4.55. Variation of yield of embedding of particles of SiC with d_{mp} = 0.4 mm, depending on frequency and amplitude

From the research carried out, it is observed that the domain of amplitude and frequency tested may present an extended range. This phenomen is possible due to the fact that granular particles have different shapes such as: needle, polygonal, spherical and their behaviour depends on local conditions, on their positioning.

Table 4.10. Variation of yield of embedding of particles of SiC with d_{mp} = 0.04 mm in alloy A6061, in function of working parameters accordinaly of mixing method with vibration

Cod sample	Non-embedded mass particles (g)	SiC Yield of embedding η (%)	Amplitude (m x10^{-3})	Frequency (Hz)	Speed V_E (mm/s)	Temperature (^oC)	Time (min)
I_1	7.41	1.14	0.55	62.6	62.14	730	10
I_2	7.40	1.32	0.9	62.5	119.63	730	10
I_3	7.36	1.75	1.15	56	137.5	730	10
I_4	7.29	2.77	2.1	46.9	221.2	730	10
I_5	7.5	0	2.7	44.5	289.63	730	10
I_6	5.76	23.18	3.2	35	245	730	10
I_7	7.5	0	3.35	47	339.82	730	10

From table 4.10 and graph from figure 4.56 we can observe that the chosen domain in the frame of experiments did not succeed to highlight a good embedding, which demonstrates that fine particles of silicon carbide, without an additional coating treatment are not embedded.

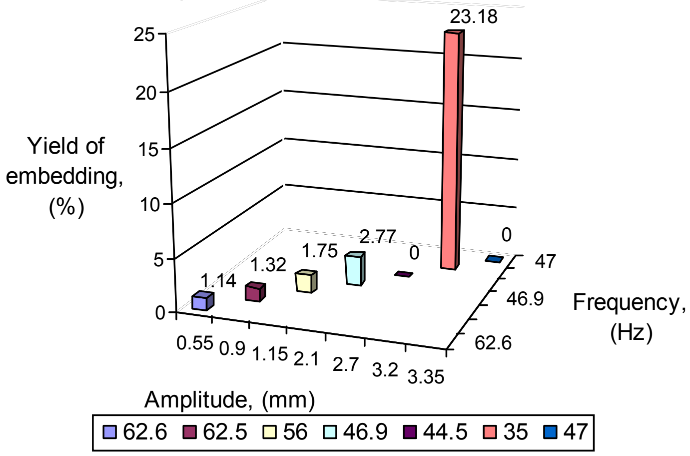


Figure 4.56. Variation of yield of embedding of particles of SiC with d_{mp} = 0.04 mm, depending on frequency and amplitude

In order to increase the percentage of embedded particles, it is necessary that additional coating technologies be designed. A future solution can be given by extending the domain of amplitude and frequency.

4.8. Characterization of composite materials from system A6061/SiC

For analysis of composite material the following investigation methods were used:

- Chemical composition EDX, which highlights the modification of chemical composition at interface metallic matrix – solid particles in granular state.
- Metalographic method, which highlights in sample field aspects such as: particle dispersion, pores which form,yield of embedding, influence of mixing method on obtaining process of composite materials.
- Electron microscope analysis (SEM) and optical, to highlight macrostructure and microstructure of composites elaborated.

4.8.1. Chemical analysis by EDX method on sample surface of composite material from system A6061/SiC

In figure 4.58 are presented areas where analysis was made, and in figure 4.59 are presented spectra which show variation of chemical composition in three area investigated of composite materials A6061/SiC.

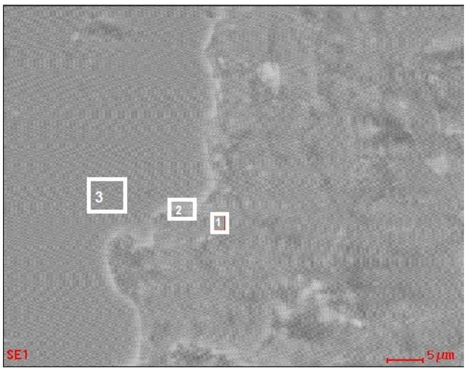
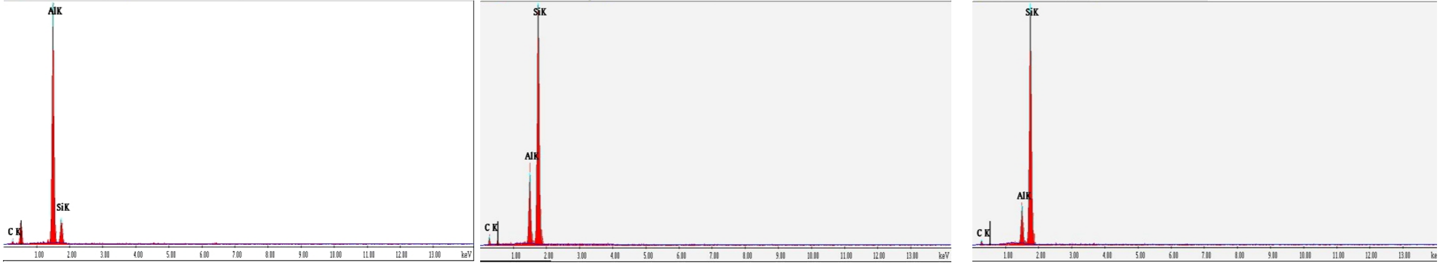


Figure 4.58. SEM imagine of composite material A6061/SiC with d_{mp} = 0.4 mm



1 2 3

Figure 4.59. Spectra 1, 2, 3 regarding elemental analysis – by EDX method for composite material A6061/SiC

Tabel 4.12. Chemical analysis of composite material

No.	Analysed region	Chemical composition (%)		
		Al	Si	C
1	Metallic matrix A6061	56.88	15.67	27.45
2	Interface	10.25	51.03	38.72
3	Particule SiC	7.35	60.98	31.68

The following aspects are noticeable from figure 4.59: chemical composition on particle highlights the presence of aluminium in 7.35%, as a result of intense mechanical mixing process by vibration method, when are created thermodynamical conditions for bidirectional diffusion and for the formation of compound Al_4C_3. Thus, it can be observed that was produced wetting process of silicon carbide particles by metallic matrix and also embedding at high yields of these.

At the interface, the chemical composition for aluminium is of 10.25% (mass percent) an intermediary value between those of metallic matrix 56.88% (mass percent) and those on the particle of 7.35% (mass percent), a value which may be explained by its diffusion on interface area. Compound Al_4C_3 can be formed if temperature and maintenance time conditions from obtaining composite material are fulfilled.

4.8.2. Macrostructural analysis of samples of composite material from system A6061/SiC

Macrostructural analysis aims at highlighting on macro scale aspects regarding the embedding of granular particles, as well as aspects related to particles distribution, their shapes, appearence of defects, chemical reaction presence between chemical elements presented in heterogeneous system, especially at interface metallic matrix-particles.

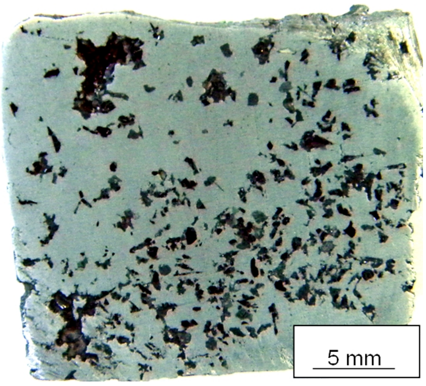


Figure 4.60. Macrostructure of sample G_3, composite material A6061/SiC by vibrations method, time = 10 min and d_{mp} = 0.8 mm

Figure 4.60 presents a vertical section by sample G_3 of composite material obtained by mechanical mixing with vibrations. In this macrostructure are presented the following aspects: non-uniformity of the sample both vertically and horizontally, presence of pores both in upper and lower parts, as a consequence of particles agglomeration and how to achieve quenching the sample. The particles embedded are diverse in shape, preponderantly needle-shaped.

4.8.3. Microstructural analysis of samples of composite material from system A6061/SiC by optical microscopy

Similar with other system previously analysed, for microstructure analysis, samples have been taken from composite material A6061/SiC and were prepared by grinding and polishing in the sample preparation laboratory. Microstructural analyses of samples aimed at highlighting the following details: dispersion, shape and size of particles, appearance of new phases following the process of diffusion of elements from heterogeneous system when metallic matrix is in liquid state, as well as presence of pores.

In figures 4.63 – 4.64 were highlighted microstructures on which granular particles of silicon carbide with d_{mp} = 0.8 mm and 0.04 mm have been embedded in metallic matrix of A6061.

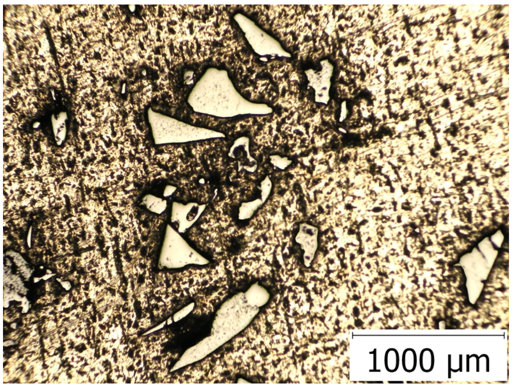


Figure 4.63. Micrography of composite material A6061/SiC, obtained by vibration method, time = 10 min and d_{mp} = 0.8 mm (without attack)

Silicon particles with d_{mp} = 0.8 mm highlighted in microstructure from figure 4.63 presents the following characteristics: they have different shapes from elongated to polygonal, a significant presence of pores near particles, homogeneous distribution of these and formation of agglomerations.

4.8.4. Microstructural analysis of samples of composite material by system A6061/SiC by electron microscopy (SEM)

For analysis was used electron microscope SEM Fei Quanta 200. Microscopic analysis (SEM) aimed at highlighting details of sample surface, details which cannot be observed with the aid of optical microscopy. Sample surface was prepared by grinding and polishing.

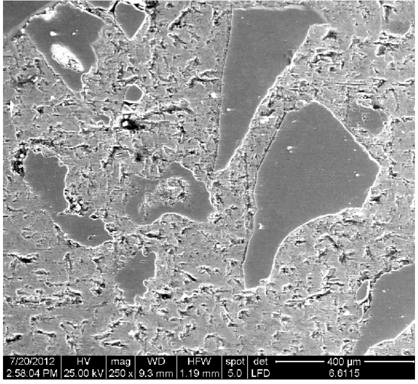


Figure 4.65. SEM imagine of composite material A6061/SiC, obtained by vibration method, time = 10 min and d_{mp} = 0.4 mm (whitout attack)

From microstructure presented in figure 4.65 the following are observed: homogeneous distribution of granular particles, various shapes, mostly elongated, there is apossibility of compound formation Al_4C_3 at interface by localized diffusion of aluminium atoms and carbon atoms from silicon carbide from interface areas matrix-particle, an aspect also favoured by mechanical mixing with aid of vibrations. Also, are highlighted pores present in metallic matrix and not at the edge of granular particles.

4.9. Partial conclusions regarding the obtaining and characterization of composite materials from systems A6061/FeSi45, A6061/FeTi30 and A6061/SiC

1. The technology of obtaining composite materials by the method of mechanical mixing with aid of vibrations is based on arranging in overllaped layers (sandwich type) of two blocks of aluminium alloy A6061 and granular particles of FeSi45, FeTi30 and SiC with medium diameter 0.8 mm, 0.4 mm and 0.04 mm.
2. For the study of yield of embedding of particles as well as of chemical, mechanical and structural properties of composite materials elaborated by this method, an installation was designed and manufactured, which comprises a system of vibrated table, a heating system and a crucible in which are made composite material, crucible which can be vibrated uniaxially on vertical.
3. For particles with d_{mp} = 0.4 mm it was found that yield of embedding are higher than large particles with d_{mp} = 0.8 mm, which is at odds with that the higher average particle diameter is greater the higher permeability of the layer of molten particles is better. The explanation can be given by the fact that wetting of particles is better in case of system A6061/FeSi45, although the number of particles increased.
4. For composites which are based on system A6061/FeTi30 it was found that yield of embedding is favoured in case of large particles d_{mp} = 0.8 mm, when amplitude and frequency are large in comparison with particles with d_{mp} = 0.4 mm, when it was found that at high amplitude and smaller frequency of vibrations,yields were very high. This thing is explained by the fact that, by lowering d_{mp}, the apparent density of particles has dropped, which can explain maintaining their duration time in melt without decantation to take place, so were created infiltration conditions of aluminium alloy among solid particles.
5. For very small particles of FeTi30 (d_{mp} = 0.04 mm) yield of embedding have been much lower than for particles of 0.8 mm and 0.4 mm, due to the fact that on very fine granulation specific surface contact has greatly increased, which has led to a clustering tendency of particles, so that for large quantities of particles their agglomeration is carried out and they have the tendency to separation in slag.
6. For composites type A6061/SiC obtained by vibration method, when medium diameter of particles have been 0.8 mm yields of embedding have been much higher reaching up to values of 99.64%, in the case of amplitude and frequency much lower of vibrations, while for particles with d_{mp} = 0.4 mm, yields of embedding were better throughout the studied area.

7. Elongated irregular shape, has been found to be advantageous from the point of view of yield of embedding in case of large particles (d_{mp} = 0.8 mm) for amplitude and frequency high of vibrations, as a result of compact decreased.

8. The procedure has proven its valability by embedding between 7.7 ÷ 97% for FeSi45; 1.74 ÷ 100% for FeTi30; 1.14 ÷ 99.64% for SiC, in function of granulation, amplitude and frequency of oscillation.

9. At all three systems elaborated, A6061/FeSi45, A6061/FeTi30 and A6061/SiC for all granulations: 0.8 mm, 0.4 mm and 0.04 mm took place limited diffusion processes at interface, highlighted, by chemical composition determination in three areas (matrix, interface, particle) observing, for exemple, for concentration variation of aluminium in respective areas. Also, at chemical analysis was observed variation of oxygen concentration ascertaining an high concentration on particle (so an oxidated surface exists), and in metallic matrix, as a result of presence of Al_2O_3 and other oxides.

10. Distribution of chemical elements determined by EDX method has shown based on color tones and area with high concentrations of main chemical element (aluminium) on particle but and for silicon distribution both in matrix as well as on the particles. Also, it can be evaluates the degree of oxidation of surfaces by distribution of oxygen chemicaly linked under the form of SiO_2 and Al_2O_3.

11. Macrostructural analysis has shown a diversity of situations, such as areas with a homogenous dispersion, but also areas with decanted particles to the bottom of sample as a result of density difference. The presence of pores was identified both around particles and in sample volume, as a result of an insufficient mechanical mixing, which usually favors leaving the melt. It may also be observed the formation of areas with high particle concentrations and areas without particles, as a result of amplitude and frequency which allowed embedding particles but was not succeeded their dispersion. Microscopical analysis (optical and SEM) shows granular particles embedded, with polygonal shapes for FeSi45 and FeTi30 with rounded edges and elongated shapes for SiC with sharp edges. Also, we can observe a broad field of dimensions, possibly due to mechanical breakage produced during the process of obtaining.

12. Installation of elaboration is based on mixing metallic bath with refractory particle by inducing vibrations, which present superior advantages Stir Casting Vortex method and can be recommended for testing on microproduction level.

4.10. Thermal characterization of composite materials with metallic matrix of aluminium alloy A6061 and refractory particles

Analysis of composites by differential scanning calorimetry (DSC) allows highlighting temperature domain and the thermal effect which accompanies theprocess that occurs in the analysed systems. These allow optimization desired for composite materials elaborated.

The particle effect (FeSi45, FeTi30 and SiC) was studied on transformation temperature which takes place on obtaining composites in liquid state.

The following processes were highlighted :

- Exo/endothermal processes which take place for analysed systems;
- Peak displacement with obtaining composite material
- Appearance of new phases which may belong to binary systems Al – Fe, Al – Ti, Al – SiC, C – Al or ternary systems Al – Fe – Si, Al – Fe – Ti, Al – Si – C.

In table 4.13 is presented temperatures for begin of transformation $T_{în}$, end of transformation T_{sf}, peak temperature, free enthalpy (ΔH), and crystallinity for system A6061/FeSi45.

Table 4.13. Specific parameters DSC analysis for composite material A6061/FeSi45, respectively T_{in}, T_{sf}, T_{pic}, crystallinity

No.	Analysed material	$T_{in.}$ (^oC)	$T_{sf.}$ (^oC)	T_{pic} (^oC)	Energy corresponding to peak area (mJ)	Sample mass (mg)	ΔH (J/g)	crystallinity (%)
1	A6061	654.96	673.47	658.41	-12850	32.265	398.76	100.28
2	A6061/FeSi45 0.8 mm	560.69	588.32	566.93	-11440	50.550	226.31	56.59
		610.04	677.84	666.33	-1296,76	50.550	25.65	6.52
3	A6061/FeSi45 0.04 mm	560.23	582.19	568.61	-16660	68.106	244.61	61.60
		659.06	677.27	668.65	-2729,87		40.08	10.15
4	A6061 + FeSi45 0.04 mm powder	659.83	678.28	667.21	-8924,00	41.208	216.56	54.54

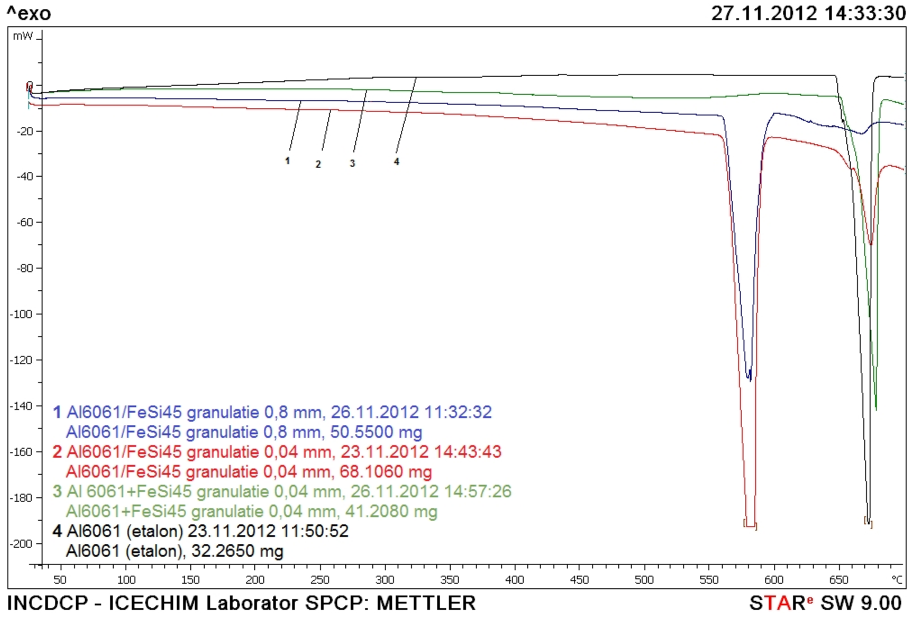


Figure 4.71. Overlapping DSC curves for composites belonging to the system A6061/FeSi45

In figure 4.71, by overlapping the four curves, thermal effects which have accompanied the process of obtaining composite material A6061/FeSi45 are emphasized for the three cases studied, for granular particles with d_{mp} = 0.8 mm, d_{mp} = 0.04 mm and obtaining „in situ” (aluminium alloy + powder). Thus, we can observe two peaks corresponding to the thermal effects presented; first peak corresponding to eutectic (αAl + Si) with 1.65 % Si and the second corresponding to solid solution α (of Si in Al) and solid solution α of Fe in Al from eutectic $FeAl_3$ + (Al), as well as displacement of specific peaks temperatures.

Table 4.14. Specific parameters DSC analysis for composite material A6061/FeTi30, respectively T_{in}, T_{sf}, T_{pic}, crystallinity

No.	Analysed material	$T_{in.}$ (^oC)	$T_{sf.}$ (^oC)	T_{pic} (^oC)	Energy corresponding to peak area (mJ)	Sample mass (mg)	ΔH (J/g)	crystallinity (%)
1	A6061	654.96	673.47	658.41	- 12850	32.265	398.76	100.28
2	A6061/FeTi30 0.8 mm	648.33	675.94	667.12	- 13890	53.096	261.60	65.90
3	A6061/FeTi30 0.04 mm	638.49	672.27	665.08	- 8702.07	44.641	197.93	48.85
4	A6061/FeTi30 0.04 mm powder	660.63	681.25	670.31	- 8328.39	35.271	236.13	59.47

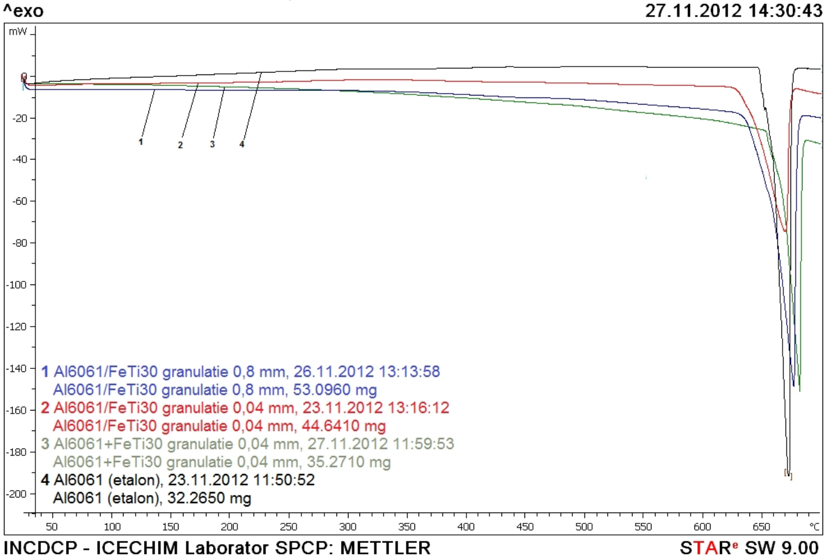


Figura 4.75. Overlapping DSC curves for composites belonging to the system A6061/FeTi30

In figure 4.75, by overlapping the four curves, are emphasized by comparison, the changes transformation temperatures and increasing areas of transformation, thus showing the influence of particles of FeTi30 in granular state at obtaining composite material. It is observed that there has been a peritectic transformation (Al + $TiAl_3$) with 0.147%Ti on temperature 665°C and solid solution α from Fe in Al, from eutectic $FeAl_3$ + (Al) at temperature 655°C.

Table 4.15. Specific parameters DSC analysis for composite material A6061/SiC, respectively T_{in}, T_{sf}, T_{pic}, crystallinity

No.	Analysed material	$T_{in.}$ (^oC)	$T_{sf.}$ (^oC)	T_{pic} (^oC)	Energy corresponding to peak area (mJ)	Sample mass (mg)	ΔH (J/g)	crystallinity (%)
1	A6061	654.96	673.47	658.41	-12850	32.265	398.76	100.28
2	A6061/SiC 0.8 mm	659.88	668.82	668.32	-4247.39	18.489	229.73	57.92
3	A6061/SiC 0.4 mm	653.21	679.85	665.19	-22720	87.889	258.51	65.15
4	A6061/SiC 0.04mm powder	661.44	680.30	670.55	-7976.73	34.122	233.77	58.54

Table 4.15 presents the specific parameters for DSC analysis for composite material A6061/SiC. DSC analysis of the corresponding curves, in figures 4.76 ÷ 4.79 thermal effects are observed corresponding to phase transformations (solid - liquid).

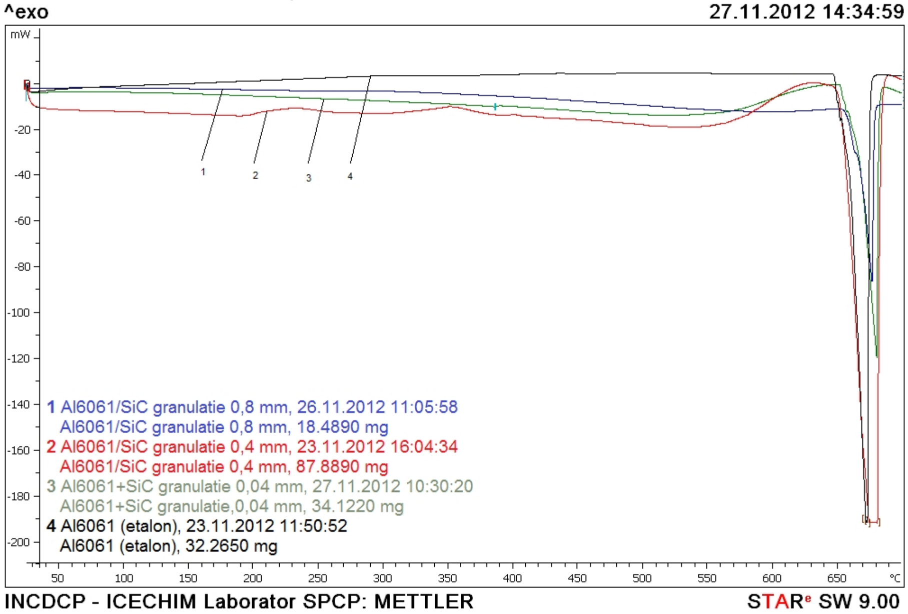


Figura 4.79. Overlapping DSC curves for composites belonging to the system A6061/SiC

In figure 4.79, DSC analysis is presented by the overlapping of the four cases of curves, observing the thermal effects generated from heating. Thus, analysing figure 4.79 we can present the following:

- the appearance of peak corresponding to the transformation from solid phase in liquid phase of metallic matrix;
- the result of obtaining technology on composite material is reflected by temperatures start, end of tranformation near to those of aluminium alloy,and due to reduced solubility of carbon in aluminium was formed compound Al_4C_3;
- it is not placed out on other constituents thermograms due to the process of obtaining metalograph;

4.10.1. Partial conclusions regarding thermal effect on obtaining composite materials

1. DSC analysis for composite systems studied in the paper allowed the determination of several process parameters: temperatures for the beginning of transformation $T_{în}$, end of transformation T_{sf}, peak temperature, free enthalpy (ΔH), and crystallinity.
2. Following DSC analysis performed for the system A6061/FeSi45, it was confirmed the presence of eutectic (αAl + Si), with 1.65 % Si, in the range of 560 – 588°C, in the range of 610 – 677°C beginning, respectively ends transformation from solid phase in liquid phase with formation solid solution α (of Si in Al) and solid solution α of Fe in Al, from eutectic $FeAl_3$ + (Al).
3. Following DSC analysis performed for the system A6061/FeTi30, it can be observed presence of peritectic reaction:

$L + TiAl_3 \xrightarrow{665^\circ C} (Al)$ and formation of solid solution α of Fe in Al from eutectic $FeAl_3$ + (Al) at temperature 655°C.

4.Following DSC analysis, made for system A6061/SiC, it is observed, by comparing the specific trend curves of composite material A6061/SiC and of aluminium alloy, that the beginning and end of temperature of transformation have close values due to a reduced solubility of carbon in aluminium. Between aluminium and carbon is formed compound Al_4C_3 leading to the increasing of silicon content from liquid phase as a result of reaction $Al(l) + SiC(s) = Si(l) + Al_4C_3(s)$.

4.11. Determination of microhardness for composite materials with metallic matrix of aluminium alloy A6061 and refractory granular particles of FeSi45, FeTi30 and SiC

Microhardness Vickers was determined in order to evaluate the composite materials elaborated from their mechanical properties point of view, respectively hardness. Tests have been made on multiple area of composite material, especially in area from the near of interface matrix/particle and on surface of solid particles.

For system A6061/FeSi45

Multiple determination of microhardness Vickers to rise results precision were made.

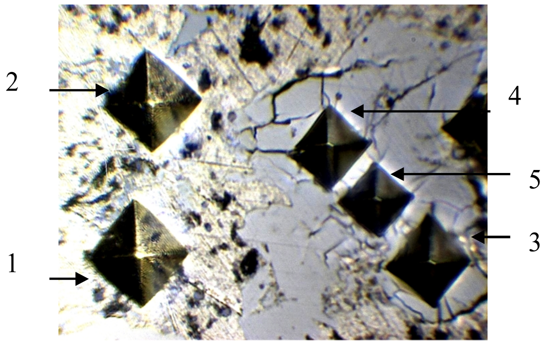


Figure 4.80. Appearance indentation on microhardness determination for composite material A6061/FeSi45 with metallic matrix and granular particles

In figure 4.80 footprints 1 and 2 are on matrix of aluminium alloy and 3, 4, 5 on particle FeSi45.

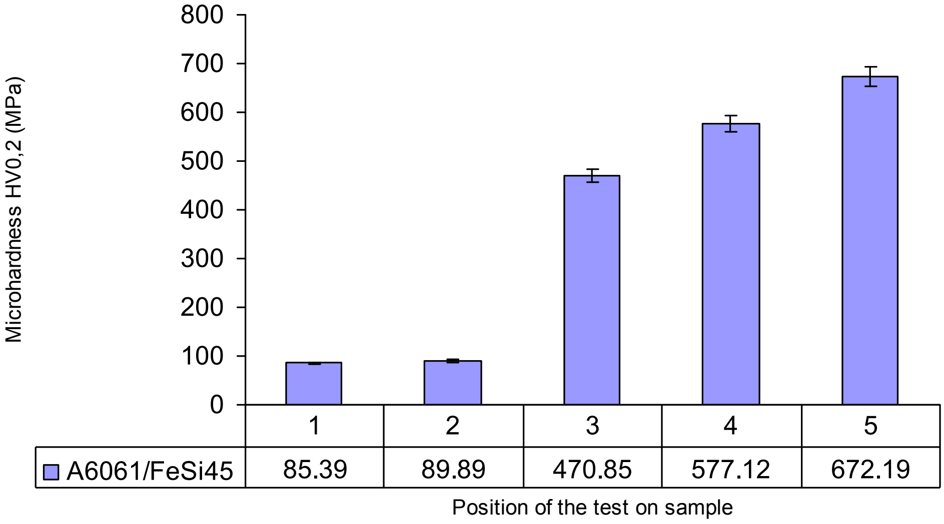


Figure 4.81. Variation of microhardness according to the position of footprints left for composite A6061/FeSi45

In figure 4.81 microhardness variation is presented according to the position on sample of footprint, and a rising of microhardness values, and a medium value of 573 MPa are observed.

For sistem A6061/FeTi30

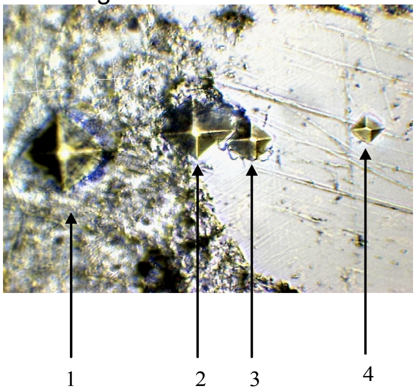


Figure 4.83. Appearance indentation on microhardness determination for composite material A6061/FeTi30 with metallic matrix and granular particles

From figure 4.83 we may observe for system A6061/FeTi30 the determination way of microhardness in this three specific areas, namely, particle and interface area. In interface area also appear new phases due to diffusion of some elements from matrix in particle and from particle in composite matrix.

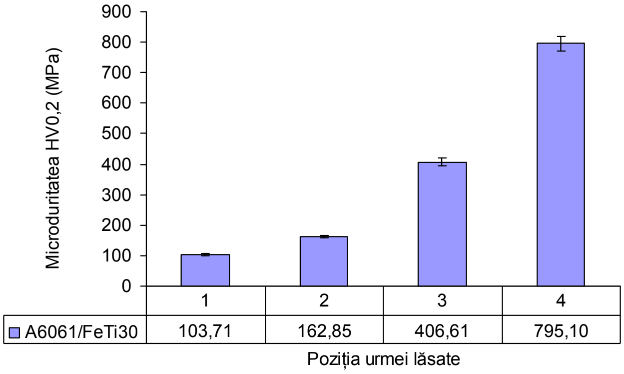
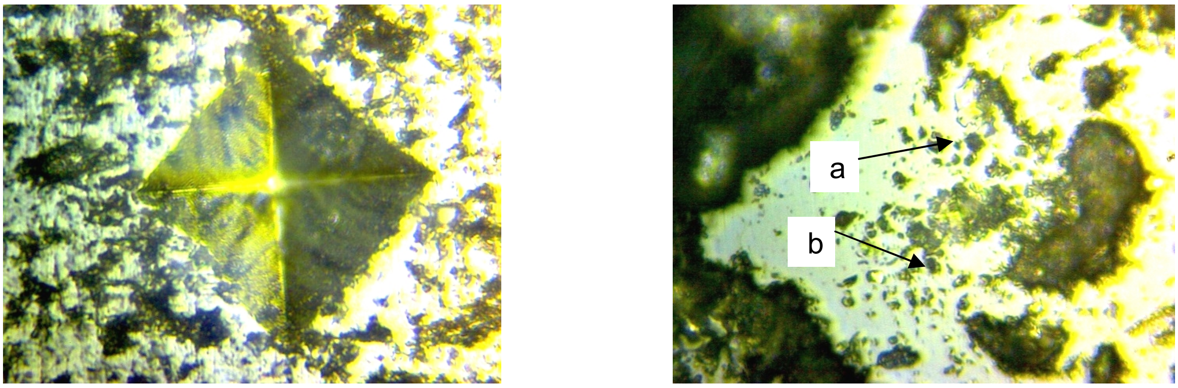


Figure 4.84. Variation of microhardness according to the position of measuring

In figure 4.84 a graph regarding the variation of microhardness is presented, following multiple determination. It is an extended domain of values, especially in the area which suggest modification appeared in chemical composition and appearance of new phases by diffusion.

For system A6061/SiC



1 2

Figure 4.86. Appearance indentation on microhardness determination for composite material with metallic matrix and refractory granular particles SiC

1 – footprints left on the aluminum alloy; 2. a,b - footprints left on SiC particle

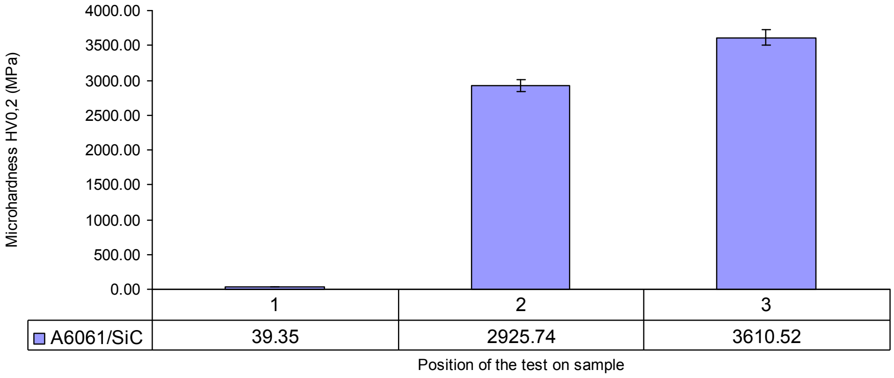


Figure 4.87. Variation of microhardness according to the position of measuring on sample

4.11.1. Partial conclusions regarding the determination of microhardness of composite material

1. Microhardness Vickers of matrix has specific values according to properties and nature of particles dispersed in metallic matrix. Differences are due to obtaining process and phenomena occurring at the interface matrix – particles. Metallic matrix (A6061) is a ductile material, soft and following melting process its surface is often covered with oxide Al_2O_3 which represents a physico-mechanical barrier.
2. Granular particles are in general fragile, hard with medium values of microhardness, and fragile character, hardness is confimed by the appearence of cracks in the macrostructure and microstructure analysed.
3. Interface is the area with the most important impact in obtaining some acceptable values of microhardness. Physico – chemical process from obtaining composite material influences the chemical, mechanical andthermodynamical properties at interface, properties which sometimes may lead to hindering the compatibility of phases. Thus: by formation of phase Al_2O_3 which is a physico-mechanical barrier, and capture of chemical elements from the atmosphere (oxygen, nitrogen and moisture) lead to appearence of defects in volume: micro and macropores.
4. Because these assessements are made with respect to the thermodynamic conditions and local processes, some aspects can vary because the process of elaboration of composite material is a dynamic process with global character.

4.12. Structural analysis by x-ray diffraction of composite materials

Analysis by diffraction with x-ray aims at highlighting the phases and structural modification which can appear in the studied systems. X-ray spectra have been recorded on room temperature with the help of a diffractometer DRON 3 equpped with a x-ray tube anode of Co ($\lambda_{K\alpha}$ = 1,7903 $\overset{o}{A}$). A field of measurement = 15 – 80° was assessed, and step and measuring time were 0.02° and respectively 3s.

Using an software MATCH 3 – demonstration version of phase analysis and free data bases **Crystallography Open Database**, diffraction spectra for were analysed: aluminium alloy A6061, refractory particles FeSi45, FeTi30 and SiC and composite materials A6061/FeSi45, A6061/FeTi30 and A6061/SiC.

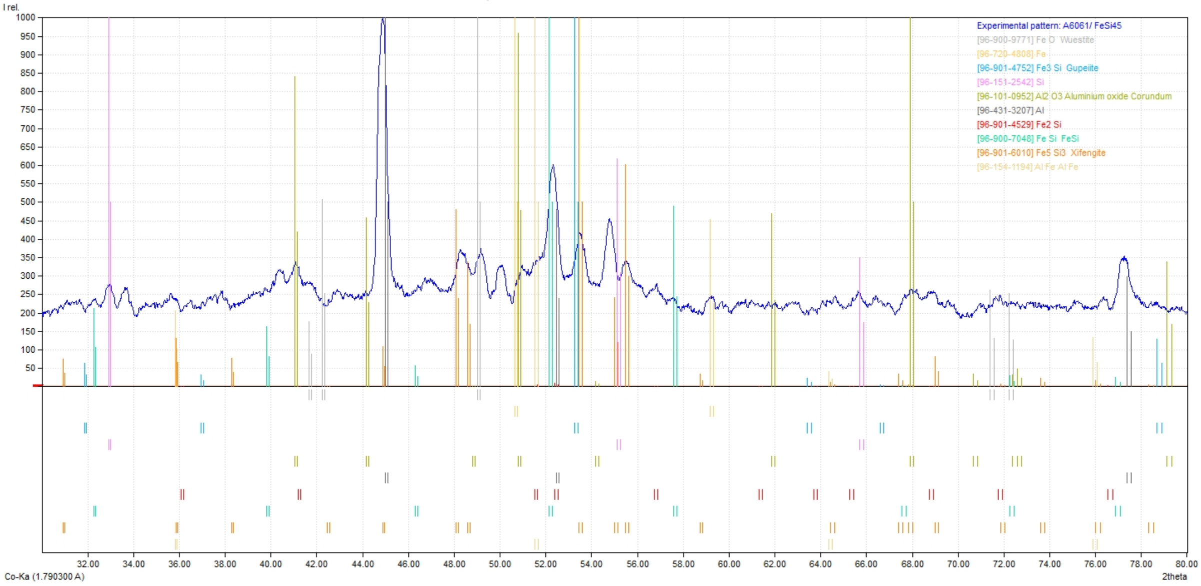


Figure 4.91. Diffractogram and difraction data, sample A6061/FeSi45

Figure 4.91 identifies the characteristic peaks of composite material with metallic matrix alloy A6061 and refractory particles of FeSi45: chemical elements aluminium, iron, silicon, specific oxides of elements such as: αAl_2O_3, SiO_2, FeO, Fe_2O_3, compounds FeSi, Fe_2Si, Fe_5Si_3 specific ferroalloy FeSi45 and intermetallic compound $FeAl_3$ [216] are identified. Thermodynamic conditions of temperature and pressure as well as duration time of composite material have favoured the appearance of new phases at interface, following bidirectional diffusion process.

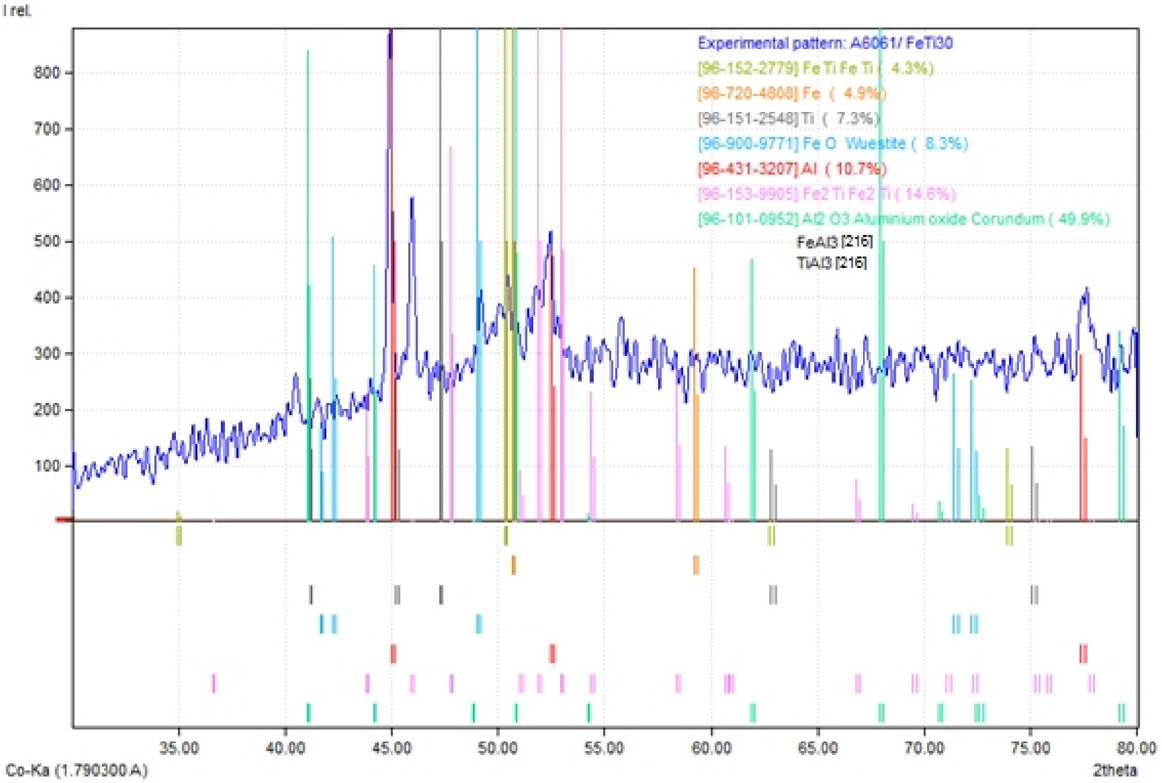


Figure 4.93. Diffractogram anddiffraction data, sample A6061/FeTi30

In diffractogram from figure 4.93 are identified the characteristic peaks of composite material A6061/FeTi30 with metallic matrix and dispers particles. Also highlighted are the specific oxides of chemical elements: FeO, Fe_2O_3, TiO_2, Ti_2O_3.

Thermodynamic conditions (temperature and pressure) of obtaining composite materials as well as reduced duration time, have limited the chemical reaction at interface, while still having conditions for diffusion of aluminium from melt in interface area and iron and titanium in alloy melt, may result intermetallic compound $FeAl_3$, $TiAl_3$[216].

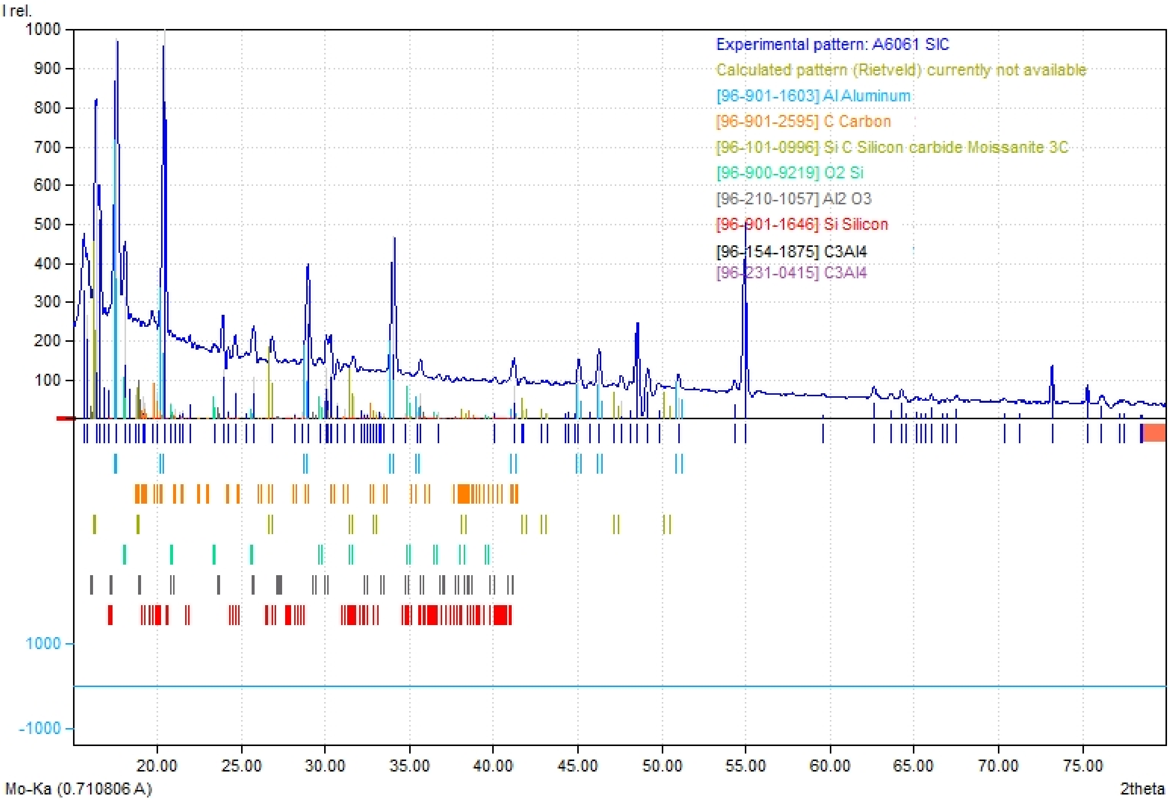


Figure 4.95. Diffractogram and diffraction data, sample A6061/SiC

Figure 4.95 presents the characteristic peaks of composite material from system A6061/SiC with granular particle of SiC. Analysing diffractogram also highlighted phases aluminium, silicon carbide. There is an additional phase, aluminium oxide, which is at interface solid/liquid and phase Al_4C_3.

4.12.1. Partial conclusions regarding analysis by x-ray diffraction of composite material

1. Structural analysis by x-ray diffraction method (XRD analysis), highlights the coresponding phases of composites of type A6061/FeSi45, A6061/FeTi30 and A6061/SiC.
2. The occurrence of new phase $FeAl_3$ is confirmed, which is formed at temperature 730°C and melting time of 30 min. For system A6061/FeTi30 is highlighted the formation of solid solution α (of Fe in Al) from eutectic $FeAl_3$ + (Al) at temperature 665°C and compound $TiAl_3$ formed at temperature 665°C. For system A6061/SiC is highlighted the formation of compound Al_4C_3 due to reduced solubility of carbon in aluminium.
3. In correlation with technological role of composite materials, we can make the observation that occurrence of intermetallic compound in large quantity is not recommended because newly complex chemical bond harden deoxidation and alloying process by necessity of suplimentary consumption of thermal energy for breaking complex chemical bonds and can determine arise in impurities in composite material.

CHAPTER 5
RESEARCH REGARDING SUPERFICIAL TENSION AND DYNAMIC VISCOSITY OF COMPOSITE MATERIALS A6061/FeSi45, A6061/FeTi30 AND A6061/SiC UNDER LIQUID STATE

Technological processes are characterized by surface phenomena such as: coalescence, embedding or expulsion of solid particles in granular state, adsorbtion and desorbtion of gases, crystallization and modification of interaction structure with installation surfaces and casting molds [217].

5.1. Superficial tension

Boundary layer and volumic phase which come in contact, in conditions of temperature and pressure from the elaboration, fundamentally influence the obtaining of composite materials with metallic matrix.

5.2. Contact angle between liquid alloy and solid phase

The degree of wetting of a solid particle in granular state by metallic melt can be appreciated on the bases on values of contact angle, θ, (figure 5.1), which is used in Young equation:

$$\sigma_{sg} = \sigma_{lg}\cos\theta + \sigma_{sl} \qquad (5.1.)$$

or:

$$\cos\theta = \frac{\sigma_{sg} - \sigma_{sl}}{\sigma_{lg}} \qquad (5.2.)$$

where indices s, g and l refer to solid, gas and respectively liquid.

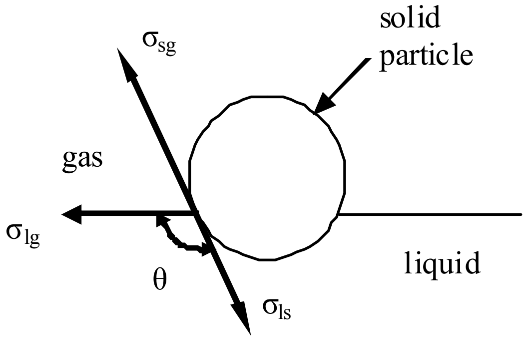


Figure 5.1. Contact angle between a solid particle and an liquid

5.3. Experimental method for superficial tension determination

Experimental determination of superficial tension,in the present paper, was made by imobile droplet method, known in literature under the name of "sessile drop".

Base plate by which droplet is formed is from refractory particle from composite material, namely FeSi45, FeTi30, SiC and spectral purity graphite (to have a reference with literature data).

The droplet placed on horizontal plate has at equilibrium the shape presented in figure 5.2.

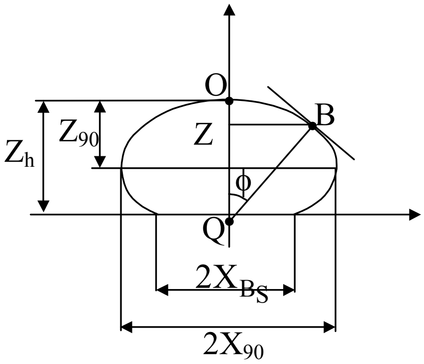


Figure 5.2. Transversal section in vertical plane (meridional plane) by sessile drop placed on plate (droplet of metal which do not wet the material of plate) [217]

For simplifying the expression, by convention a parameter called shape factor is introduced, given by the relation:

$$\beta = \frac{\rho b^2 g}{\sigma} \tag{5.10}$$

and Laplace equation and his form (5.10) dimensionless take the form:

$$\frac{1}{R/b} + \frac{\sin\phi}{x/b} = 2 + \beta \cdot \frac{Z}{b} \tag{5.11}$$

Equation (5.11) describes the surface curvature meridional section of droplet seated, containing indirect superficial tension σ, through size factor b and shape factorβ.

This is a differential second-degree equation with two unknowns and is solved only numerically by procedure of succesive approximation.

The solutions were obtained by using tables authors F. Bashforth and J.C. Adams [218] of some empirical relations. F. Bashforth and J.C. Adams used solving numerical method in imposed conditions with successive increments and were obtained numerical solution used on achieving tables for:

- ratio X/Z and values of β, for $\phi = 90^0$;
- ratio X/b and Z/b for different values of shape factor βand for angle ϕ.

Measurement of the above mentioned sizes, dimensions and angles is achieved using photograph of sessile drop.

5.4. Experimental installation designed for determination of superficial tension and contact angle

Experiments were performed in the laboratory of alloy elaboration and casting at Dunărea de Jos University of Galaţi, on a facility specifically designed for this purpose.

By abiding by the principles of construction and operation of the measuring device for determining surface tension by sessile drop method and one imagined by Kingery and Humenik, such equipment has been designed [219].

The system consists of a heater, a mechanical vacuum pump, a site with possibility to create vacuum and a heated droplet placed on a support and connected to the vacuum system as shown in the diagram in figure 5.3. The heating source is represented by a low voltage transformer 1 to 30 V and high amperage 1 – 30 A to adjust.

Figures 5.3 presents installation of determination of superficial tension

Data extracted are: droplet shape with X_{90} and Z_{90} coordinates to calculate the superficial tension and contact angle.

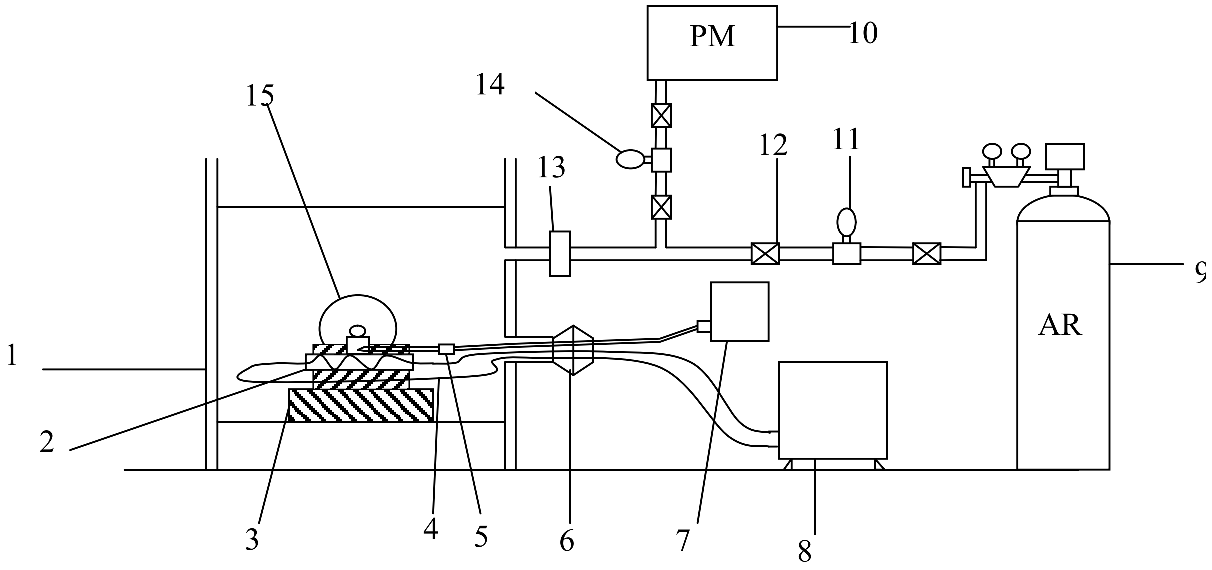


Figure 5.3. Installation for determination of surface tension:

1 – vacuum chamber, 2 – quartz tube, 3 – medium heating element, 4 – heating element (kanthal wire), 5 – Cr – Al thermocouple, 6 – feedthrough for power and temperature, 7 – digital multimeter (measuring temperature and voltage),8 – power supply(transformer), 9 – argon container, 10 – vacuum pump,11 – gauge, 12 – valve manually, 13 – vacuum and pressure feedthrough, 14 – vacuummeter, 15 – alloy drop [220]

5.5. Experimental determination of superficial tension and contact angle

Superficial tension was determined by using materials which have been used for obtaining composite material, whose chemical composition is given in table 5.1.

Table 5.1. Chemical composition of metallic alloys

No.	Symbol	Chemical composition (%)							
		C	Si	Ti	Fe	Cr	Mn	Mg	Al
1	Graphite	99.9	-	-	-	-	-	-	-
2	FeSi45	-	43.6	-	55.53	0.33	0.54	-	-
3	FeTi30	-	6.24	34.87	53.67	-	0.67	-	4.55
4	SiC	30	69.99	-	-	-	-	-	-
5	A6061	-	0.8	0.1	0.39	0.04	0.01	0.8	97.86

Considering the role of the degree of wetting of solid particles, by the melt metallic matrix (aluminium alloy) assessed based on the value of the contact angle from equation of Young the contact angle contained in table 5.2 were determined by sessile drop method, with the help of designed and manufactured installation.

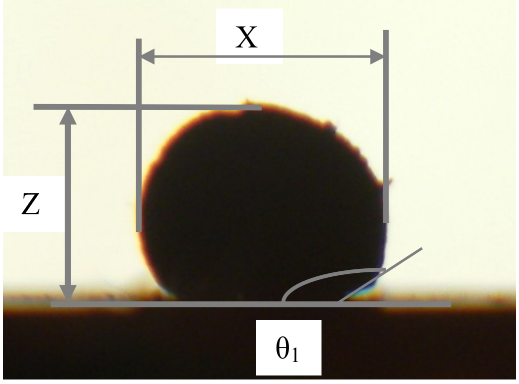


Figure 5.6. Droplet of alloy A6061 ongraphite support in vacuum, at temp.= 730^{o}C

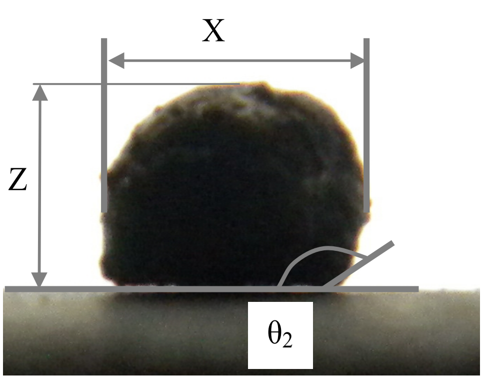


Figure 5.7. Droplet of alloy A6061 on graphite support in normal atmosfere, on temp. = 730^{o}C.

Figure 5.6 presents an image of a sessile drop, vacuum condition of 0.1 mbar, at temperature 730^{o}C. This has a high coefficient of sphericity X / Z = 1.16518; Spectral purity graphite support was prepared by mechanical polishing for reducing the roughness and for not influencing the shape of droplet during the experiment. This allowed the reference to data from literature for validation of results [222].

In figure 5.7 an image of a sessile drop, in normal atmosfere conditions is presented. This has a high coefficient of sphericity X / Z = 1.15569, it can be noted that the surface is discontinuous, showing intense oxidation, such as the presence of oxide changes superficial tension and contact angle.

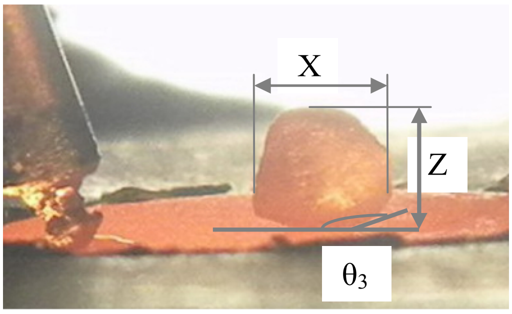


Figure 5.8 Droplet of alloyA6061 on support*of FeSi45 in vacuum, temp. = 730^{o}C*

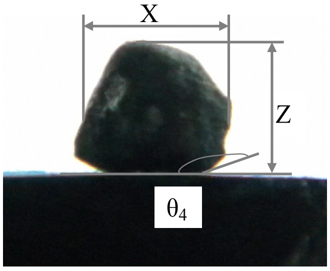


Figure 5.9. Droplet of alloy A6061 on support of FeSi45 in normal atmosphere, temp. = 730^{o}C

Figure 5.8 presents the aspect of droplet of liquid alloy formed on support FeSi45 in vacuum. The support was preliminarily prepared by mechanical polishing for reducing roughness and for the reduction of energy consumption, due to level diferences, with direct effect on formation of a higher angle.

In figure 5.9 a droplet of liquid alloy formed on support FeSi45 is presented. The sample was heated and cooled in normal atmosphere at 730^{o}C. The effect of normal atmosphere on droplet consists in the formation of a film with enlarged thickness, as a result of interaction of oxygen from atmosphere with suprface of liquid alloy and formation of aluminium oxide and magnesium which, in general, is not continuous.

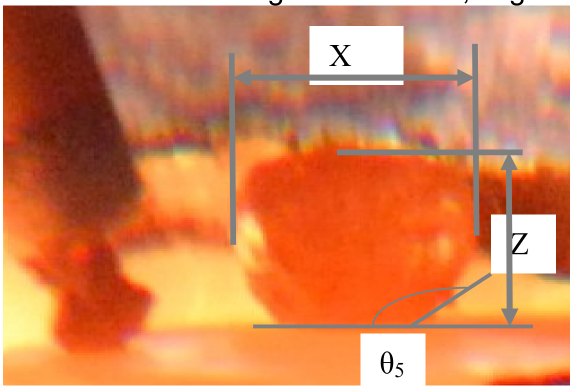


Figure 5.10. Droplet of alloy A6061 on support of FeTi30 in vacuum, temp. = 730^{o}C

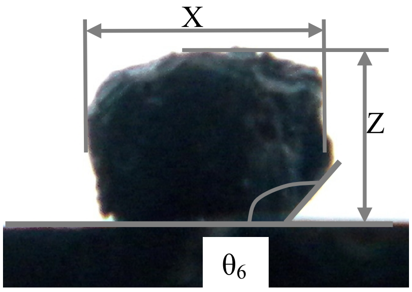


Figure 5.11. Droplet of alloy A6061 on support of FeTi30 in normal atmosphere, temp. = 730^{o}C

In figure 5.10 the aspect of droplet of liquid alloy formed on support of FeTi30 is presented. This was prepared by mechanical polishing for reduction of roughness and of the difficulties which may appear, so that the droplet of alloy to cover eventual creases present on surface.

In figure 5.11 the form of a droplet of liquid alloy formed on an support of FeTi30 is presented. Sample was obtained in condition of normal atmosphere.

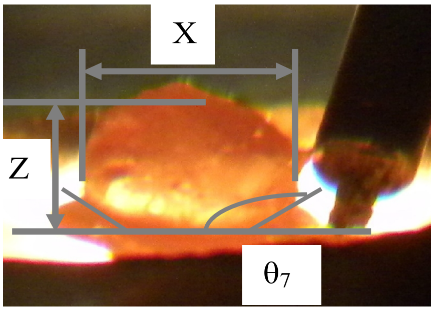


Figure 5.12. Droplet of alloy A6061 on support of SiC in vacuum, temp. 730°C

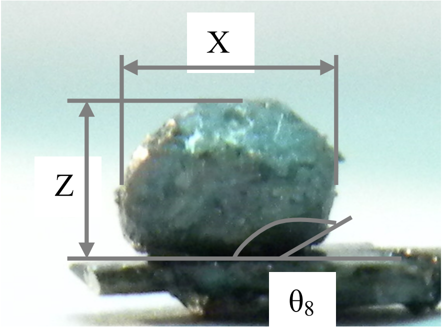


Figure 5.13. Droplet of alloy A6061 on support of SiC in normal atmosphere, temp. 730°C

In figure 5.12 the shape of droplet ofliquid alloy on support of SiC is presented. The support of SiC has a composition identical with the SiC particles from elaboration of composite material.

Figure 5.13 presents the shape of droplet of liquid on support of SiC in normal atmosphere conditions in which presence of oxygen significantly enlarges thickness of aluminium oxide layer and thus gets worse condition of formation of composite material.

Table 5.2. Surface tension of aluminium alloy determined experimental by sessile drop method

No	Droplet	X_{90}	Z_{90}	b	β	σ	θ	g	ρ_{Al}	Mass	Temp
		(mm)		-	-	(mNm)	(°)	(m/s^2)	(kg/m^3)	(g)	(°C)
1	A6061/graphite in vacuum	4.7571	4.0827	5.331	1	751	θ_1.142.5	9.8	2700	0.024	730
2	A6061/graphite in normal atmosphere	4.7574	4.1165	5.373	1	763	θ_2. 145	9.8	2700	0.024	730
3	A6061/FeSi45 in vacuum	4.8495	4.1999	5.4779	1	794	θ_3. 132	9.8	2700	0.025	730
4	A6061/FeSi45 in normal atmosphere	4.8708	4.2184	5.502	1	801	θ_4. 134	9.8	2700	0.025	730
5	A6061/FeTi30 in vacuum	4.8281	4.1814	5.4537	1	787	θ_5. 136	9.8	2700	0.018	730
6	A6061/FeTi30 in normal atmosphere	4.7979	4.15526	4.111	1	778	θ_6. 137	9.8	2700	0.018	730
7	A6061/SiC in vacuum	9.187	6.1445	14.248	7.7	697	θ_7. 147	9.8	2700	0.053	730
8	A6061/SiC în normal atmosphere	9.1946	6.1449	14.259	7.7	698	θ_8. 148	9,8	2700	0.053	730

Experimental research on manufactured installation have followed the creation of specific condition from elaboration. Thus, after making contact (particle/metallic matrix of A6061) by replacing graphite support with a material in solid state of ferroalloy and silicon carbide with a preparation of surface similary with graphite plate on an installation known from literature for determination of contact angle and other characteristics of droplet which is in contact with the support specialy prepared.

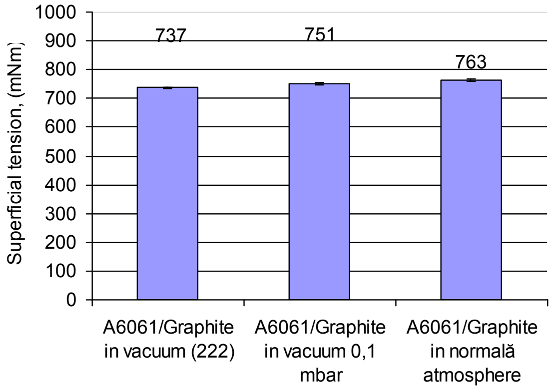


Figure 5.14. Superficial tension for system A6061/Graphite, in vacuum and normal atmosphere

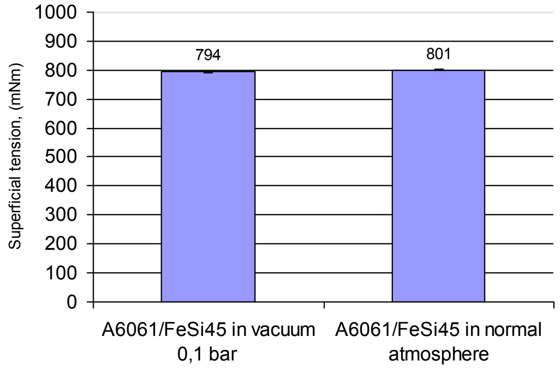


Figure 5.15. Superficial tension for system A6061/FeSi45, in vacuum and normal atmosphere

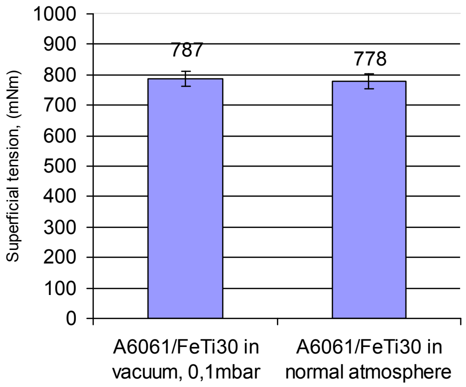


Figure 5.16. Superficial tension for system A6061/FeTi30, in vacuum and normal atmosphere

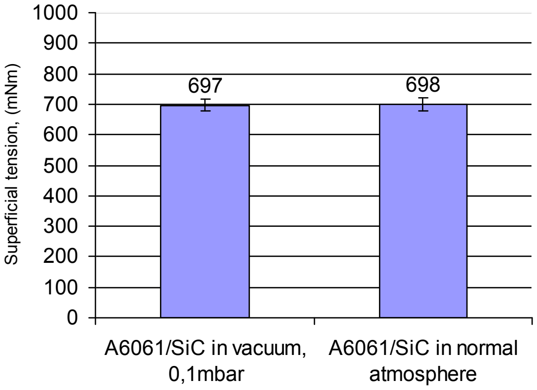


Figure 5.17. Superficial tension for system A6061/SiC, in vacuum and normal atmosphere

From the figures above results that superficial tension was determined in vacuum conditions, when it was used as a support material (particles) with the same chemical composition with those introduced in composite material melt, especially processed, is much lower than the value of superificial tension determined in normal atmosphere.

Following experimental determination, for calculation of contact angle in vacuum condition, at 730°C and in normal atmospheric condition, graphs from figure 5.18 – 5.21 were drawn.

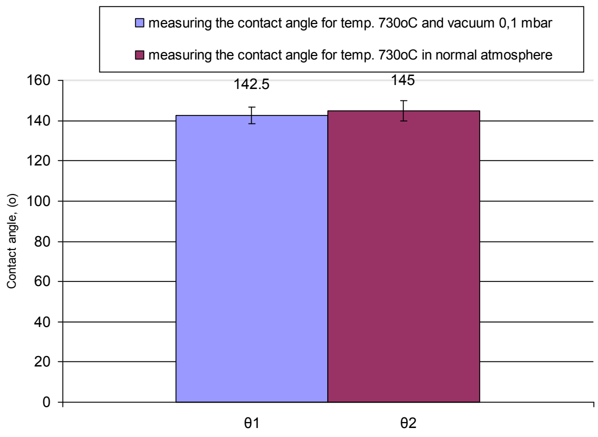


Figure 5.18. Variation of contact angle for alloy A6061 on graphite support

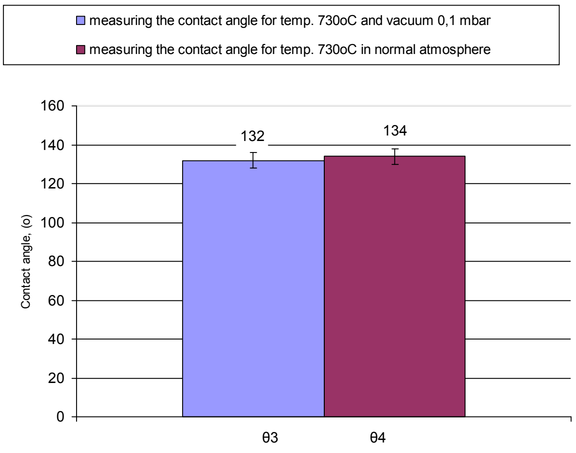


Figure 5.19. Variation of contact angle for alloy A6061 on support of FeSi45

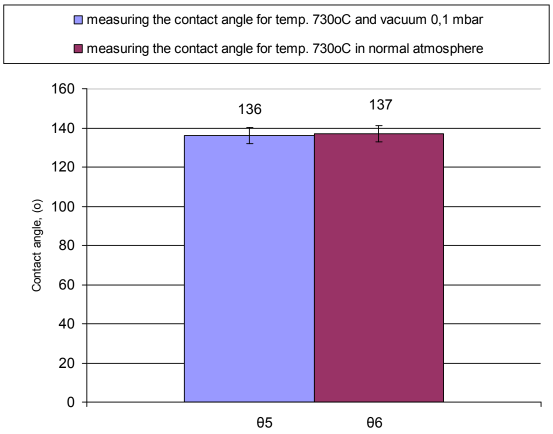


Figure 5.20. Variation of contact angle for alloy A6061 on support of FeTi30

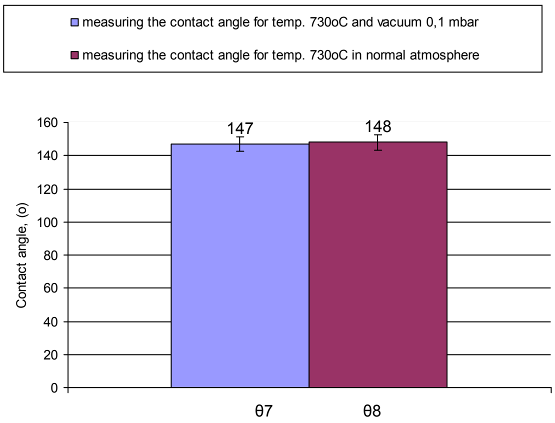


Figure 5.21. Variation of contact angle for alloy A6061 on support of SiC

By analysing graphs from figure 5.18 – 5.21, we observe that contact angle in normal atmosphere conditions is larger than contact angle in vacuum conditions. In normal conditions a thin film of aluminum oxide which enlarges contact angle is formed in a very short time.

Knowing the values of superficial tension as well as contact angle between the two phases, adhesion and cohesion can be determined, as well as spreading coefficient made between two phase solid and liquid.

Spreading coefficient S reflects the tendency of phase to spread on the surface of other.

$S = W_a – W_c$

where: S – spreading coefficient, N/m

Table 5.3. Adhesion, Cohesion and spreading coefficient for analysed systems

No.	Phases in system	Cohesion W_c [N/m]	Adhesion W_a [N/m]	Spreading coefficient S [N/m]
1	A6061/graphite, in vacuum 0.1mbar, T = 730^{o}C	1.502	0.1554	-1.3465
2	A6061/graphite, in normal atmosphere, T = 730^{o}C	1.526	0.1159	-1.4100
3	A6061/FeSi45, in vacuum 0.1mbar, T = 730^{o}C	1.588	0.2628	-1.3251
4	A6061/FeSi45, in normal atmosphere, T = 730^{o}C	1.602	0.2451	-1.3568
5	A6061/FeTi30, in vacuum 0.1mbar, T = 730^{o}C	1.574	0.2022	-1.3717
6	A6061/FeTi30, in normal atmosphere, T = 730^{o}C	1.556	0.2092	-1.3467
7	A6061/SiC, in vacuum 0.1mbar, T = 730^{o}C	1.394	0.1129	-1.281
8	A6061/SiC, in normal atmosphere, T = 730^{o}C	1.396	0.1060	-1.2899

By analysing data from table 5.3, we can observe that for analysed sistems A6061/grafit, A6061/FeSi45, A6061/FeTi30 and A6061/SiC result that $W_a < W_c$ (adhesion is numerically lower that cohesion) and the sign of spreading coefficient is negative. This indicates that droplet of aluminium alloy partially wetted the surface. This situation is specific to metallurgical systems in which wetting is not produced spontaneously but needs a supplementary energy from the exterior of the system and a time period for that the wetting to occur.

5.7. Partial conclusions regarding the determination of contact angle, superficial tension and interphasic tension

1. In order to obtain high yields of granular particles, it is important to know the values of superficial tension and contact angle in systems A6061/FeSi45, A6061/FeTi30 and A6061/SiC. These superficial sizes have been determined by using sessile drop method in

normal atmosphere and in condition of preliminary vacuum in an installation designed and manufactured by the author.

2. Experimental installation is based on principles of Kingery and Humenik, and is made from an vacuum chamber, an indirect heated table on which ferroalloy is laid, and on this is formed droplet from an aluminium alloy mass which will melt. Experimental device is completed with optical systems of view and photography of droplet. Experimental system is completed with the following important subsystems: system for adjusting temperature, adjusting vacuum (vacuum relay),system for capturing images as well as calculator for editing and interpreting contact angle and droplet dimensions.

3. Based on the experimental data resulted, we observed that the most favorable by superificial tension and contact angle for embedding are in order: A6061/FeSi45, A6061/FeTi30 and A6061/SiC.

4. Based on the superficial tension values and contact angle, the spreading coefficient was determined as a diference between adhesion and cohesion. In all cases, spreading coefficient is negative, resulting that droplet of alloy of aluminium will partialy wet the surface. This situation is specific to metallurgical systems, in which wetting is not produced spontaneously, requiring a supplementary energy from outside and a duration period for that wetting to be produced and thus particles to be embedded.

5.8. Determination of dynamic viscosity at the obtaining of composite materials from systems A6061/FeSi45, A6061/FeTi30 and A6061/SiC

Rheological aspects of complex systems, corresponding of studied composites, are different from those described by Newton Law.

For highlighting the particular character specific to every system, of which dynamic viscosity was studied, we used the rotational method of cylindrical concentric given by Searle model. This method entails using a stationary crucible in which a measuring sensor with calibrated form is introduced. Thus, the measuring sensor is introduced in liquid alloy and is rotated at a known rotation speed. The moment which opposes rotation is given by: internal friction between layers of liquid metal, by the system of particles in granular state, and the moment is directly proportional with dynamic viscosity. Thus, rheometry of system matrix/particles in granular state insoluble has been determined by using a Brookfield type mechanical viscometer.

For the determination of values for dynamic viscosity Newton Law was applied:

$$\eta = \frac{\tau}{\dot{\gamma}}, \quad [\text{mPa.s}] \qquad (5.20)$$

where: τ – shear stress, [g/cm·s]; $\dot{\gamma}$ - shear rate, $\left[\sec^{-1}\right]$

Shear stress:

$$\tau = \frac{M}{2\pi R_b^2 L}, \quad [\text{g/cm·s}] \qquad (5.21)$$

where: M - moment of force measured by apparatus, 10^{-7} [Nm] ; R_b - radius of the measuring sensor, [cm]; L – effective length, [cm]

Shear rate:

$$\dot{\gamma} = \frac{2\omega R_c^2 \cdot R_b^2}{x^2\left(R_c^2 - R_b^2\right)}, \quad \left[\sec^{-1}\right] \qquad (5.22)$$

where: ω –angular speed of measuring sensor, [rad/sec]; R_c – the inner radius of the cylindrical crucible, [cm]; R_b - radius of the measuring sensor, [cm]; x – location position of measuring sensor, [cm].

Dynamic viscosity of metals and alloys in liquid state was determined by rotative method. In an alloy mass in liquid state well determined from a quantitative point of view a body from a high temperature resistant materialis rotating at a certain speed (cylinder, propeller). The couple which is opposed to rotation is proportional with dynamic viscosity of melt.

Experiments were made in lab of alloys elaboration and casting at Dunărea de Jos University of Galaţi, on an installation designed and manufactured for this purpose.

The installation consists of a heating furnace with forced bars of SiC (four pieces) which is powered by a transformer with possibility to control temperature in the 0 – 1000°C domain, other components of installation are: stainless steel tube with the purpose of container where experiments are made, input system of inert gas (Ar), system for lifting – lowering of mechanic viscometer type Brookfield.

Figures 5.22 shows the installation for determination of dynamic viscosity.

Aluminium alloy in known quantity, 150g, is melted and brought to temperature 730°C (temperature of experiment). After temperature was set, a quantity of particles with known weight representing: 0; 0.5; 1; 1.5; 2; 2.5; 3 % (mass percent from quantity of melted alloy and with known granulation 0.04 mm; 0.4 mm; 0.8 mm) is introduced. Mixing aluminium alloy with refractory particles is perfomed under inert gas (argon) with protection role. The role of gas is to make an inert atmosphere above the melt in order to reduce impurification with oxygen by lowering partial pressure of oxygen. Also, it is aimed that layer of oxide aluminium which is formed above the balth, to not significantly influence measurements. Therefore, the quantity of oxide aluminium (Al_2O_3) should be diminished. Relative density of argon (γ_{Ar} = 1.38) [226] is larger than that of oxygen (γ_{O2} = 1) [226], therefore argon has the tendency to lay down on metal bath.

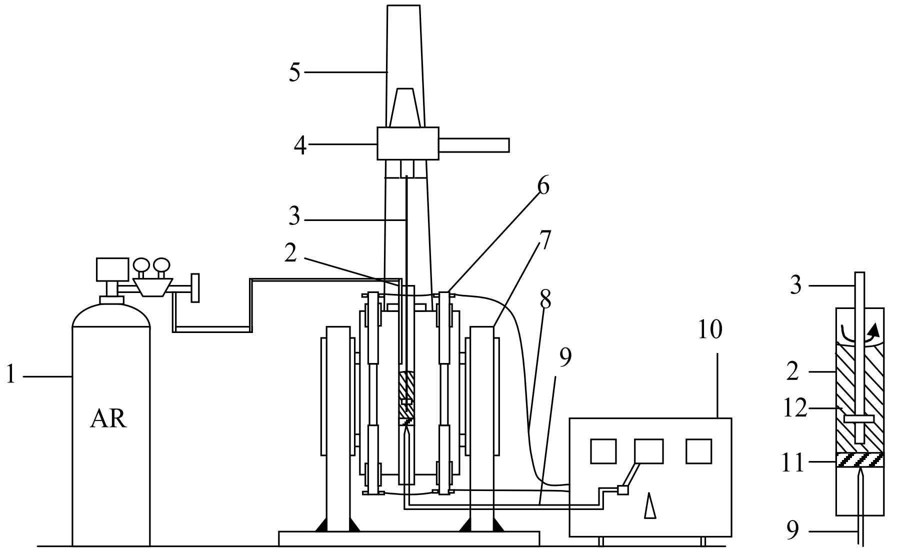


Figure 5.22. Installation for determination dynamic viscosity

1 – gas tank for Argon with reduction; 2 – stainless steel tube; 3 – agitator (measuring sensor); 4 – Brookfield viscometer; 5 – system for lifting – lowering viscometer; 6 – heating elements (forced barsof SiC); 7 –support system for furnace; 8 – power cables; 9 – thermocouple Cr – Al; 10 – transformer; 11 – graphite bottom; 12 – semi-solid metal

Figure 5.23. Installation for determination of dynamic viscosity

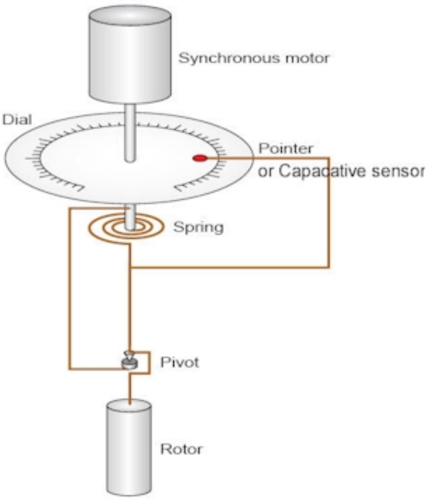


Figure 5.24. Representation of Brookfield viscometer [227]

Table 5.8. Constructive date of container where measurments of dynamic viscosity are made

No.	Effective lenght of the rod L	Radius of rod R_b	Radius container R_c	Radius at location x
	(mm)	(mm)	(mm)	(mm)
1	2	6.3	17.5	6.3

In table 5.8 we have constructive data ofcontainer where measurments of dynamic viscosity are made.

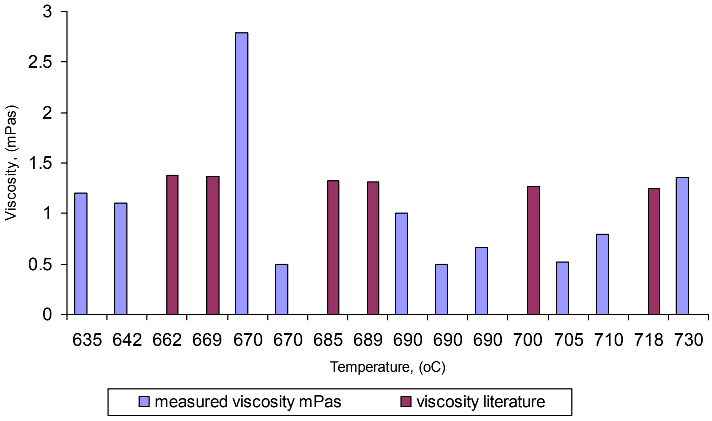


Figure 5.26. Variation of dynamic viscosity in function of temperature [229]

In figure 5.26 a comparison is made between the values of viscosity experimental determined and those from literature [229]. A good agreement of data can be observed, thus validating the results.

The effect of small concentration of solid particles in granular state is an indicator regarding influence on dynamic viscosity.

The generally accepted tendency is that in the moment of introducing material in solid granular state in matrix of composite material is by rising viscosity, but there can be situations in which, at low concentration due to a „bearing effect”, areas with internal friction lower, liquid medium and material under granular form, dropping viscosity are not significant.

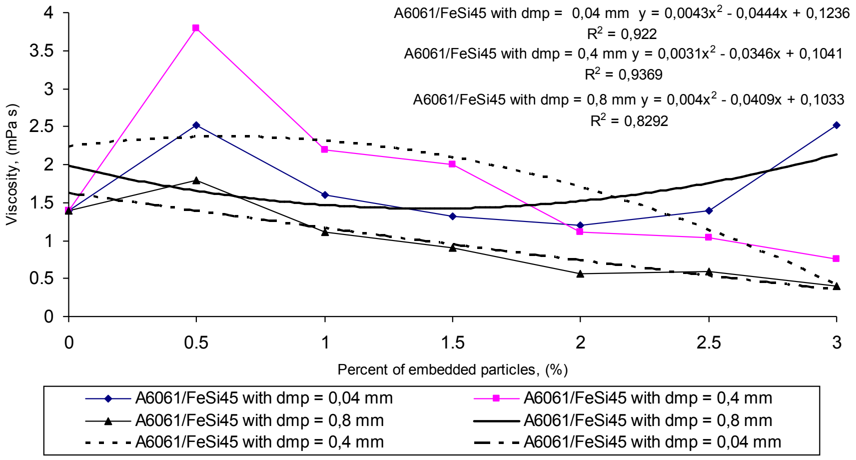


Figure 5.31. Variation of dynamic viscosity for three dimension of particle from system A6061/FeSi45, on T = 730°C

For system A6061/FeSi45, by analysing data from table 5.11 and figure 5.27 – 5.31 a slight rising tendency in case of dynamic viscosity for all melts of aluminium alloy with solid materials in granular state (0.8 mm, 0.4 mm and 0.04 mm) of FeSi45 can be observed.

Based on the obtained results, the functions of linear regression were determined;

For A6061/FeSi45 d_{mp} = 0,04 mm $y=0.0043x^2 – 0.0444x + 0.1236$, $R^2 = 0.922$
For A6061/FeSi45 d_{mp} = 0.4 mm $y = 0.0031x^2 – 0.0346x + 0.1041$, $R^2 = 0.9369$
For A6061/FeSi45 d_{mp} = 0.8 mm $y = 0.004x^2 – 0.0409x + 0.1033$, $R^2 = 0.8292$

From figure 5.31 results influence of granulation of material in solid state for low concentration, on viscosity, is ascending with granular particle dimension from 0.8 mm to 0.04 mm.

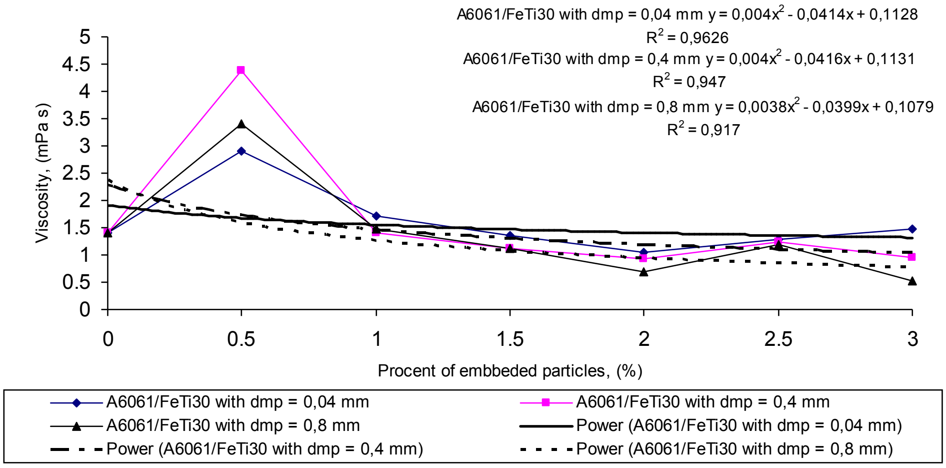


Figure 5.36. Variation of dynamic viscosity for three dimension of particle from system A6061/FeTi30, on T = 730°C

For system A6061/FeTi30, by analysing data from table 5.12 and figure 5.32 – 5.36, one observes a slight rising tendency in case of dynamic viscosity for all melts of aluminium alloy with solid materials in granular state (0.8 mm, 0.4 mm and 0.04 mm) of FeTi30.

Based on the obtained results, functions of linear regression were determined;

For A6061/FeTi30, d_{mp} = 0.04 mm $y = 0.004x^2 – 0.0414x + 0.1128$, $R^2 = 0.9626$
For A6061/FeTi30, d_{mp} = 0.4 mm $y = 0.004x^2 – 0.0416x + 0.1131$, $R^2 = 0.947$
For A6061/FeTi30, d_{mp} = 0.8 mm $y = 0.0038x^2 – 0.0399x + 0.1079$, $R^2 = 0.917$

From figure 5.36 results that influence of materials granulation in solid state (for low concentration) on viscosity is ascending from granulation 0.8 mm to 0.04 mm.

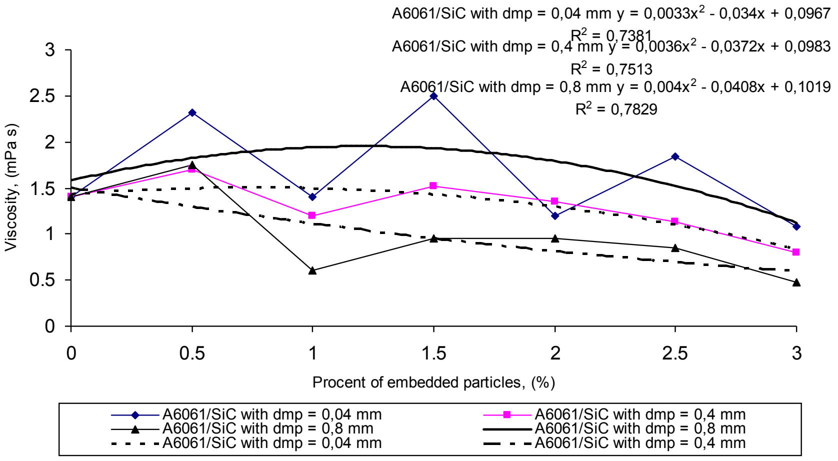


Figure 5.41. Variation of dynamic viscosity for three dimension of particle from system A6061/SiC, on T = 730°C

For system A6061/SiC, by analysing data from table 5.132 and figure 5.37 – 5.41, one observes a slight rising tendency of dynamic viscosity for all melts of aluminium alloy with solid materials in granular state (0.8 mm, 0.4 mm and 0.04 mm) of SiC.

For A6061/SiC, d_{mp} = 0.04 mm $y = 0.0033x^2 – 0.034x + 0.0967$, $R^2 = 0.7381$

For A6061/SiC, d_{mp} = 0.4 mm $y = 0.0036x^2 – 0.0372x + 0.0983$, $R^2 = 0.7513$

For A6061/SiC, d_{mp} = 0.8 mm $y = 0.004x^2 – 0.0408x + 0.1019$, $R^2 = 0.7829$

From figure 5.41 results that influence of materials granulation in solid state (for low concentration) on viscosity is ascending from granulation 0.8 mm to 0.04 mm.

5.8.1. Partial conclusions regarding determination of dynamic viscosity for the systems A6061/FeSi45, A6061/FeTi30 and A6061/SiC

1. Dynamic viscosity significantly influences the yield of embedding. The more viscosity is reduced, the more hydrodynamic conditions are created in order for granular particles to move in liquid and be embedded. Also, the rise of dynamic viscosity on some dimensions and concentrations of particles from melt which negatively influences their embedding.
2. In order to characterize composite material from the point of view of dynamic viscosity an installation using rotative method (Searle) was designed and manufactured.
3. Installation uses Brookfield viscometer, for which was adapted a thermal system made of crucible in which metallic melt is introduced whose temperature must be determined. Also, above metallic melt was introduced inert gas (argon) with protection role of bath surface and for the reduction of thickness of layer of Al_2O_3. The equipment contains subsystems for adjusting temperature at constant values, in order to allow determination of viscosity as precisely as possible.
4. A series of factors significantly influence dynamic viscosity, worth mentioning being: internal friction between layers of liquid aluminium, wall crucible roughness, temperature variation inside crucible (from the walls toward the crucible centre), internal friction between layers of liquid and granular particles as well as the number of particles introduced and their uniform distribution.
5. Calibration of measuring device was made by employing two methods: using glycerine with 99.5% concentration on known temperature and measuring dynamic viscosity of aluminium alloy (A6061) which are compared with data from literature. Results show a good agreement with data already published, which proves their validity.
6. Determination of dynamic viscosity is important in the case of obtaining composite materials by Stir Casting Vortex method because granular particles are introduced in small quantities to be assimilated in the matrix.
7. Research was carried out by introducing reduced mass percentage 0 – 3% particles of FeSi45, FeTi30 and SiC for analysing the influence of particles properties on dynamic

viscosity of metallic melts. For systems A6061/FeSi45, A6061/FeTi30 and A6061/SiC we observed that dynamic viscosity ascended with the decrease of medium diameter of particles (from d_{mp} = 0.8 mm to 0.04 mm).

CHAPTER 6
GENERAL CONCLUSIONS, ORIGINAL CONTRIBUTIONS AND FUTURE RESEARCH DIRECTIONS

6.1. **General conclusions**

1. Due to the fact that in extractive metallurgy, at steel making sorting is not provided on granulometric classes but only excluding from use for alloying or deoxidation processes of granular particles of ferroalloys type such as FeSi45, FeTi30 or silicon carbide SiC, with medium diameters under standard dimensions (under 3.5 mm), which have become in this way waste and pollute environment, research for efficient reuse of this refractory particles under other form,such as composite materials with metallic matrix and particles in granular state, has become compulsory.

2. Experimental research presented in this paper aimed at obtaining and characterizating composite materials with metallic matrix from aluminium alloy and refractory disperse particles FeSi45, FeTi30 and SiC by casting, using a known method, namely Stir Casting Vortex method (SCV) and a new method for mechanical mixing, respectively mixing with vibrations.

3. Part of the equipment and installations necessary for achieving the experiments and analysis, which were designed and manufactured in the frame of doctoral thesis are installations and equipment already extant in laboratories of Universitatea „Dunărea de Jos” University of Galați, especially at Faculty of Engineering, to which equipment from ICECHIM S.A. Bucureşti was added for thermal analysis.

4. Development of experimental research for establishing conditions for obtaining of composite materials with technological role for the yield of embedding of solid granular particles to be high and distribution to be as best as possible, was based on systems A6061/FeSi45, A6061/FeTi30 and A6061/SiC, and the technologies of elaboration were based on uniaxial vibration method on vertically using sandwich procedure. This method consists in embedding of a quantity of granular particles between two blocks of aluminium alloy A6061. The procedure has been proven valid by embedding up to 97% for FeSi45, up to 100% for FeTi30 and up to 99.64% for SiC, depending on particles granulation as well as on aplitudine and frequency of oscillations. It was also studied the possibility of using Stir Casting Vortex method on the same types of composites with matrix of aluminium alloy and granular particles, but satisfactory yields of embedding were obtained, respectively values between 30 ÷ 40%.

5.From all the granulometric classes analysed, of interest for the present topic are fractions with medium diameter of particles 0.8 mm, 0.4 mm and 0.04 mm for FeSi45, FeTi30 and SiC.

6. The chemical composition of solid materials in granular state of FeSi45, FeTi30 and SiC was determined in order to assess the influence of some elements from their chemical composition on metallic matrix (aluminium alloy A6061) at elaboration temperature of composites, which may influence embedding of particles in this metallic matrix, as well as their distribution in solidified material.

7. There were also determined the surface and volume of particles, number of particles on gram and the lenght of the channel which has to be occupied by liquid alloys with the purpose of appreciation of the infiltration yield of aluminium alloy between granular particles.

8. The author of the thesis designed and manufactured equipment for obtaining composite materials by Stir Casting Vortex method, which are based on the mechanical mixing of aluminium melted alloy and portioned introduction by refractory granular particles into vortex created by the stirrer. The shape of the propeller and the rotation speed of significantly favor yield of embedding by creating a vortex in the central area of the crucible.

9. The influence of the process parameters for obtaining highlighted that a longer mixing time is preferred in order to facilitate particle dispersion as good as possible in the melt, in order to achieve a proper yield of embedding, thus there have been tested three times of obtaining: 10 min. for A6061/FeSi45, 30 min. for A6061/FeTi30 and 15 min for A6061/SiC.For temperature of obtaining a temperature of approximately 700^{o}C has been set.

In the case of rotation speed, 3 rotation speeds were tested, namely 150, 170 and 420 rpm, noticing that the highest rotation speed (420 rpm) creates the most suitable conditions for a good embedding.

10. Chemical composition determination by XRF method shows that a significant variation of chemical composition of composites occurs depending on surface area on which the analysis is made and on the number of present granular particles (on a 10 mm^2 surface). Chemical composition determined by EDX method at particles surfaces, at interface particle/matrix and also in the area of metal matrix shows a variation of chemical composition and to localized diffusion around the particles where intermetallic or chemical compounds are formed,such as $FeAl_3$ for the system A6061/FeSi45, $TiAl_3$ for the system A6061/FeTi30 and Al_4C_3 for the system A6061/SiC.

11. Microstructure of samples of composites obtained by Stir Casting Vortex method presents aspects which depend on particles distribution in metallic matrix, on granular particles state, on obtaining conditions (temperature, rotation speed, duration) or on their clustering with negative consequences since a high quantity of gases adsorbed can be on their surfaces, which can, in turn, diminish the positive effects of complex deoxidation by rising hydrogen concentration in elaborated steel. For composite material to be efficient in deoxidation process, new intermediary phases with different behavior in steels deoxidized and alloyed are not desirable at interface metallic matrix/granular particles, because complex deoxidation of steels is made with aluminium, silicon, titanium and not with chemical or intermetallic compounds formed.

12. Obtaining process of composite materials by Stir Casting Vortex method should be directed in such way thus: rapid mixing in order to avoid formation of a large quantity of intermetallic compounds and segregation pronounced on height of solidified composite material.

13. Method of elaboration of composite material, with the use of mechanical mixing of metallic bath with refractory particles by vibration, shows some advantages over Stir Casting Vortex method, and may be recommended for testing on microproduction level.

14. Technology of obtaining of composite materials by mechanical mixing with vibrations is based on the arrangement of two blocks of aluminium alloy A6061 in overlapped layers (sandwich type).Granular particles of FeSi45, FeTi30 and SiC with medium diameter 0.8 mm, 0.4 mm and 0.04 mm have been brought to temperature of obtaining (730°C) and was vibrated uniaxially with known amplitude and frequency, for a determined time.

15. For the study of yield of embedding of granular particles as well as for chemical, mechanical and structural properties of obtained composites by this method, an installation has been designed and manufactured. This allows the study of physico – chemical processes from the composites' elaboration. The plant contains a system consisting of a vibrating table and a heating system and a crucible in which composite material is obtained, crucible which can be vibrated uniaxially on vertical plane.

16. For composites based on system A6061/FeTi30, it has been found that yield of embedding is favoured in case of large particles with d_{mp} = 0.8 mm then when amplitude and frequency are high in comparison with particles with d_{mp} = 0.4 mm, when it was found that when the amplitude is high and frequency is low, yields are very high. This thing is explained by the fact that, by lowering d_{mp} density of the granular particleshas apparently declined , which may explain maintaining a long time of particles without their decantation to occur, so are created for conditions of infiltration for aluminium alloy among solid particles.

17. For very small particles of FeTi30 (d_{mp} = 0.04 mm) yield of embedding have been much lower than for particles of 0.8 mm and 0.4 mm, due to the fact that on very fine granulation specific surface contact has greatly increased, which has led to a clustering tendency of particles, so that for large quantities of particles, their agglomeration is carried out and they have the tendency to separate in slag.

18. For composites type A6061/SiC obtained by vibration method, when average diameter of particles has been of 0.8 mm, yields of embedding have been much higher reaching up to values of 99.64%, in the case of amplitude and frequency much lower of vibrations, while for particles with d_{mp} = 0.4 mm, yields of embedding were better throughout the studied area.

19. At all three systems elaborated A6061/FeSi45, A6061/FeTi30 and A6061/SiC for all granulations: 0.8 mm, 0.4 mm and 0.04 mm limited diffusion processes took place at interface, highlighted, by chemical composition determination in three areas (matrix, interface, particle) observing, for exemple, on concentration variation of aluminium in respective areas. Also, at chemical analysis a variation of oxygen concentration was observed, ascertaining a high concentration on particle (so there is an oxidated surface), and in metallic matrix, as a result of presence of Al_2O_3.

20. Distribution of chemical elements determined by EDX method has shown, based on color tones, an area with high concentrations of main chemical element (aluminium) on particle but also for silicon distribution both in matrix as well as on the particles.

21. Macrostructural analysis has shown a diversity of situations, such as areas with homogenous dispersion, but also areas with decanted particles to the bottom of sample as a result of density difference. Pores were found both around particles and in sample volume, a result of insufficient mechanical mixing, which usually favors leaving the melt. It may also be observed the formation of areas with high particle concentrations and areas without particles, as a result of amplitude and frequency that have allowed embedding particles but not succeeded in their dispersion. Microscopical analysis (either optical or SEM) shows granular particles embedded, with polygonal shapes for FeSi45 and FeTi30 with rounded edges and elongated shapes for SiC with sharp edges. Also, we can observe a broad field of dimensions, possibly due to mechanical breakage produced during the obtaining process.

22. Installation of elaboration based on mechanical mixing of metallic bath with refractory particles by inducing vibrations displays superior advantages to the Stir Casting Vortex method and can be recommended for testing at microproduction level.

23. DSC analysis for systems corresponding composite materials studied in the paper aimed at determining multiple process parameters: temperatures of beginning transformation, ending of transforming, peaks temperature, free enthalpy (ΔH), and crystallinity.

24. Microhardness Vickers determination is a useful method in evaluating mechanical properties in correlation with material microstructure. Microhardness Vickers of matrix has specific values in function of properties and nature of disperse particles in metallic matrix namely: for composite A6061/FeSi45 microhardness is 94.1 MPa, for A6061/FeTi – 100.31 MPa, and for A6061/SiC – 38.8 MPa. These differences are due to process of obtaining composite materials and to phenomena that take place at the interface matrix - particles. Metallic matrix (A6061) it is a soft ductile material, and after the melting process his surface is often covered by oxide Al_2O_3 which represents a physico-chemical barrier.

25. Phase analysis, achieved by x-ray diffraction method (XRD analysis), has emphasized the appropriate phases for composites type A6061/FeSi45, A6061/FeTi30 and A6061/SiC.

26. Achieving of some high yields of embedding of granular particles requires knowledge of superficial tension values and contact angles in the systems A6061/FeSi45, A6061/FeTi30 and A6061/SiC. Thus, these have been determined by using sessile drop method in normal atmosphere and in preliminary vacuum conditions. For these, an experimental installation was designed and manufactured.

27. Experimental installation was designed by the author of the present doctoral thesis and is based on principles of Kingery and Humenik, consisting of a vacuum chamber, an indirect heated table on which ferroalloy is placed, and on this is formed the droplet from aluminium alloy mass which will melt. Experimental device is completed with visualization optical systems and photography to allow determination of surface tension using Laplace formula and Adams and Bashforth tables. The experimental system has the following important subsystems: systems for adjusting the temperature, adjusting the vacuum (vacuum relay), system for recording of images and a computer for editing and interpreting contact angle as well as droplet dimension.

28. Dynamic viscosity significantly influences the yield of embedding. The more viscosity is reduced, the more hydrodynamic conditions are created in order that granular particles to move in liquid and to be embedded. Also, rise of dynamic viscosity on some dimensions and concentrations of particles from melt which negatively influence their embedding.

29. In order to be able to characterize composite materials from the dynamic viscosity point of view of an installation using rotating bulb method (Searle) was designed.

30. The main factors which control the properties of metallic composite materials obtained using the applied technologies of elaboration include: distribution as uniform as possible of solid particles in granular state, process of wetting of granular materials by metallic matrix, chemical reactivity at interface particle/matrix and content of pores result after obtaining process.

31. Analysis and characterization of composite materials focused on determination of physico-chemical, structural and mechanical properties of solid materials by different methods, such as: SEM, EDX, XRF, DSC, XRD, microhardness HV property. For composites in liquid state surface tension and dynamic viscosity were determined, because we took into consideration their correlation with experimental results regarding macrostructure and microstructure of sample composites, with those resulted from the achievement of chemical, structural and thermal analysis.

6.2. Original contributions

The present paper is the result of different activities which the author has carried out during the training sessions, achievement of research and interpretation of the obtained results. The majority of data presented throughout the thesis belong to the category of author's personal contributions:

1. Achievement of bibliographical study presented, especially in the first chapter;

2. Identification of the research problems appeared in the practice of making steels and development of efficient solutions. The problem generated by the crushing of ferroalloys and the necessity of solving this problem by embedding small particles into a metallic matrix of aluminium alloy A6061 were analysed. Obtaining composite materials with technological role, which would have had consequences in the reuse of a high proportion of ferroalloys FeSi45 and FeTi30, as well as silicon carbide (SiC), in granular state and which constitute reusable materials which have to be deposited with high costs, or are used inefficiently in the process of steel-making.

3. Development of experimental research for establishing technologies of obtaining of composite materials with technological role (for deoxidation of steels) leading to high yields of embedding of granular particles for systems that are not presented in specialty literature:

- Composite materials based on the systems A6061/FeSi45, A6061/FeTi30 and A6061/SiC were elaborated, by vibration method uniaxially on vertical plane using sandwich procedure, which consists in embedding of a quantity of granular particles between two blocks of aluminium A6061. The proposed method requires mechanical mixing and controlled cooling in order to avoid the formation of chemical or intermetallic compound, an unwanted effect, because it is prognosed that composite material to be used at combined deoxidation and alloying of steels;
- The possibility of using Stir Casting Vortex method on all systems of composite materials, but for satisfactory yields of embedding (values between 30 ÷ 40%) was confirmed.

4. Obtaining and data interpretation regarding composite materials elaborated have confirmed the recycling posibility and the superior recovery of some standard granular particles of ferroalloys and silicon carbide with medium diameters under 3.5 mm in the process of deoxidation and alloying steels. Using these composites, a quantity of aluminium is in fact introduced into metallic bath, a quantity which will be subtracted from that needed to the deoxidation processes of, and refractory materials in granular state will contribute on advanced deoxidation of steels. Obtaining of such composite materials for which the research was pursued in the frame of doctoral thesis can be achieved close to the technological line of steel-making.

5. Designing and achieving equipment and installations used for obtaining and studying of some properties of composites in solid or liquid state, focused on the following directions:

- Experimental system in order to achieve composite materials A6061/FeSi45, A6061/FeTi30 and A6061/SiC by Stir Casting Vortex method was based on equipment of elaboration and on subsystems of control and automatization designed and entirely produced for the development of experimental research. Control equipment and automatization contain: measuring system of speed, regulating system for temperature and testing a number of stirrers. Installation Stir Casting Vortex designed and manufactured led to good results during experimental research.

- A new technology and the appropriate equipment for elaboration of some composites which have on base systems A6061/FeSi45, A6061/FeTi30 and A6061/SiC have been designed. The installation which allowed the study of physico-chemical processes from obtaining composites contains a system of vibrated table (extensively presented in chapter 4), a heating system and a crucible in which composite material is realized, and which can be uniaxially vibrated on vertical plane. Vibrated table allows the achievement of some frequencies between 0 ÷ 80 Hz and amplitude between 0 ÷ 5 mm, in conditions in which the oscilating table is of max. 20 kg. The equipment is semi–automated with timing system, regulating system for temperature, frequency measuring of vibration and oscillation amplitude. These characteristics have allowed the accomplishment of the proposed experimental plan;

6. Experimental research regarding the behaviour of granular particles (ferroalloys or silicon carbide) in the process of elaboration of composites type of metallic matrix - granular particles, were correlated with results of analysis regarding variation of superficial tension in the respective heterogeneous systems. An experimental equipment based on the principles of Kingery and Humenik was designed and manufactured, which consists in a vacuum chamber and a system of indirect heating, on which a calculated quantity of aluminium alloy is placed, which by melting realizes a droplet of alloy for study. The experimental device is completed with optical systems for visualizing and photography for allowing later determination using formulas of Laplace and Adams and Bashforth tables of superficial tension. The experimental installation has a number of important subsystems which allow adjusting the temperature, adjusting the vacuum, capturing droplet image and a computer for editing and interpreting the contact angle as well as the dimensions of droplet.

7. The determination of viscosity of metallic melt in function of time was based on the method of rotating bulb (Searle). Experimental equipment using Brookfield viscometer was designed and manufactured, for which a thermic system was adapted, consisting in a crucible in which metallic melt is introduced, whose the viscosity has to be determined. Also, inert gas (argon) was introduced above of metallic melt with protection role of bath surface and to reduce the thickness of the film Al_2O_3. Metallic alloy which has represented metallic matrix of composites elaborated and investigated was melted and was maintained in temperature ranging from 660 ÷ 750°C. The equipment contains subsystems of adjusting the temperature at constant values, and has allowed the determination as accurate as possible of viscosity.

8. Obtaining and characterization of composite materials has focused on the determination of physico-chemical, structural and mechanical properties of solid materials used on elaboration or resulted following solidification of metallic melts, studies made by different methods, such as: SEM, EDX, XRF, DSC, XRD, microhardness HV. For composites in liquid state were determined surface tension, dynamic viscosity, because we have taken into consideration the corelation and interpretation of results about macrostructure and microstructure of samples of composites with data resulted of chemical analysis on small areas (EDX analysis) and large areas (XRF analysis), with those regarding thermal phenomena that accompany structural transformations at interface (DSC analysis), rising microhardness at interface particle/matrix and appearance of new phases represented by intermetallic or chemical compounds, such as: $FeAl_3$, $TiAl_3$, Al_4C_3.

6.3. Future directions of research

Further research with the purpose of a more thorough discussion on the issues obtaining composite materials with metallic matrix and granular particles:

1. The obtaining and characterization of other composite materials with metallic matrix and granular particles;
2. A wider field of use of granular particles under form of nanometric granular fractions with the purpose of obtaining nanostructured composite materials;
3. Development of other obtaining technologies of composite materials with metallic matrix, such as magnetohydrodynamic method;

4. Improvement of Stir Casting Vortex installation by providing inert medium over the metallic bath by reduction of partial pressure of oxygen and implicitly reduction of the film thickness of Al_2O_3;
5. Improvement of the introduction way of granular particles by using multiple lance of injection using an inert carrier gas (argon), at Stir Cast Vortex method;
6. Study of efficiency from an economic point of view of obtaining technology of some composite materials with technological utility by mechanical mixing with vibrations, enlarging percentual ratio refractory particles/metallic matrix, in conjunction with enlarging frequency and amplitude of vibrations.

Listă de lucrări

Lucrări ISI

[1] **Vasile BASLIU,** Maria Vlad, Gelu Movileanu, *Composites with technological role by embedding granular particles of FeTi30 into a metallic matrix*. Journal of Science and Arts – acceptat la publicare în numărul 4(37) din 2016.

Lucrări ISI procedings

[1] **Vasile BASLIU,** Maria Vlad, Gelu Movileanu, *Obtaining composite materials with technological role by incorporating granular particles in metal matrix.* 7th Conference on Material Science & Engineering – UGALMAT 2016, mai 19 – 21, 2016 – acceptat la publicare.

Lucrări BDI

[1] **Vasile BASLIU,** Ionut CONSTANTIN, Gina Genoveva ISTRATE, Ionel PETREA, *Melting-casting plant using vibrating of melts in order to obtain composite with technological utility,* The Annals of "Dunarea de Jos" University of Galati, Fascicle IX Faculty of Metallurgy, Materials Science and Environment, martie 2010, nr.1, p. 68 – 74, ISSN 1453-083X

[2] **Vasile BASLIU,** Florentina POTECAȘU, *Research regarding the obtaining of some composite materials with metallic matrix from aluminium and FeTi (32% Ti) refractory particles*, The Annals of "Dunarea de Jos" University of Galati, Fascicle IX. Metallurgy and Materials Science, may 2011, nr. 1, p. 57 - 61, ISSN 1453 – 083X

[3] **Vasile BASLIU,** Gina Genoveva ISTRATE, *Design of an installation for determine surface tension with sessile drop method and its use for characterization metal matrix composite systems with hard particles fuse,* The Annals of "Dunarea de Jos" University of Galati, Fascicle IX. Metallurgy and Materials Science, Special issue, mai 2012, p. 198 – 203, ISSN 1453 – 083X

Lucrări publicate în volumele unor conferințe internaționale

[1] **Vasile BASLIU,** *Researches regarding the obtaining of some composite materials with metallic matrix obtained through casting,* International Conference ARTCAST 2010, the 5th edition 14 – 15 mai, Galați, România, p. 280 - 283

[2] **Vasile BASLIU,** *Development of a new installation of elaboration-casting in order to obtain composite materials with metallic matrix*, International Conference ARTCAST 2010, the 5th edition, 14 – 15 mai, Galați, România, p. 375 – 379

Lucrări comunicate (prezentare orală la Conferințe naționale)

[1] **Vasile Bașliu**, Maria Vlad, Tamara Radu, *Metode alternative și utilizarea lor la recuperarea/reutilizarea deșeurilor în stare granulară de FeSi cu 45%Si prin înglobarea lor într-un material compozit de utilitate tehnologică în sistemul A6061/FeSi45,* Workshop organizat Centrul de Cercetare Calitatea Materialelor și a Mediului de la Universitatea Dunărea de Jos din Galați, "Managementul deseurilor si al poluantilor industriali", 22.06.2016

[2] **Vasile Bașliu**, Maria Vlad, Florentina Potecașu, *Obținerea materialelor compozite de utilitate tehnologică A6061/FeTi30 prin reciclarea deșeurilor în stare granulară de feroaliaje FeTi cu 30%Ti,* Workshop organizat Centrul de Cercetare Calitatea Materialelor și a Mediului de la Universitatea Dunărea de Jos din Galați, "Managementul deșeurilor și al poluanților industriali", 22.06.2016

[3] **Vasile Bașliu**, Maria Vlad, Lucica Balint, *Dezvoltatea unor soluții tehnologice alternative de înglobarea a materialelor în stare granulară de SiC (deșeu tehnologic) și utilizarea lor pentru obținerea materialelor compozite cu rol tehnologic din sistemul A6061/SiC,* Workshop organizat Centrul de Cercetare Calitatea Materialelor și a Mediului de la Universitatea Dunărea de Jos din Galați, "Managementul deseurilor și al poluanților industriali", 22.06.2016

Lucrări comunicate (poster la Conferințe internaționale)

[1] **Vasile BASLIU,** *Research on obtaining aluminum metal matrix composites reinforced with hard particles fuse*, "Dunărea de Jos" University of Galaţi POSDRU/107/1.5/S/76822, The Second PhD Student Symposium, 13th - 14th December 2012

[2] **Vasile BAŞLIU,** Viorica MUŞAT, Simona POP, Constantin GHEORGHIEȘ, *Thermal analysis of some materials used for preparation of aluminium based composite with hardly fusible dispersed phases*, UGALNano3, Scientific Conference of Doctoral Schools from "*Dunărea de Jos*" University of Galati, 16 - 17 May 2013

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Level in national or international classification	

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Other language(s)	

Self-assessment	Understanding		Speaking		Writing
European level (*)	Listening	Reading	Spoken interaction	Spoken production	

Language english	B2	independent user	B2	independent user	B2	independent user	B2	independent user	B2	independent user
Language french	B1	independent user	B1	independent user	B1	independent user	B1	independent user	B1	independent user

(*) Common European Framework of Reference for Languages

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