ORIGINAL PAPER

COMPOSITES WITH TECHNOLOGICAL ROLE BY EMBEDDING GRANULAR PARTICLES OF FeTi30 INTO A METALLIC MATRIX

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Abstract. *Pollution of the production spaces within steel mills with particles in granular state may represent an environmental problem unless appropriate measures are taken. Thus, a viable solution may be the making of a composite material with a technological role, thanks to the distributed particles in granular state into metallic matrix.*

The research in the present paper aims at finding some obtaining technology for composite materials with metallic matrix (aluminium alloy A6061) and FeTi30 granular particles dispersed within metallic matrix, with complex deoxidant role in steel processing. Also, composite materials symbolised A6061/FeTi30 have been characterised from the chemical, structural and morphological points of view. From among the methods of obtaining composite materials, the application of vibration method has been taken into consideration, with the purpose of ensuring a high yield of embedding particles in granular state. In the process of steel making a rigorous control of technological parameters (amplitude and frequency of vibrations, temperature, time) was carried out.

Keywords: *composite, matrix, particles in granular state, vibrations, amplitude.*

1. INTRODUCTION

The steel making processes uses important quantities of auxiliary materials among which are ferroalloys. These materials are easily brittle and from this point of view are created the best conditions of productions spaces for a less local pollution. This problem can be solved by obtaining a composite material with a technological role by embedding particles into a metallic matrix of aluminium alloy A6061.

Composite materials are composed of an aluminium alloy with dispersed phases represented by heavy fusible particles of FeTi30 and they have been designed also for technological purpose of deoxidation by precipitation, combining synergist positive effects of two elements A6061 aluminium alloy and FeTi30 and using them by introducing them into the steel.

Various researches are conducted worldwide [1-7] for obtaining composite materials by using different methods. In the process of mechanical mixing by vibrations for achieving of high yield embedding, a series of technological and physico-chemical factors have an influence on the properties of the composite material.

Technological factors represented by amplitude of vibration by the vertical displacement process at a medium frequency (30 – 65 Hz) allow the particle layer to be dispersed into the metallic matrix in liquid state. Also, based on the observation of a similarity between the height of oscillation (i.e. amplitude) and the diameter of particles, action may be

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taken in order to ensure the appropriate conditions for the obtaining process of composite material.

The physical-chemical factors pertaining to the diameter, form, edge of particle, as well as their oxidation degree, also contributes to achieving a good yield embedding. An important influence on obtaining materials composites is the thickness of film of aluminium oxide (Al_2O_3), thus this is preferred to be as thin as possible for the particles to traverse and penetrate into the melt volume.

2. MATERIALS AND METHODS

2.1. MATERIALS

The present experiments used aluminium alloy A6061 and granular particles FeTi30 with granulometric fractions d_{mp}= 0.8 mm; 0.4 mm and 0.04 mm. The chemical composition of the materials used in the obtaining process, by mechanical vibrations is provided in table 1.

Table 1 Chemical composition of materials

No.	Materials	Chemical composition,[%]									Obs.
		Si	Mg	Cr	Cu	Fe	Mn	Ti	Zn	Al	
1	A6061	0.8	1.2	0.35	0.4	0.7	0.15	0.15	0.25	rest	EDX method
2	FeTi30	6.24	-	-	-	53.67	0.67	34.87	-	4.53	XRF method

2.2 .INVESTIGATIONS METHODS

For the study of surfaces of composite material, macro and microstructural analysis is used. This method allows highlighting the detailed morphological aspects of composite material such as: distribution, form, dimensions of particles embedded into metallic matrix.

In this respect, samples of composite material A6061/FeTi30 were prepared by grinding and polishing. For the metallic gloss obtained in this way, the prepared surface for emphasizing dispersion, forms and sizes of particles were studied. The samples were analysed after metallographic attack with reagent (10%HF).

Macro analysis was achieved by photography using NIKON COOLPIX L100 device. The morphological structure and chemical composition of samples were performed by Scanning Electron Microscopy - Energy Dispersive X-ray Spectroscopy (SEM-EDS) using FEI Quanta 200 equipment. X-ray spectra have been recorded at room temperature using DRON 3M diffractometer, equipped with an X-ray tube with Co anode ($\lambda_{K\alpha}$ = 1.7903), as well. The measurement range was $2\theta = 15 – 80^o$, the step and measuring time were 0.02 and respectively, 3s.

Phase identification was carried out on diffraction peaks corresponding to the crystallographic planes specific to each chemical element, oxide, chemical compound resulted from chemical reaction between constitutive phases, and formation of eutectics [9].

Using a software – demonstrative version for phases analysis MATCH3 [9] and free databases Crystallography Open Database, were able to investigate the diffraction spectra for the composite material A6061/FeTi30.

3. COMPOSITE MATERIAL A6061/FeTi30 PREPARATION

Composite materials were obtained by mechanical mixing using an installation designed by the authors [8]. Mechanical vibrations induce, in the system composed by aluminium alloy A6061 in liquid state and particles of FeTi30, 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 (Fig. 1). The top layer and bottom are of aluminium alloy and the third from the middle consists in granular particles of FeTi30.

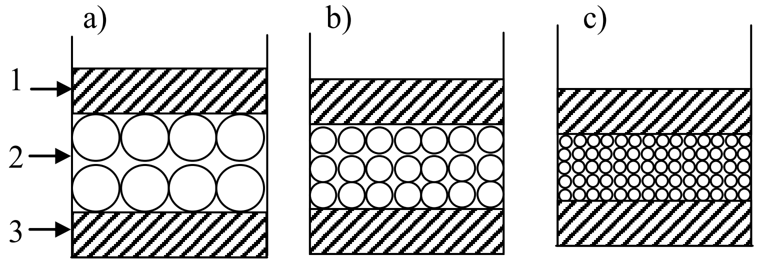


Figure 1. Successively arranged layers (sandwich): 1 - compact layer of A6061; 2 - layer of heavy fusible particles FeTi30; 3 - compact layer of A6061.

Metallic melt was superheated up to 730 ^{o}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 voids occur – interstitial spaces. The diameters of interstitial spaces can have the minimum dimension equal to the amplitude value, or higher, equivalent to the amplitude resonance given by summing table amplitude and amplitude due to inelastic collision between particles. In these voids (interstitial) liquid aluminium alloy can be infiltrated and lead to homogenization of the mixture. Some differences regarding embedding granular particles into metallic matrix by vibration are obviously caused by the frequency of vibration and by granulometric size.

These results depend on the static load of the system, respectively on the system aluminium alloy – particles submitted to vibration.

The methodology respect the following stages: material arrangement in crucible, namely the calculated quantity of aluminium alloy, has been placed at the lower and upper part of the granular particle layer of FeTi30, followed by melting up to fixated temperature in the preliminary tests and vibrations of melt.

It was approximated that a comparable quantity of slag was produced on all samples because they have been obtained in similar condition of time, temperature, equipment and quantity of aluminium alloy.

For the determination of yield embedding (η) the amount of non-embedded particles was weighed with the following formula:

$$\eta = \frac{m_{imp} - m_{nmp}}{m_{imp}} \cdot 100 \ (\%) \qquad (1)$$

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

4. RESULTS AND DISCUSSION

This study investigates the possibility of embedding a high percentage of particles from a range of 0.04 – 1.0 mm. In order to underline the particular character of each system metallic matrix/particles in granular state, three granulometric sizes (0.8, 0.4, 0.04 mm) were chosen. Regarding the frequency and amplitude of vibration, similar conditions have been created for each system. In the experimental program of obtained composite material (i.e. A6061/FeTi30) for a high yield of embedding, unlike what is known in the literature, percentage ratio metallic matrix/particles in granular state of 23% was imposed; this corresponds to 25 g aluminium alloy and 7.5 g particles.

In Fig. 2, yields of embedding obtained for composite material A6061/FeTi30 with d_{mp} = 0.8 mm are presented.

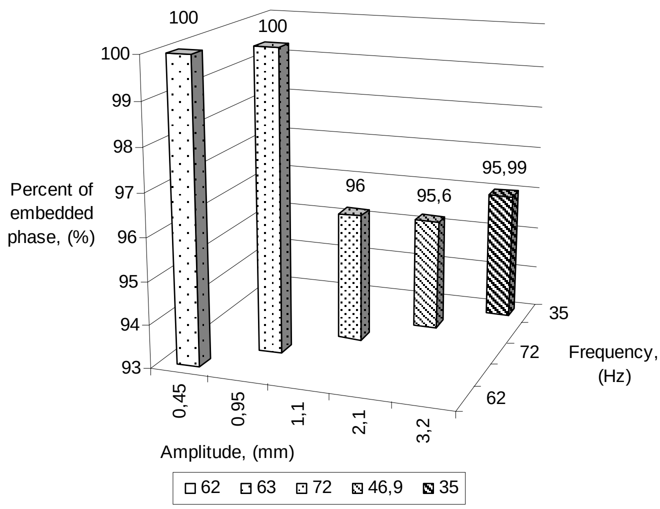


Figure 2. Particle quantity of FeTi30 with d_{mp} = 0.8 mm embedded for 23% dispersed phase from elaboration batch.

Maximum embedding starts from low amplitude 0.45 mm and frequency 62 Hz and a ratio amplitude/diameter of particle was 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, a high embedding yield was observed.

In Fig. 3 are presented the embedding yields obtained for composite material A6061/FeTi30 with grain size 0.4 mm.

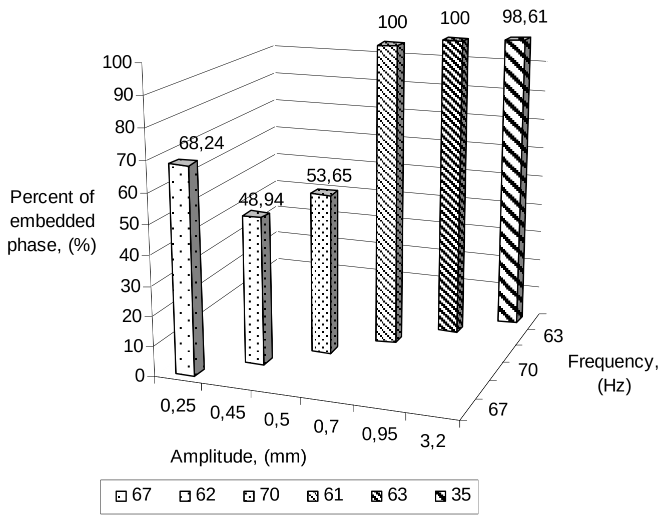


Figure 3. Particle quantity of FeTi30 with d_{mp} = 0.4 mm embedded for 23% dispersed phase from elaboration batch.

By analysing the graph from Fig. 3, can be observed a wide range of amplitudes and frequencies at which embedding was produced. From performed experiments on the range with high yield embedding begin from amplitude (0.7 – 3.2 mm) and frequency (63 – 70 Hz). The embedding process of particle FeTi30 starts from a lower amplitude of 0.25 mm and frequency of 67 Hz, resulting that the influence of lower diameter of intergranular void was significant for a relatively satisfactory yield (68.24%).

In Fig. 4 are presented the embedding yields values, obtained for composite material A6061/FeTi30 with grain size 0.04 mm.

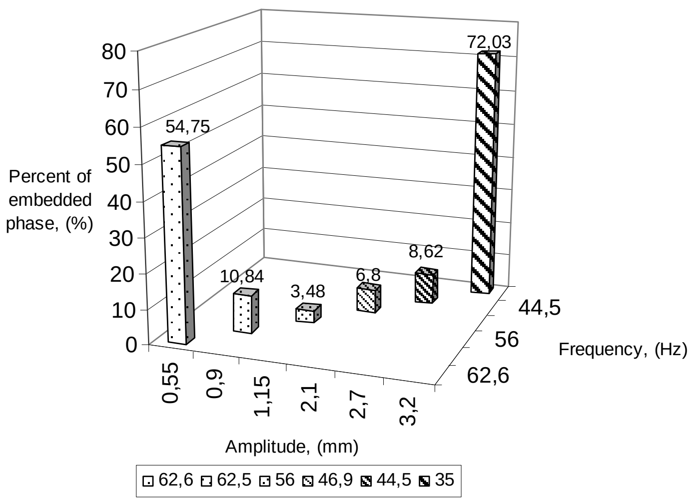


Figure 4. Particle quantity of FeTi30 with d_{mp} = 0.04 mm embedded for 23% dispersed phase from elaboration batch

By analysing the graph shown in Fig. 4, it can be observed that were obtained low embedding rates in the 0.9 – 2.7 mm amplitude range. This may be due to the following factors: layer thickness, high number and form diversity of particles, high wetting angle, and oxide layer formed at the liquid surface. Because the average diameter of particles is relatively reduced (e.g. 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 higher than the analysed cases). Above 3.2 mm it can be observed the growth embedding as a result of high amplitude.

4.1. 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 and the formation at the interface of a passing area.

The data concerning chemical and structural investigation are represented in Figs. 5-7.

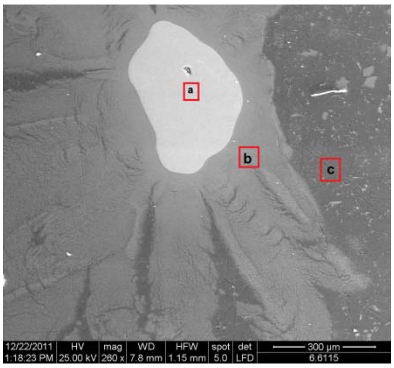
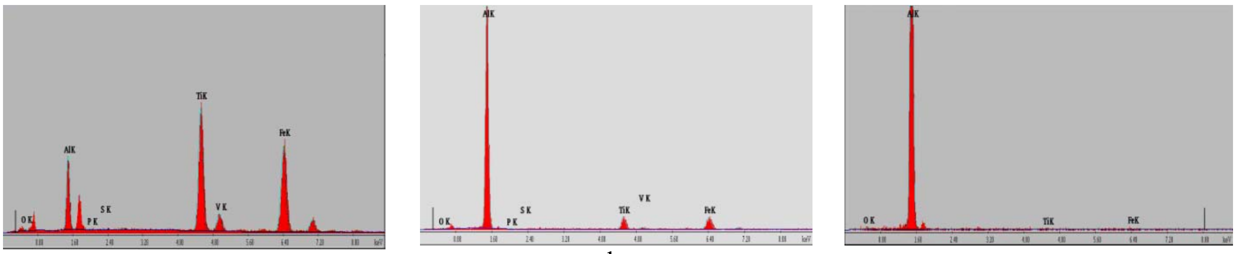
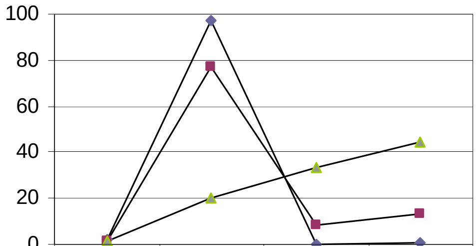


Figure 5. SEM-EDS image for composite material A6061/FeTi30



a b c

Figure 6. Spectra a, b, c renders chemical composition in 3 points on the surface sample 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 7. EDX chemical analysis of composite material A6061/FeTi30

The data presented in Figs. 5-7 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 was 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 areas are due to mass transfer of aluminium from matrix towards the other two areas and because of oxides washed and partial wetting of particle by liquid alloy which also determine a superficial dissolution.

4.2. MACROSTRUCTURAL ANALYSIS OF SAMPLE OF COMPOSITE MATERIAL A6061/FeTi30

Macrostructural analysis is a common method for the study of composite materials surfaces obtained by casting, with particles of micrometric and submicrometric dimensions. This analysis is compulsory as it can highlight relevant aspects, such as the uniform distribution and casting defects – porosity.

Figure 8. Macrostructure of composite material A6061/FeTi30 obtained by vibrations method, time = 10 min and d_{mp} = 0.8 mm.

In Fig. 8, in right side 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.

4.3. MICROSTRUCTURAL ANALYSIS OF SURFACE SAMPLE FOR COMPOSITE MATERIAL A6061/FeTi30 BY SCANING ELECTRON MICROSCOPY (SEM)

Microstructural analysis was carried out on sample of composite material by vibration method. In Figs. 9 – 11 it was presented SEM images of composite material with granular particles with d_{mp} = 0.8 mm, 0.4 mm and 0.04 mm.

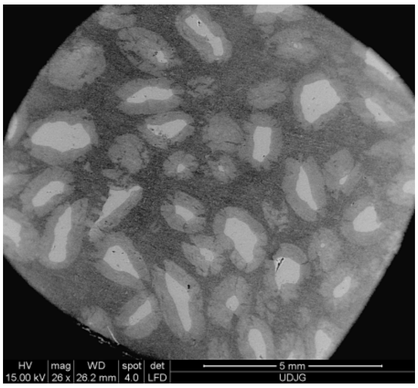


Figure 9. Composite material A6061/FeTi30 with d_{mp} = 0.8 mm

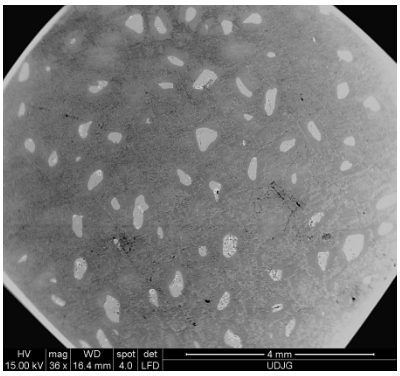


Figure 10. Composite material A6061/FeTi30 with d_{mp} = 0.4 mm

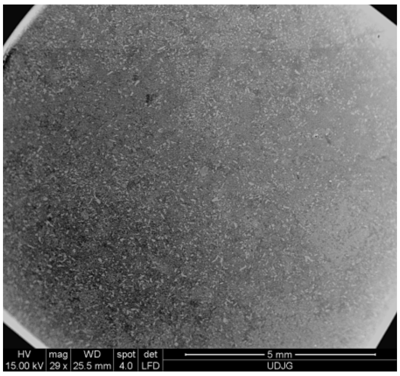


Figure 11 Composite material A6061/FeTi30 with d_{mp} = 0.04 mm.

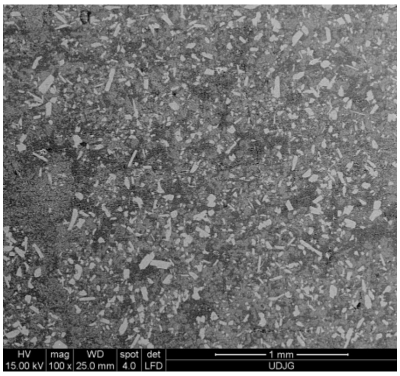


Figure 12 Composite material A6061/FeTi30 with d_{mp} = 0.04 mm.

Analysing the morphology of sample (Figs. 9 – 12) it can be seen that the particle distribution in the metallic matrix was more uniform than 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 was created for many of the particles to move vertically in the metal matrix. In this respect, an important role is the time of vibration and, also, the choosing the right moment of cooling the melt.

Particulate forms are various, predominant those in polygonal form with round edges and the spherical ones. Also, the homogeneity of particulate dimensions can be observed, which was d_{mp} 0.8 mm, (Fig. 9), 0.4 mm, (Fig. 10), and 0.04 mm (Figs. 11 and 12). In the analysed samples a proportion of particles from other granulometric size, with lower medium diameter, have also been identified, due to insufficient granulometric ranking or the phenomenon of crumbling of particles in the obtaining process time. It can be observed that composite material was homogenous and did not show defects such as pores that may occur at an improper amplitude and frequency. Being a composite material with a technological role, eventual transition zones are not desirable because they may contain intermetallic chemical compounds with superior melting temperature of steel making.

4.4. STRUCTURAL ANALYSIS BY X-RAY DIFFRACTOMETRY FOR COMPOSITE MATERIAL A6061/FeTi30

The result of obtaining process of composite materials is traced with the help of structural analysis by x-ray diffractometry. Thus, are highlighted the chemical elements present in composite, phase distribution because of mechanical mixing process by vibration.

Characteristic peaks for composite material A6061/FeTi30 were identified, in which we highlighted component phase's αAl, αFe, αTi, as well as chemical combination Fe_2Ti, $FeAl_3$, $TiAl_3$ and oxides Al_2O_3, FeO.

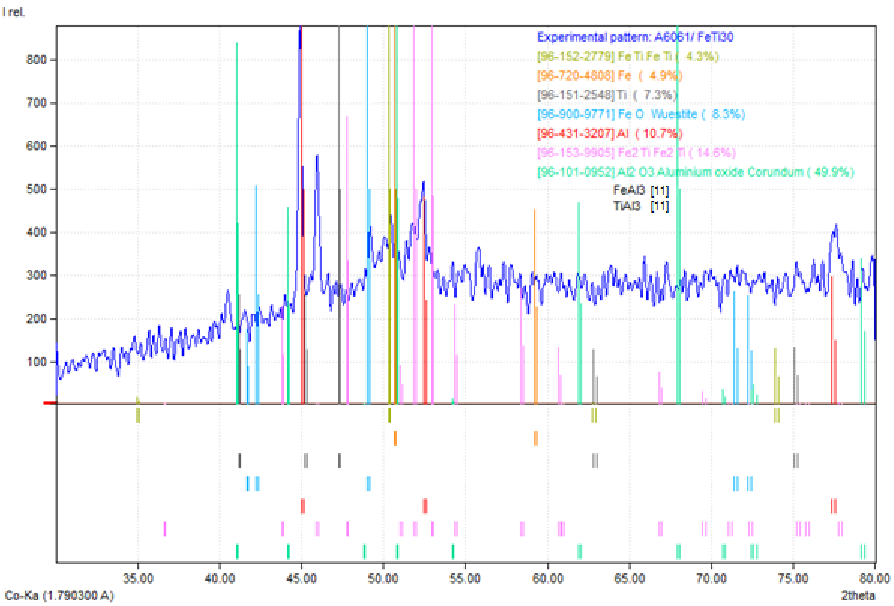


Figure 13 Diffractogram and diffraction data from sample A6061/FeTi30

Characteristic peaks of phases are shown in Fig. 13. From diffractogram analysis, no other phases are observed, may be due to possible chemical reactions between elements. Thermodynamic conditions (i.e. temperature and pressure) of obtained composite material, as well as the reduced time of process, have reduced the interfacial chemical reactions between the chemical elements and their oxides, although existing conditions for aluminium diffusion from melt at interface area and iron and titanium in alloy melt, may have as result a intermetallic compound $FeAl_3$, $TiAl_3$ [11].

By the identification of minimum three characteristics peaks, were highlighted the components of composite material: for Fe $2\theta = 50.64$, $2\theta = 50.76$, $2\theta = 59.19$; for αTi $2\theta = 47.26$, $2\theta = 41.19$, $2\theta = 45.23$; for Al_2O_3 $2\theta = 67.90$, $2\theta = 50.80$, $2\theta = 41.04$; for FeTi $2\theta = 50.31$, $2\theta = 50.43$, $2\theta = 73.90$; for FeO $2\theta = 42.24$, $2\theta = 49.00$, $2\theta = 72.23$; for Fe_2Ti $2\theta = 51.87$, $2\theta = 52.92$, $2\theta = 58.36$; for Al $2\theta = 45.01$, $2\theta = 52.47$, $2\theta = 77.57$, for $FeAl_3 2\theta = 50.68$, $2\theta = 51.94$, $2\theta = 52.46$, for $TiAl_3 2\theta = 45.74$, $2\theta = 55.23$, $2\theta = 76.97$.

5. CONCLUSIONS

The solution for embedding granular particles into a metallic matrix of aluminium alloy and reinclusion locally into technological flux of steels elaboration can represent a viable alternative to maintain clean the industrial production spaces.

Obtaining method by vibrations is an alternative method of obtaining composite materials in liquid state with potential by embedding of an important percentage of particles of FeTi30, respectively 23%. We carried out research for three classes of particle size 0.8 mm, 0.4 mm and 0.04 mm.

The embedding process of FeTi30 particles starts from a reduced amplitude, respectively 0.25 mm and a 67 Hz frequency, resulting that the influence of lower diameter of intergranular void is significant for a relatively satisfactory yield (68.24%).

Macro and microscopic morphological analysis has shown a homogeneous composite material with a good distribution of particles, their forms being predominantly polygonal or spherical, with round edges and a lower percentage of defects contained in the metallic matrix.

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