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# SYNTHESIS OF IN SITU Al-Si MATRIX COMPOSITES BY STIR CASTING TECHNIQUE

ABSTRACT

Fabrication technique by stir casting of Al-Si alloy with Cr and Fe powders as well as chromium steel substrate has been described. Morphology of the composites has been analysed using SEM images and EDX chemical composition analysis. Improved microhardness of the *in situ* formed Al, Si, Fe, Cr phases indicates that the composites may be the candidate materials for Al-Si alloy moulded elements of improved abrasion resistance.

## 1. INTRODUCTION

Aluminum alloys discontinuously reinforced with ceramic particles are currently being developed for various high performance applications. SiC, TiC, $B_4C$, $TiB_2$, $Si_3N_4$ and $Al_2O_3$ particles have been examined for their potential reinforcement application in aluminum matrix composites [1]. However, there are many problems to overcome before such composite materials find wider applications. Meanwhile, other solutions of cost-effective reinforcement of metals are sought with reduced particles size as well as a strong mechanical bond at the interface without the presence of a chemical reaction products which lead to the improvement in strength of the composite. The new processing techniques based on the concept of precipitation *in situ* of reinforcement in metal matrix are now being employed. Reinforcement phases in these processes are generated *in situ* by an exothermic, chemical reaction between selected elements [2]. Particles resulting from this reaction are of sub-micron size and have very clean surfaces enabling better wetting and bonding between them and the matrix. Thus, they do not embrittle the material and may constitute the effective reinforcement once their properties (e.g. stiffness) and the morphology (e.g. aspect ratio) are well selected. The $TiB_2$ *in situ* Al composites have been fabricated by stir casting technique [3, 4]. TiB has been used as an effective *in situ* reinforcement in titanium [5, 6]. In [1] the TiB has been obtained as *in situ* reinforcement of Al-Cu (4,5%Cu). Without subjecting the composite to secondary processing such as extrusion, *in situ* TiB reinforcement (10 vol %) resulted in tensile strength increase of 21% whereas the *in situ* $TiB_2$ reinforcement (3,3 vol %) resulted in a tensile increase of 5,6%.

A process called XD [7] has been developed to fabricate *in situ* ultrafine ceramic particle--reinforced metal matrix composites (MMCs). The basic principle of this technique is that the mixture of Al powders and ultrafine metal and ceramic particles is milled, cold compacted and heated to above 800ºC in vacuum, then cooled down to 600ºC and hot pressed. The pressed billets are extruded at 420ºC. The $TiB_2$ (the size of 0,1 μm), $Al_2O_3$ or $Al_3Ti$ particulates are formed in this way by *in situ* reaction. Depending on the composition of powders the variation

in the structure and properties of composites can be obtained with as high UTS as 446 MPa for 15 vol % TiC [7] and 334 MPa for 20 vol % $TiB_2$, whereas the highest reinforcing effect obtained for SiC whiskers (20 vol % SiC) was 278 MPa as compared with 103 MPa for pure Al.
Other processes of *in situ* fabrication of composites include Mixalloy [8], Dimox [9] and RGI [10]. Mixalloy, elaborated at MIT uses turbulent eddies and jet impingement to mix two or more liquid metal streams of turbulent velocities in a chamber having an intricate geometry. The resulting mixture then solidifies fast enough to preserve the required microstructure. Continuous research and development of process has resulted in commercial manufacture of boride dispersion strengthened copper alloys. Numerous other materials have been evaluated. Reactive gas injection (RGI) uses reactive carbon or nitrogen containing gas to blow through the molten metal. As a result of dissociation the elements capable of reacting with the components of the liquid alloy are released. The products of the reaction remain in the liquid solution thus forming the composite after solidification. This technique can be used to obtain the composites based on Al, Ti, Cu, NiAl, NiTi, TiAl reinforced with particles of carbides eg. TiC, TaC, $B_4C$, SiC or nitrides e.g. $Si_3N_4$, AlN, BN. In [11] the synthesis of TiC carbides in Al and Cu matrices using methan as reactive gas was described. The TiC particles of the mean diameter 1-4 μm and volume fraction up to 12% were formed.

The present study, is a continuation of the previous work [12] on cost effective fabrication of MMCs by means of *in situ* formation of Al, Si, Fe and Cr reinforcement phases in Al-Si matrix. The use of tool steel as a source of Fe and Cr for *in situ* reaction leading to the development of hard reinforcement has been studied. The new composites were fabricated by stir casting technique.

## 2. EXPERIMENTAL PROCEDURE

### 2.1. Composites fabrication

The eutectic and hypoeutectic Al-Si alloys (11% and 26% Si) were used as matrix materials. The following substrate materials as a source of Fe and Cr have been used alternatively: 1. 5 wt % Fe powder (50 μm in diameter) and 5 wt % Cr powders (200 μm in diameter) 2. 10 wt. % milled tool steel scraps (1,65% C 10% Cr) 3. chromium steel stirrer (1,65% C, 10% Cr) being dissolved in liquid aluminum.
The *in situ* composites were made by stir casting using the following procedures:

(1) Cr powder was gradually added to the liquid aluminum alloy while stirring the mixture continuously. Next Fe powder was added and the mixture was stirred together for 60 min. at 700ºC. Test samples were moulded after each 30 minutes of stirring.

(2) Steel scraps were milled and sieved. The maximum size of the particles was 500 μm. The particles were gradually added to the liquid alloy as in the case of the Fe, Cr powders and stirred for 60 minutes. The slurry was poured into the small, metallic cylindrical mould (30 mm in height) and solidified at ambient temperature.

(3) In the next group of materials no powder was added to the Al-Si liquid alloy. The substrate for chemical reaction in the Al-Si alloy was the stirrer made of the same tool steel as the scraps. While turning at 1500 revolutions per minute at 700ºC the stirrer gradually "dissolved ", which finally resulted in the formation of new phases in the Al alloy after solidification. Every 15 minutes the samples for metallographic examinations have been moulded. Finally, the cylindrical samples of some of the materials have been cast into the cylindrical metallic moulds (20 mm in diameter, 150 mm high) for tensile testing.

### 2.2. Composites characterization

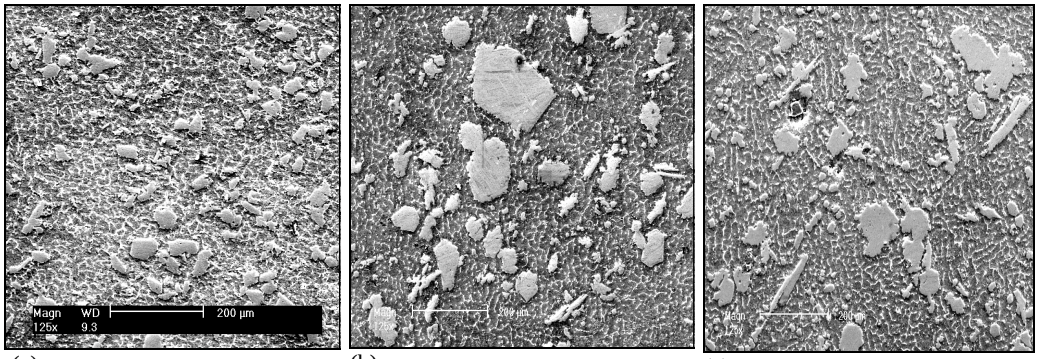
The morphology of the materials obtained by stir casting has been examined using scanning electron microscope coupled with the roentgen spectroscope EDX. The maps of distribution of the elements have been made followed by point chemical analysis of the precipitates observed in the structure. The approximate composition of phases has been identified. The volume fractions of individual precipitates have been determined by quantitative image analysis.

The Brinell hardness was tested. Next the microhardness tests have been performed using microhardness tester, the PMT-3. The tensile tests have been attempted, however due to some porosity of the samples they were abandoned.

## 3. RESULTS AND DISCUSSION

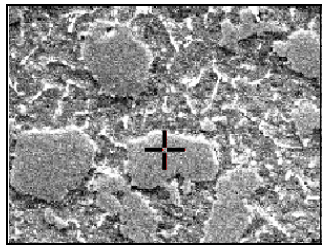
### 3.1. Eutectic Al-Si alloy – Fe, Cr powders system

Fig. 1 shows the structures of the materials obtained at different stages of the fabrication of the composite. The first sample (Fig. 1a) has been moulded after all Cr powder has been added to the liquid metal (15 minutes from the beginning of the process). The structure consists of Al-Si matrix with grey particles – the product of the reaction of Cr with Al. From point chemical analysis of the particle it can be seen (Fig. 2) that the grey particles are Al- rich non-equilibrium Al-Cr phase. The Cr contents is slightly varied for different precipitates depending on the size of the particle.




(a) (b) (c)

**Fig. 1.** SEM micrographs of eutectic Al.-Si matrix composites after incorporation of: a) Cr powder, b) Cr+Fe powders and stirring 30 minutes, c) 120 minutes. Magnification 125x



| | Wt % | At % |
|---|---|---|
| Al | 76,4 | 83,9 |
| Si | 1,2 | 1,3 |
| Cr | 19,5 | 11,1 |
| Fe | 0,8 | 0,4 |

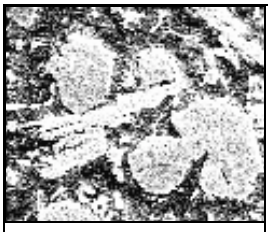
**Fig. 2.** SEM micrograph and the approximate chemical composition of the particle (marked +) of the same sample as in Fig. 1a. Magnification 500x

The SEM image shown in Fig. 1b illustrates the structure of the composite obtained after all Fe powder has been incorporated into the liquid metal and stirred for 30 minutes. Larger, round and smaller, elongated precipitates are observed. The map of distribution of the elements (Table 1b) clearly shows that the round particles are Al-Cr rich phase which is in reaction with Fe (brighter areas on round Al-Cr flakes in Cr distribution map). Elongated precipitates are Al-Fe rich phases. 2h stirring of the mixture leaded to structure showed in Fig. 1c.

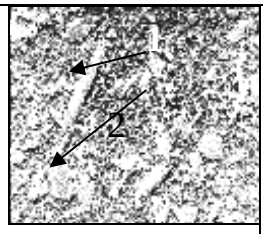
The variation of Fe, Cr and Si contents in the precipitates during the stirring period has been given in Table 1a,b. It can be seen that Fe has its greatest contents in elongated phase. However, for longer elaboration time the distribution of Fe becomes more equitable. This is also seen in the maps b, c, d, where Fe is observed in all the phases, but differences in the intensity of colour between the elongated and other phases is decreasing with increased stirring time. Chromium, initially present mainly in the round precipitates, due to diffusion can be observed in the increased amount (ca. 8% in the needles). In the final structure obtained in this study there is still some difference in the composition between the phases, i.e. there exists a Cr –rich non-equilibrium phase of the type $Al_{18}Cr_2Fe$ and a Fe rich phase $Al_{19}Fe_2Si_2Cr$.

Microhardness of the particles in Fig. 2 was 380 HV 0.05. Microhardness of the intermetallic phases shown in Table 1a (c) was (450-530) HV 0.05.

***Table 1a.*** *Chemical composition of the phases (EDX analysis) of eutectic Al-Si matrix composites after incorporation of Cr+Fe powders followed by stirring for: a) 30, b) 60, c) 120 minutes*

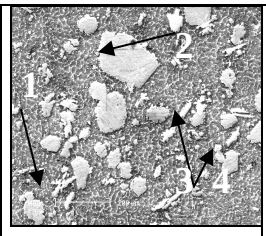


(a)



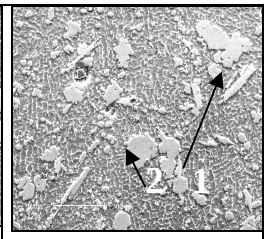

(b)

| | Wt % | At % |
|---|---|---|
| **1** | | |
| Al | 63,5 | 75 |
| Si | 1,9 | 2,2 |
| Cr | 4,5 | 2,8 |
| Fe | 29,2 | 16,8 |
| **2** | | |
| Al | 69 | 78,7 |
| Si | 2,3 | 2,5 |
| Cr | 19,3 | 11,3 |
| Fe | 7 | 3,8 |




(c)

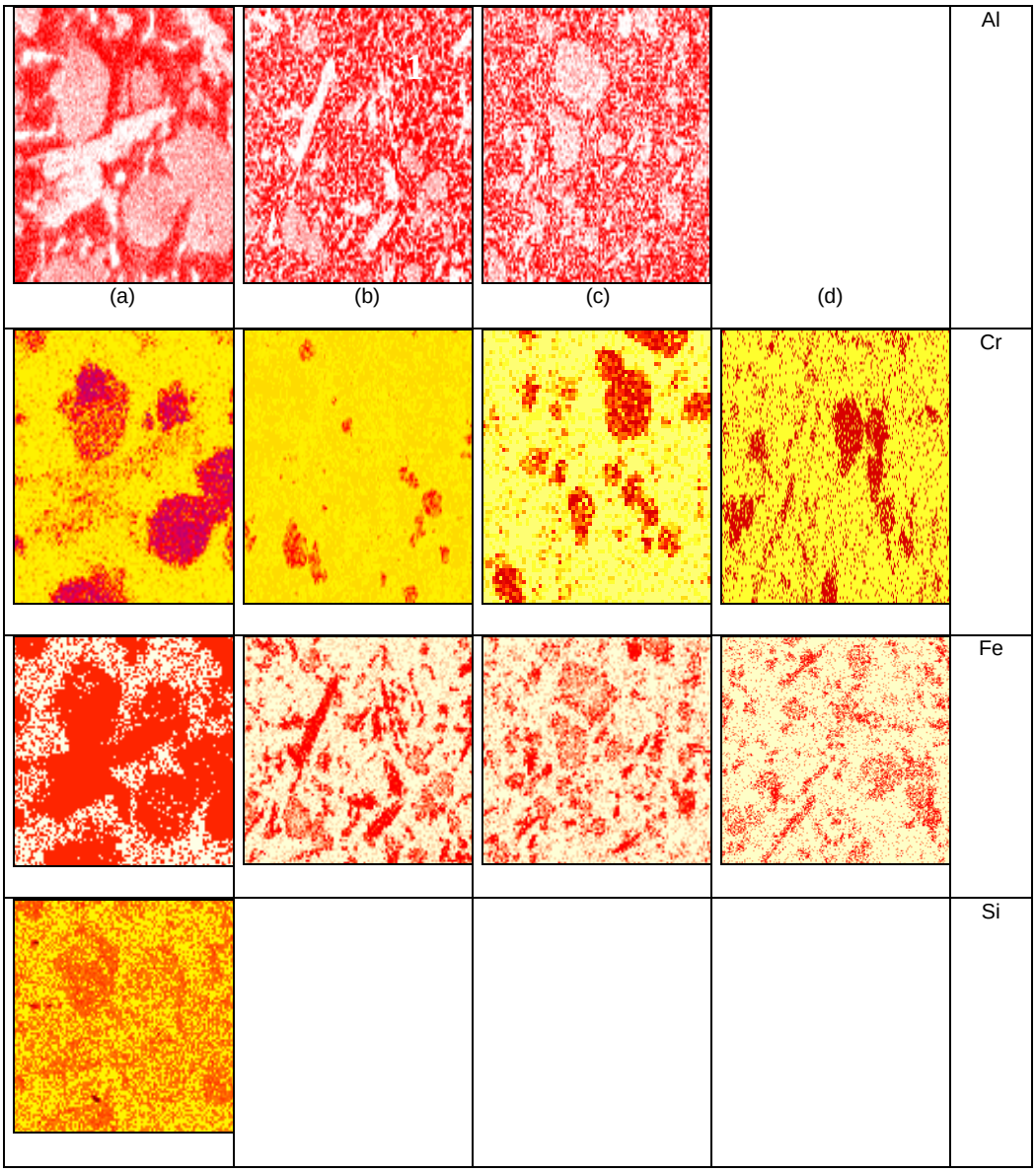
| | Wt % | At % |
|---|---|---|
| **1** | | |
| Al | 64,8 | 77,3 |
| Si | 1,7 | 2 |
| Cr | 3,0 | 1,8 |
| Fe | 29,8 | 17 |
| **2** | | |
| Al | 70,5 | 79,3 |
| Si | 2,5 | 2,7 |
| Cr | 22,8 | 13,3 |
| Fe | 3 | 1,6 |
| **3** | | |
| Al | 69,5 | 79,9 |
| Si | 1,4 | 1,5 |
| Cr | 17,4 | 10,4 |
| Fe | 10,7 | 5,9 |
| **4** | | |
| Al | 65,9 | 75,2 |
| Si | 6,2 | 6,7 |
| Cr | 8,3 | 4,9 |
| Fe | 18,3 | 10,1 |




(d)

| | Wt % | At % |
|---|---|---|
| **1** | | |
| Al | 67,3 | 78,5 |
| Si | 6,9 | 7,6 |
| Cr | 7,8 | 4,7 |
| Fe | 16,9 | 8,2 |
| **2** | | |
| Al | 70,2 | 80,2 |
| Si | 1,9 | 2,2 |
| Cr | 16,9 | 9,7 |
| Fe | 10,6 | 5,9 |

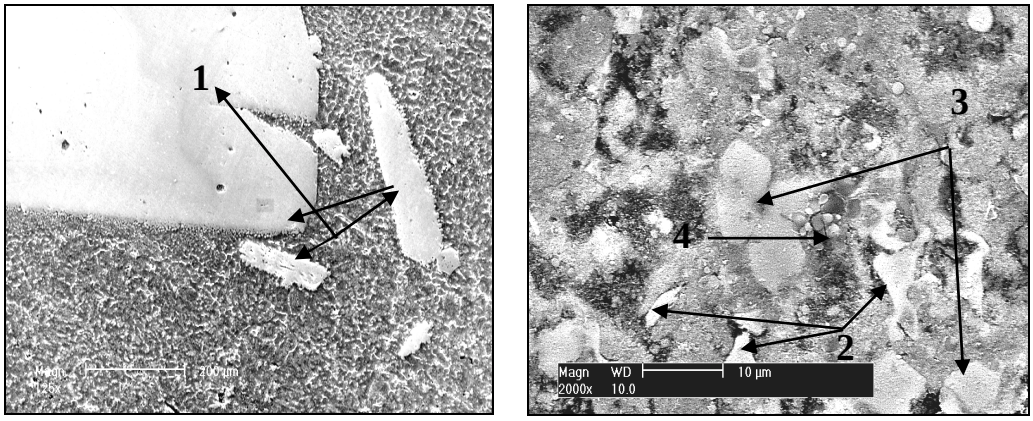
***Table 1b.*** *EDX map of distribution of the main elements in the eutectic Al-Si matrix composites after incorporation of Cr+Fe powders followed by stirring for: a) 30, b) 60, c) 120 minutes*




## 3.2. Eutectic Al-Si alloy – chromium steel scraps system

Since the powders of pure metals are quite expensive, the replacement of Fe and Cr powders by the scraps of chromium steel (10% Cr) was attempted. Fig. 3 shows the structure obtained by stirring Al-Si fused alloy with Cr steel scraps for 1 hour at 700ºC. The initial structure of the steel was chromium ferrite with carbides. In the final structure large and small scraps have similar composition of approximately $Fe_3Al$. In addition the EDX maps of the composite structure

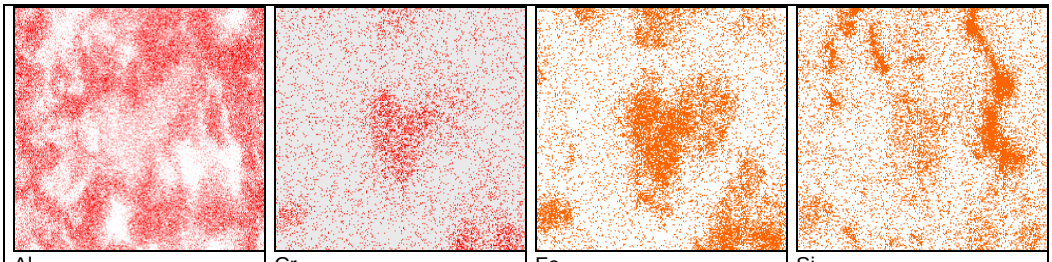
at 2000x showed the precipitates rich in Al, Fe and Si. Point chemical analysis indicates that they are the non-equilibrium phases $Al_{21}Fe_3Si_2Cr$.
Microhardness at point 1 (Fig. 3a) was 635 HV 0.05.




(a) (b)

**Fig. 3.** Macrophotograph of the structure of the alloy obtained by mixing the eutectic Al-Si alloy with chromium steel scraps and stirring for 60 minutes (a) Magnification 125x, (b) new precipitates of Al, Fe, Cr, Si at magnification 2000x

***Table 2.*** *EDX map (see Fig. 3b) and chemical composition at the points indicated in Fig. 3a,b of the composite obtained by mixing eutectic Al-Si alloy with chromium steel scraps*



| Al | | | Cr | | | Fe | | | Si | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Wt % | At % | | Wt% | At% | | Wt % | At % | | Wt % | At % |
| 1 | 62,47 | 75,7 | 1 | 2,0 | 1,2 | 1 | 33,4 | 19,5 | 1 | 1,5 | 1,8 |
| 2 | 87,3 | 87,4 | 2 | 0,8 | 0,4 | 2 | 2,2 | 1,1 | 2 | 5,5 | 5,6 |
| 3 | 67,2 | 76,2 | 3 | 5,7 | 3,3 | 3 | 18,8 | 10,3 | 3 | 6,5 | 7,1 |
| 4 | 82,7 | 87,8 | 4 | 2,5 | 1,4 | 4 | 9,7 | 5,0 | 4 | 2,7 | 2,8 |

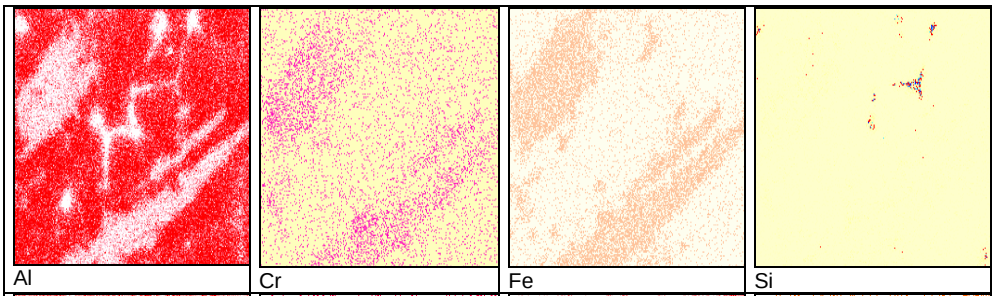
### 3.3. Eutectic Al-Si alloy – chromium steel stirrer system

Since the preparation steel scraps proved time consuming a new approach has been adopted to the fabrication of Al-Si composite with Fe and Cr. The structure of the composites obtained by dissolving the steel stirrer in liquid alloy has been showed in Fig. 4. The needles present after the stirrer has been turning for 1 hour have the shape of short fibers and seem promising as a reinforcement of Al-Si alloy. However, the prolonging of the process leaded to the enlargement

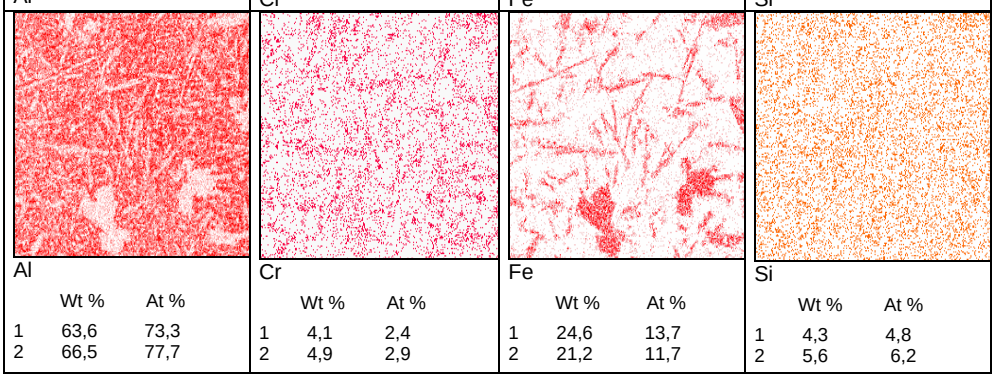
of the individual needles at the expense of their number and the precipitation of round particles. Point analysis of both the needle-like phase and particles as well as EDX maps of the distribution of elements in the composite structure showed that both phases differ only slightly in Fe, Si and Cr contents. The particles have almost the same compositions as the small precipitates in the chromium steel scraps system. The needles are richer in Fe.
Microhardness of the needles (point 1 in Fig. 4) was 535 HV 0.05.

***Table 3.*** *EDX maps of the distribution of the main elements in the eutectic Al-Si matrix composites obtained by chemical reaction of the chromium steel stirrer and liquid alloy after stirring for: a) 60 minutes, b) 90 minutes*

(a)




(b)




| | Wt % | At % |
|---|---|---|
| 1 | 63,6 | 73,3 |
| 2 | 66,5 | 77,7 |

| | Wt % | At % |
|---|---|---|
| 1 | 4,1 | 2,4 |
| 2 | 4,9 | 2,9 |

| | Wt % | At % |
|---|---|---|
| 1 | 24,6 | 13,7 |
| 2 | 21,2 | 11,7 |

| | Wt % | At % |
|---|---|---|
| 1 | 4,3 | 4,8 |
| 2 | 5,6 | 6,2 |

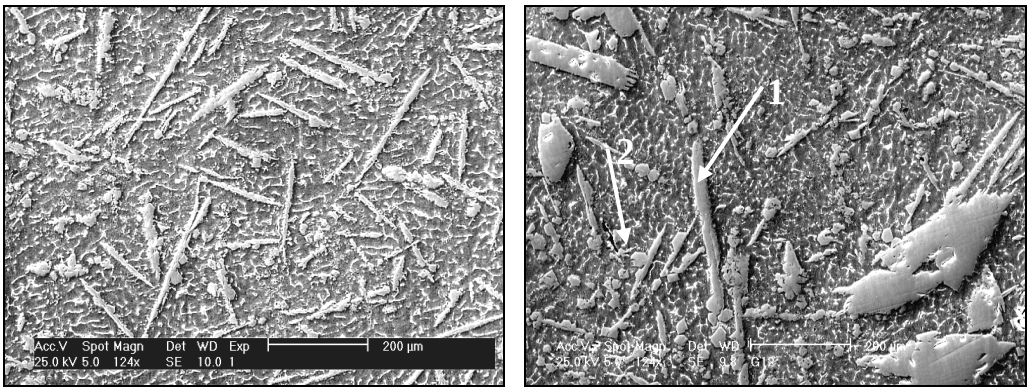
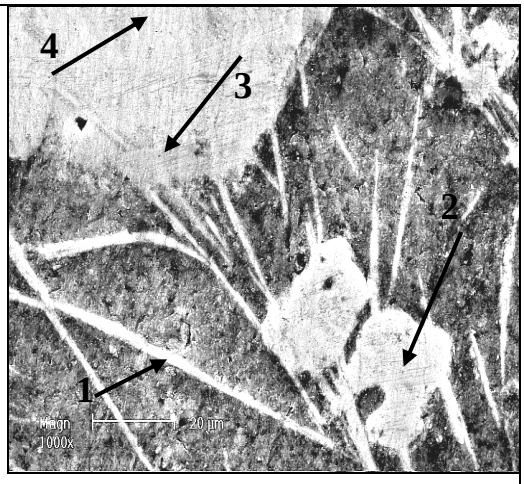



**Fig. 4.** SEM micrograph of the eutectic Al-Si matrix composites obtained by chemical reaction of the stirrer and liquid alloy after stirring for: a) 60 minutes, b) 90 minutes

### 3.4. Hypoeutectic Al-Si alloy – chromium steel stirrer system

The final synthesis of composites was performed using hypoeutectic Al-Si alloy, the same as in the former study [12]. The substrate material was in the form of a stirrer made of tool steel. Fig. 5 illustrates the morphology of the composite obtained at 700ºC after stirring the liquid alloy with the chromium steel stirrer for 1 hour at 700ºC. Two clearly distinct phases have been formed: the „short fibers" and small particles. The point analysis of the composition of both elongated and flaky phases (Fig. 5) shows that the elongated phase at this stage of elaboration can be described as $Al_8Si_2Fe$ and contains no Cr while the particles are the complex phase of all the principal elements including approximately the same amount of Cr as in the case of the composite obtained from eutectic Al-Si alloy.

Microhardness at point 2 in Fig. 5 was 677 HV 0.05. The microhardness of big precipitates (point 3, 4) in Fig. 5 was different at different points: (550-713) HV 0.05, due to differences in chemical composition.




| | 1 | | 2 | | 3 | 4 |
|---|---|---|---|---|---|---|
| | Wt % | At % | Wt % | At % | Wt % | Wt % |
| Al | 65,4 | 70,1 | 61,3 | 70,1 | 61,1 | 60,8 |
| Si | 16,5 | 17 | 9,9 | 10,8 | 9,0 | 9,7 |
| Cr | 0,3 | 0,2 | 3,4 | 2,0 | 4,0 | 5,6 |
| Fe | 15,5 | 8 | 22,9 | 12,6 | 23,9 | 20,8 |

**Fig. 5.** SEM micrograph and chemical composition of the precipitates of the Al-Si hypoeutectic alloy obtained by chemical reaction of chromium steel stirrer with liquid alloy while stirring for 60 minutes

## 4. CONCLUSIONS

Composites were fabricated by *in situ* reaction of the materials containing Fe and Cr in Al-Si alloy matrix. The replacement of Fe and Cr powders by chromium steel scraps or „dissolution" of chromium steel stirrer resulted in very similar morphology of the composites. The precipitates ofnon-equilibrium phases of the type: $Al_{18}Cr_2Fe$ and $Al_{19}Fe_2Si_2Cr$ have been identified. The volume fraction of the precipitates has been found $V_f$ = 15-18%. The microhardness of the precipitates was

in the range *(*450-650) HV 0.05 as compared with 60 HV 0.05 for the matrix material. Hardness of the composites was (100-120) HB as compared with 70 HB for eutectic Al-Si alloy, which gives the increase of 40-70%. Although the efficiency of the reinforcement does not seem very high the results obtained could be promising in terms of abrasion resistance.
Fabrication of Al Si composites by *in situ* reaction of metallic powders with liquid alloy proved interesting from the point of view of improved hardness of the material and possibly its abrasion resistance. However, in order to obtain the composites with higher reinforcement efficiency, using the same fabrication technique, the effect of other powders should also be studied.

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