MICROSTRUCTURAL CHARACTERIZATION OF SIC REINFORCED ALUMINUM MATRIX COMPOSITES

Project No: 106M328

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1. INTRODUCTION

1.1. State of the Art

Metal matrix composites (MMCs) are a class of material with the ability to blend the properties of ceramics (high strength and high modulus) with those of metals or alloys (ductility and toughness) to produce significant improvements in the mechanical performance of the composites over those of the metals or alloys. Due to relatively inexpensive reinforcements and the processes resulting in reproducible microstructures and properties, MMCs are being increasingly sought for a wide range of applications in the electronics, automotive and aerospace industries. Particle reinforced MMCs also have an advantage over other types of composites since they show isotropic behavior, and can be formed using the traditional metal working practices such as extrusion, rolling and forging.

SiC or Al_2O_3 particle reinforced aluminum alloy matrix composites have been extensively studied. These materials have been produced by industry in relatively large quantities. Many mechanisms responsible for their mechanical characteristics are well understood, including the roles of damage development, internal stresses, reinforcement clustering, interfacial bond strength and the effects of the presence of the reinforcement on aging of the matrix. However, much work remains to be done before required property combinations can be systematically achieved via microstructural design.

The processing of MMCs has also generated much interest. Many publications have appeared over the past decades on this subject. Commonly, such studies have presented novel composite materials or processes, without advancing the underlying science concerning the transport phenomena involved or their relationship with microstructural features of the product. Particle reinforced MMCs have been synthesized using a number of different techniques that include solid phase processes, liquid phase processes and two phase (solid–liquid) processes [1]. Conventional foundry processes have long been a favored processing method as they lend themselves to the manufacture of large numbers of complex-shaped components at the high production rates and low costs required by industry. They have also been shown to be very promising for the manufacture of near-net shape composites in a simple and cost-effective manner [2]. In recent years, however, the spray atomization and co-deposition method has attracted considerable attention because of its many advantages including microstructural refinement, reduced segregation, minimal interfacial reactions and under certain conditions, the possibility of near-net shape manufacturing [3]. A novel processing method known as the disintegrated melt deposition technique is currently being investigated and developed for the synthesis of near-net shape discontinuously reinforced MMCs [4]. In the case of powder metallurgy method, powdered metal is blended with reinforcement particles, and then, densified by die pressing, canning, and extrusion or canning and hipping. This is probably the most expensive technique, but allows the greatest flexibility in choice of particle size and reinforcement concentration [5].

The rate of increase in strength with volume fraction decreases beyond approximately 30–40 vol.% SiC. However, ductility, fracture toughness, formability, and machinability tend to decrease in value with increasing levels of reinforcement [5]. Strength clearly increases with reducing the particle size of the reinforcement. It has been reported that the tensile strength of SiCp (20 vol.%) reinforced 6061-T6 matrix composite increased from 421 to 571MPa by decreasing the size of SiC particles from 100 to 1 μm. By reducing the reinforcement size, the ability to perform a secondary deformation and machinability was also enhanced. The major influence which reinforcement distribution is thought to have is on the ductility and fracture toughness of the MMC, and hence indirectly on strength. It has also been reported that, in a 7000 series of Al alloy, for a given volume fraction of reinforcement, toughness increases with a decrease in the distance between particles, as indicated by a lower Al–SiC powder size ratio [7]. Mechanical properties and stress–strain behavior for several commercial Al-matrix composites, containing up to 40 vol.% discontinuous SiC whisker, nodule or particulate reinforcement were evaluated by McDanels [8]. The billets, obtained by hot pressing of the compacted powder mixtures, were extruded and then cross-rolled into flat plates. The elastic modulus of the composites was found to be isotropic, independent of the type of reinforcement, and controlled solely by the SiC content. However, yield/tensile strengths and ductility were controlled primarily by the matrix alloy and temper condition. Type and orientation of reinforcement had some effect on the strength, but only for those in which the whisker reinforcement was highly oriented. Tsangarakis et al. [9] reported the

tensile, fatigue, and fracture toughness properties of particulate and continuous fiber SiC reinforced Al-alloy composites in plate form. They have found that tensile properties of composites are dependent on the chemistry of the matrix, however, fatigue crack growth rate may be independent of the matrix chemistry and decreased with material thickness. The particle size of SiC and Al material has an important role. If the particle size ratio is high, the particle size of them is very different and the smaller SiC particles can cluster among the bigger Al particles [10]. If the reinforcement distribution is not homogenous, then clusters develop and pores appear among them. These clusters usually function as initial place for cracks. In the composites made via powder metallurgy route the reinforcement distribution homogeneity can be affected by the particle size ratio. To improve the homogeneity reinforcements with bigger particle size are used. However, this leads to worse mechanical properties [11]. There are several quite new methods to characterize the microstructure of the particle reinforced metal matrix composites, such as pair correlation function or radial distribution function [12].

Establishing correlations between the microstructural parameters and nondestructive evaluation results could be useful for improving the process parameters and controlling the quality of the composites. Mott and Liaw [13] correlated the results of tensile and fracture toughness tests and ultrasonic measurements on the specimens fabricated by vacuum hot pressing the powder mixture of 7090Al and SiC whiskers. They reported that the inhomogeneous distribution of SiC whiskers, which significantly degrades mechanical properties, is detectable by ultrasonic measurement. Lu and Liaw [14] proposed a theoretical model establishing the relationship between ultrasonic velocity and the microstructure. They took into account the statistical particle orientation distribution in the extruded SiC-reinforced various Al-alloy matrix composites to predict ultrasonic velocities. Jeong used multiple non-destructive techniques for determining the volume fractions of reinforcement in SiCp-reinforced 7091 Al-alloy matrix composites [15]. Martin et al. [16] studied the correlation of the ultrasonic velocity and specific surface area that occur as a result of sintering for several oxide powder systems. After extensive studies on SiC particle reinforced Al-matrix composites Gür concluded that the ultrasonic velocity increases with an increase in SiC content; however, ultrasonic velocity decreases owing to microporosity caused by the segregation of SiC particles [17].

1.2. Problem Statement

Systematic investigations on particle reinforced metal matrix composites (MMCs) are required of the fundamental links between microstructure and properties. There are a few studies to provide full insight into basic microstructure-property relations, such as the link between particle size or spatial distribution and mechanical properties. Moreover, a capability for controlled processing of these materials is needed for the generation of samples and microstructures that can be used in the exploration of microstructure-property relations. MMCs provide a significant challenge for conventional non-destructive evaluation methods because of their complex microstructural characteristics.

The primary objective of this research is to use a systematic approach directed towards understanding the evolution of the microstructure of the SiCp-reinforced Al matrix composites and characterization by using various evaluation techniques. Special attention will be given to the distribution of SiC particles within the matrix, porosity and degree of anisotropy. For this purpose, specimens produced by hot-pressing of SiC and aluminum powders will be used.

1.3. Object and Scope

The object of this study is the examination of microstructures of SiC reinforced Al-matrix composites ensuring uniform distribution of particles and minimum amount of porosity. In order to achieve this object, the following tasks will be carried out on various samples prepared by hot-pressing, in which volume fraction and average size of SiC particles are the main variables:

- □ Non-destructive characterization of microstructures by ultrasonic velocity measurements will be carried out;
- □ The effect of particulate size on the distribution of reinforcement particles and the arrangement of the microstructure will be investigated;
- □ Anisotropy analyses will be performed using computer-based image processing methods.

Project is composed of 4 tasks. Task 1 & 2 belongs to Turkish side Task 3 belongs to Hungarian side. Task 4 is for all sides to collect results and making discussions.

Task 1 – Production of Samples

25 powder mixtures (6 sets) were delivered from Hungarian team. They were produced via hot pressing. After macroscopic examinations; samples were cut into pieces for investigations of Turkish group (density measurements, hardness, and ultrasonic sound velocity) and Hungarian group (microstructure analysis, distribution, anisotropy). The last samples were posted to Hungary within last week of May 2008.

Task 2 – Ultrasonic Sound Velocity, Hardness, Density Measurements

During examinations and having too many sets; it was decided to reduce the number of sets for further examinations. So Set1 (Alumix 123 AlCu), Set2 (Alumix 231 AlSi) and Set4 (Alumix 431 AlZn) were examined throughout the project.
Initial density and hardness measurements were carried out before image analysis processes. Ultrasonic sound velocity measurements for the applicable samples were finished. All details were tabulated in the results section.

Task 3 – Analysis of microstructures / Image Analysis

This task belongs to HUN team. The images obtained from scanning electron microscope were used for image analysis. Image analysis software was used to examine reinforcement distribution, clustering phenomena. Also macro and micro hardness tests, wear properties of the composites and DSC analysis of the samples were examined although they were not mentioned before in the project proposal.

Task 4 – Evaluation of the results and reporting

All results from remarks on production to discussions were presented separately in the following sections. In addition to the reports, several journal and congress papers were published.
* A.Makszimus, T.Pieczonka, C.H.Gür, E.Tan, Z. Gacsi: Microstructural Characteization of SiC Reinforced Aluminum Matrix Composite, Euro PM2009 Congress and Exhibition, 12-14 Oct 2009, Copenhagen, Denmark, Proceedings
* A.Makszimus, Z.Gacsi, C.H.Gür, Investigation of the microstructure and hardness of SiCp reinforced aluminum matrix composites, Materials Science Forum Vol. 589 (2008) 239-244
* A.Makszimus, C.H.Gür, T.Pieczonka, E.Tan, Z.Gacsi, Comparison of the microstructure and the mechanical properties of hot and cold-pressed Al-SiCp Composites, 5th International Powder Metallurgy Conf., Ankara, Turkey, Oct 8-12 2008

2. EXPERIMENTAL PROCEDURE

2.1. Material Used in Production

25 powder mixtures each weighting 50gr was used in this study. 6 set Al and alloy samples containing 0%, 10%, 20% and 30% wt SiC reinforcement were delivered from Hungarian group for production. The ingredient of powder samples was given in Table 1.

Table 1 Powder Samples

Set	Specimen No	Alloy	Al (wt.%)	SiC (wt.%)
1	1	ECKA Alumix 123 AlCuSiMg	100	0
	2		90	10
	3		80	20
	4		70	30
2	5	ECKA Alumix 231 AlSiCuMg	100	0
	6		90	10
	7		80	20
	8		70	30
3	9	ECKA Alumix 321 AlMgSiCu	100	0
	10		90	10
	11		80	20
	12		70	30
4	13	ECKA Alumix 431 AlZnMgCu	100	0
	14		90	10
	15		80	20
	16		70	30
5	17	Al 6061	100	0
	18		90	10
	19		80	20
	20		70	30

		Pure Al (wt.%)	SiC P220 (wt.%)	SiC F500 (wt.%)
6	21	90	5	5
	22	80	10	10
	23	70	15	15
	24	85	5	10
	25	85	10	5

During examinations and having too many sets; it was decided to reduce the number of sets for further examinations. So Set1 (Alumix 123 AlCu), Set2 (Alumix 231 AlSi) and Set4 (Alumix 431 AlZn) were examined throughout the project.

These powders were supplied by ECKA Granules GmbH&Co. The compositions of powders used were tabulated in Table 2 and the relevant SEM images were presented in Figure 1. The average particle size of powders was determined on the basis of images taken by optical microscope in case of Al-based powders and on the basis of SEM images in case of SiC.

Figure 1 SEM micrographs a) AlCu b) AlSi c) AlZn d) SiC powder

Table 2 Chemical composition of the powders

Base	**Element, wt%**							**Mean size**
Matrix	*Cu*	*Si*	*Mg*	*Zn*	*Lubricant*	*Al*	-	**µm**
AlCu	4.5	0.7	0.5	-	1.5	Bal.	-	23.9
AlSi	2.5	14	0.5	-	1.5	Bal.	-	16.9
AlZn	1.5	-	2.5	5.5	1.2	Bal.	-	24.2
Ceramic	*C*	*SiO2*	*Si*	*Fe2O3*	*Al2O3*	*CaO*	*SiC*	
SiC	0.08	0.15	0.04	0.015	0.004	0.003	Bal.	14.4

These powder mixtures were homogenized in an ultrasonic mixer for one hour at a temperature of 35°C before mailing to Turkish side.

2.2. Production Procedure

For production; a hot press system composed of furnace, control unit and a hydraulic press was used (Figure 2). Production was established in such a manner that; a die placed in furnace is heated to sintering temperature while applying load on it. The die (Figure.1b) which has a cavity of 5cm x 5cm square cross-section was used. A Molykote P37 brand high temperature grease was applied on the die surfaces before placing the powder samples in order to simplify the removal of sintered product.

Production sequence starts with powder mixing for one hour using a ball mill. Powder samples were mixed in a 100ml container with ten ZrO_2 cylpeb balls (7.15 gr/ball). 70 rpm mixing speed and dry medium (no gas or liquid environment) was selected.

The mixed powders were poured in the die cavity which was previously placed in the furnace. While the system was heated towards the sintering temperature; pressure was applied on the die punch. Furnace and control unit was designed in such a manner that; the sintering temperature could be reached in 2 hours approximately and stay at that temperature with ±5°C accuracy. After 10 minutes under 25MPa pressing pressure at sintering temperature; system is turned off and the die was removed. The sintered product was removed from the die when it was warm (40-50°C). It was ready after chip removal and cleaning from surface oil.

Figure 2 The system used in production (a) hot press and (b) die

2.3. Macroscopic Discussion

For the production route; liquid phase sintering was chosen. The key point for this procedure was to form small amount of liquid phase during sintering. From this point of view; decision of sintering temperature was critical in terms of final product quality. For example; sample#5 has a cast structure due to excessive liquid formation at wrongly chosen high temperature. During project meeting on November2007 at Miskolc it was decided to re-produce samples 5, 6, 13, 14.
Production of all sets was completed. Table 3 shows the sintering temperature and general macroscopic discussion:

Table 3 Sintering temperatures & general macroscopic discussion on samples

Set	Specimen No	Pressing Temperature (°C)	Observation
1	1	527	Smooth surface, rigid
	2	527	
	3	527	
	4	527	
2	5	565	Excess liquid formation, cast structure
	6	535	Small amount of liquid formation
	7	525	Good
	8	525	Perfect
	5 (Reproduced)	525	Perfect (Problem solved)
	6 (Reproduced)	525	
4	13	550	Surface is too rough, also cracking in center
	14	544	Rough surface
	15	544	Good
	16	544	
	13 (Reproduced)	544	Good (Roughness reduced)
	14 (Reproduced)	544	

Three type of fault had been found in the samples produced in the first term. First one is the samples which became bulk before sintering at airplane. Second one is the cast structure due to high temperature selection. And the last one is the surface roughness.
When the reproduced samples were examined; it was seen that chalk-like behavior of already sintered samples (sintered at plane) was eliminated. Also, second problem (excessive liquid formation) was reduced by correct adjusting the temperature. But, there was a surface roughness problem in the Set-4 samples containing “ECKA Alumix 431 AlZn”. Although the reproduced samples 13 and 14 had a rigid structure; roughness problem could not be solved totally. It was thought that; the problem arises from the reaction taking place between die material and powder alloy. It has been observed that roughness was reduced with the increase of die surface lubrication. Surface photographs of the composites were given in Figure 3.

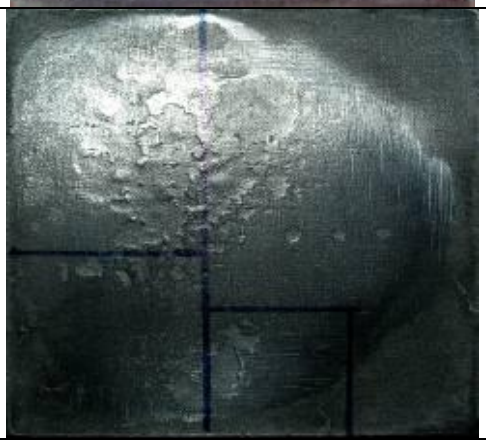
Sample #	0% SiC	30% SiC
Set 1 Alumix 123 AlCu		
Set 2 Alumix 231 AlSi		
Set 4 Alumix 431 AlZn		

Figure 3 Macro surface photographs of the produced samples

3. RESULTS

3.1. Density Measurements

Sample densities were measured via Archimedes principle. After weighting the dry samples; they were immersed into liquid medium for 24 hours to allow impregnation of the solution into open pores. Two weighting was established from these samples; one in the medium; other in air. Totally three weight was measured via Sartorius precision balance equipped with density determination kit.

M1: dry weight
M2: weight pores impregnated by liquid
M3: weight in liquid

With the use of these data two type of density was measured. The first formula without considering the pores; and second one regarding the open pores. Formulas are given below:

$$\rho_1 = \rho_{liquid} \cdot \frac{M1}{M1 - M3} \qquad \rho_2 = \rho_{liquid} \cdot \frac{M1}{M2 - M3}$$

The differences between the two densities give the amount of open porosity. To find the overall porosity; theoretical densities of the composites were calculated from rule of mixtures; since the difference between theoretical and measured density give the total porosity amount. Data for density measurements were given in Table 4.

Table 4 Sample weights and densities

Set	Specimen No	M1	M2	M3	ρ_{theo}	ρ_1	ρ_2	Porosity %	Open Porosity %
1	1	*4,6532*	*4,6641*	*2,8376*	2,7763	*2,56*	*2,55*	7,69	0,60
	2	*6,0001*	*6,0084*	*3,7319*	2,8143	*2,65*	*2,64*	6,00	0,37
	3	*5,7223*	*5,7243*	*3,6798*	2,8534	*2,80*	*2,80*	1,81	0,10
	4	*4,2693*	*4,2714*	*2,7725*	2,8936	*2,85*	*2,85*	1,43	0,14
2	5	*3,1066*	*3,1194*	*1,7645*	2,6796	*2,31*	*2,29*	13,62	0,95
	6	*5,1117*	*5,1150*	*3,0405*	2,7246	*2,47*	*2,46*	9,42	0,16
	7	*3,6241*	*3,6248*	*2,2327*	2,7712	*2,60*	*2,60*	6,01	0,05
	8	*5,9690*	*5,9706*	*3,8158*	2,8194	*2,77*	*2,77*	1,68	0,07
	5 Repro	10,7507	10,7981	6,8770	2,6796	2,39	2,36	10,82	1,22
	6 Repro	10,2850	10,3139	6,5981	2,7246	2,40	2,38	11,85	0,78
4	13	*3,4051*	*3,4289*	*2,0806*	2,7853	*2,57*	*2,53*	7,70	1,80
	14	*3,6770*	*3,6855*	*2,2323*	2,8226	*2,55*	*2,53*	9,83	0,59
	15	*4,0099*	*4,0131*	*2,5344*	2,8610	*2,72*	*2,71*	5,01	0,22
	16	*3,3309*	*3,3332*	*2,1129*	2,9004	*2,73*	*2,73*	5,71	0,19
	13 Repro	11,8283	11,8833	7,9733	2,7853	2,64	2,60	5,15	1,43
	14 Repro	10,3555	10,4542	7,0718	2,8226	2,72	2,64	3,80	3,01

Note: Measurements with italic case was measured with distilled water (ρ=1); others with Xylen (ρ=0,861).

It was observed that the porosity of the composites decreases with the increase of reinforcement amount. During liquid phase sintering small amount of liquid phase was produced. With the increase of reinforcement amount; less quantity of liquid would be formed; wetting the solid powders and rapidly solidify without letting them to move long distances.

3.2. Thermal Analysis

Samples were heated with a given route to demonstrate sintering process and examine the dimensional change during it (Figure 4). It was seen that with the increase of SiC composition; the dimensional change percentage decreases (Table 5).

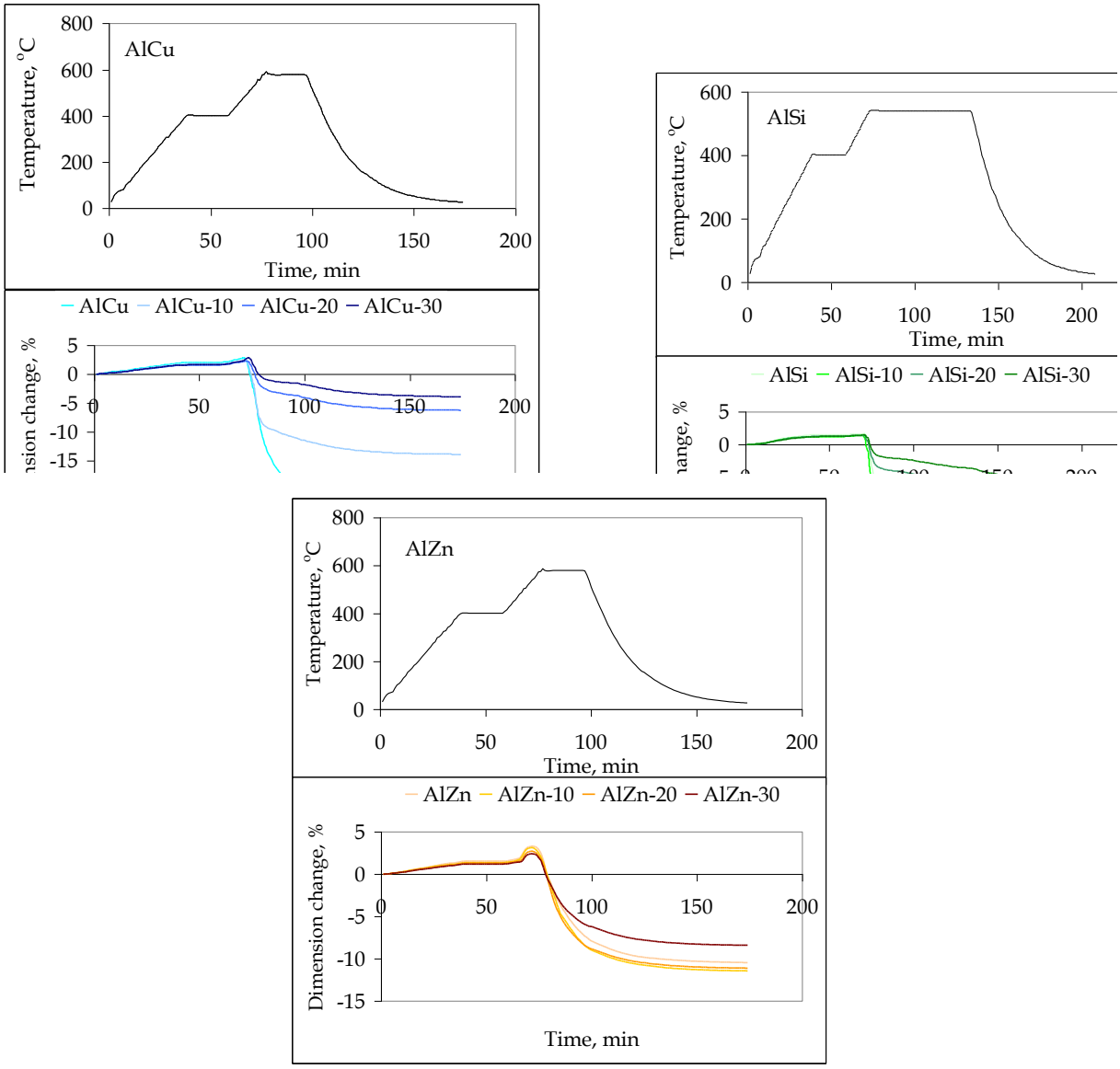


Figure 4 Dimension changes during heating (a) AlCu, (b) AlSi, (c) AlZn

Table 5 Change in dimensional reduction with SiC concentration

Matrix	SiC wt%			
	0	**10**	**20**	**30**
AlCu	23	16	6	4
AlSi	17	15	5	5
AlZn	10	11	11	8

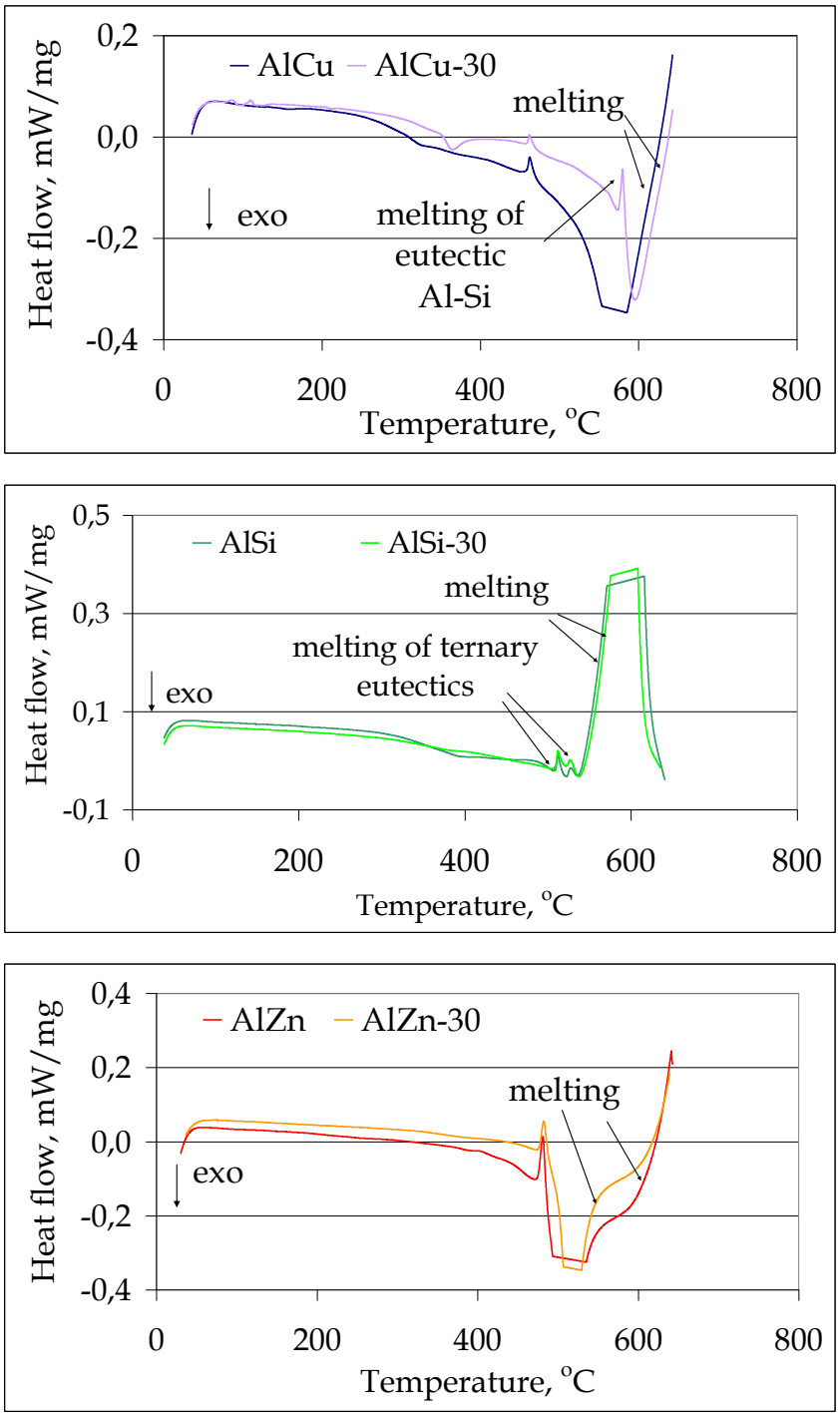


Figure 5 DSC curves for the samples

When the DSC curves for the composites investigated similar endotherms were observed namely melting of the main matrix and some ternary or eutectic phases. Also, in AlCu and AlZn systems, an exotherm peak before melting was observed as the indication of solid phase diffusion. In the AlSi system, however, enthalpy of dissolution was close up to zero, so the exotherm peak was not appearing.

3.3. Ultrasonic Sound Velocity Measurements

Sound velocity measurements were done by establishing wave transmission through the thickness of samples produced. For measurements 0.5 MHz, 1 MHz, 2.25 MHz and 5 MHz longitudinal and transverse wave probes were used. Sound velocity measurement results were tabulated as Table 6 and the change due to SiC content was shown in Figure 6.

Table 6 Ultrasonic sound velocity

Set	Sample No	Longitudinal Wave Speed (m/sec)	Transverse Wave Speed (m/sec)
Set1 Alumix 123 AlCu	1	3819	2660
	2	5147	2986
	3	6788	3566
	4	7246	3719
Set2 Alumix 231 AlSi	5 reproduced	3268	2185
	6 reproduced	2770	2858
	7	6443	3605
	8	7385	4086
Set4 Alumix 431 AlZn	13 reproduced	-	-
	14 reproduced	-	-
	15	-	-
	16	6296	-

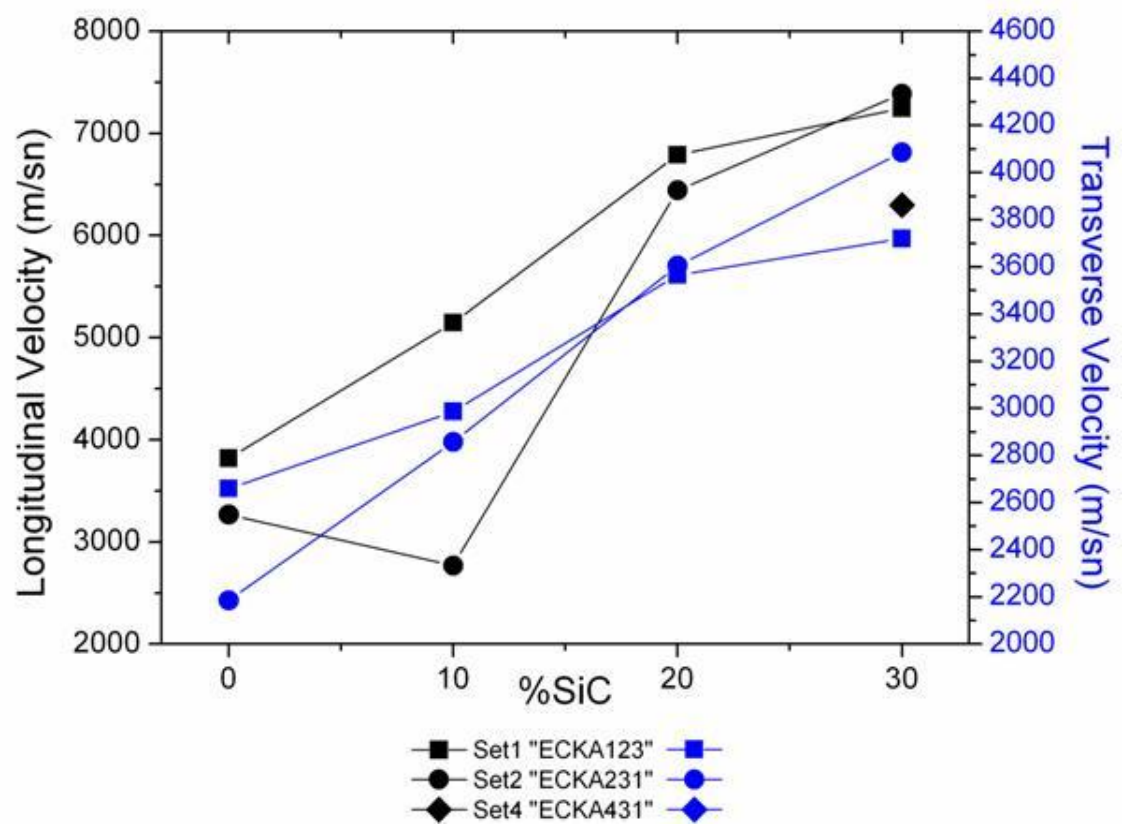


Figure 6 Change in longitudinal and transverse velocity with SiC content

Due to some impossibilities of the method (like crack inside the product); some data could not be obtained. The general trend was the increase of ultrasonic wave velocity with increase of SiC amount. For a composite material; ultrasonic wave velocity is very much dependent on average elastic modulus and density. Although SiC has a similar density as compared to aluminum; due to its nature it has higher elastic modulus. Hence, the elastic modulus of the composite increases with the increase of SiC composition. Consequently, ultrasonic waves travel faster and the velocity increases as the SiC amount increases.

3.4. Microstructure Investigations

3.4.1. General

Microstructural investigations on the produced samples were established via scanning electron microscopy. Figure 7 shows unreinforced and 30% SiC reinforced AlCu, AlSi and AlZn alloy microstructures. The EDS analysis of the phases seen in the figures was tabulated in Table 7.

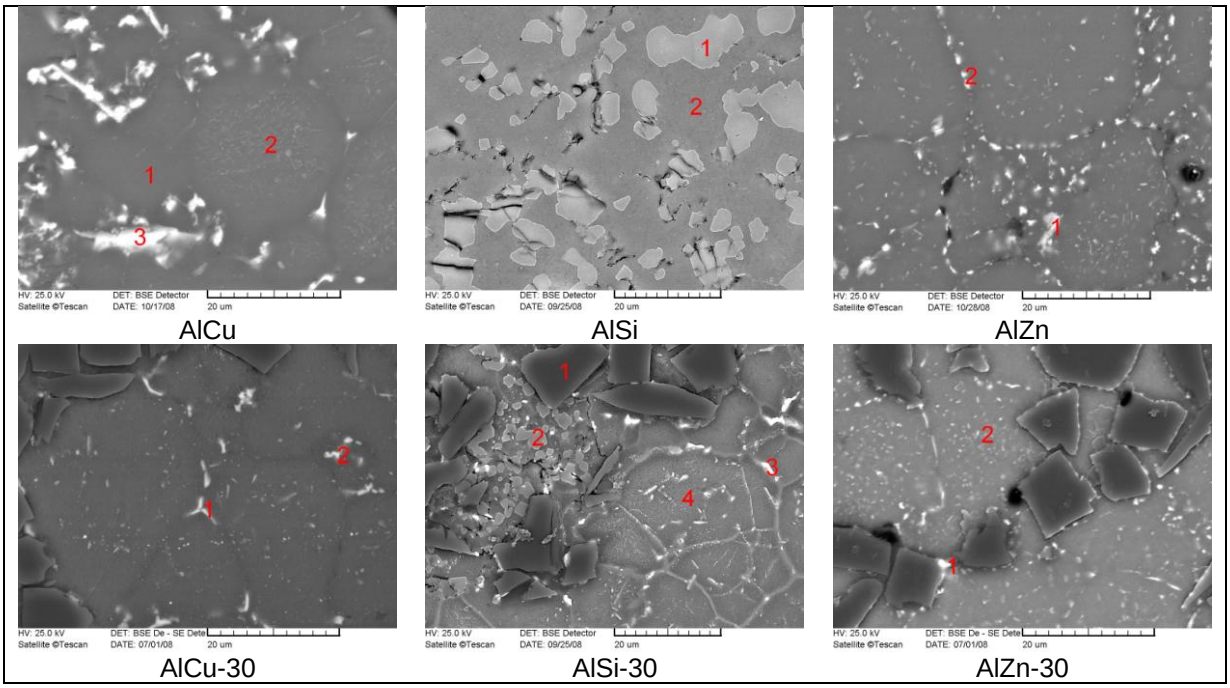


Figure 7 SEM microstructures of hot-pressed samples

Table 7 EDS analysis of some phases shown in SEM images

Analyzed Points	Elements, wt%					
	Al	**Si**	**Cu**	**Mg**	**Zn**	**Fe**
AlCu #3	68,17	0,91	22,64	1,76	-	6,52
AlZn #2	80,52	0,29	3,10	4,59	10,59	0,91
AlCu-30 #1	77,98	1,22	19,43	1,37	-	-
AlSi-30 #3	82,72	1,40	15,88	-	-	-
AlZn-30 #2	82,89	0,48	2,74	3,64	10,25	-

The investigations on SEM-images revealed that; in AlCuSiMg sets Al-Cu rich phase and in AlZnMgCu sets Al-Zn phases were observed both on the matrix grain boundaries and within the grains. At the same time they probably react with the SiC on the boundaries as well. The Al-Zn phase could be seen

within the whole structure as small points scattered all around; it was in a higher quantity at the SiC particles. Formation of tiny precipitates was also observed in the structure of AlZnMgCu alloy.
X-Ray measurements were carried out to identify the intermetallic phases in the microstructure. In none of the samples brittle Al_4C_3 was not detected. Phases found in the analysis were:

- □ AlCu and composites Al_2Cu
- □ AlSi and composites Al_2Cu & Mg_2Si
- □ AlZn and composites $MgZn_2$

During hot pressing, as a consequence of the pressing and elevated temperature the microstructure was well-densed containing few pores. Some precipitates were also observed; which were possibly due to the elevated temperature of the pressing.

In the microstructure (Figure 8) there is no interconnected porosity was observed. Typically pores were elongated perpendicularly to the pressing force. The porosity was detected in three main locations:

1) Between SiC particles/clusters (irregular shape)
2) At the Al-SiC interface (elongated shape)
3) In the Al alloy matrix (sphere-like/elongated shape)

Frequencies of porosity in the composites were given in Table 8.

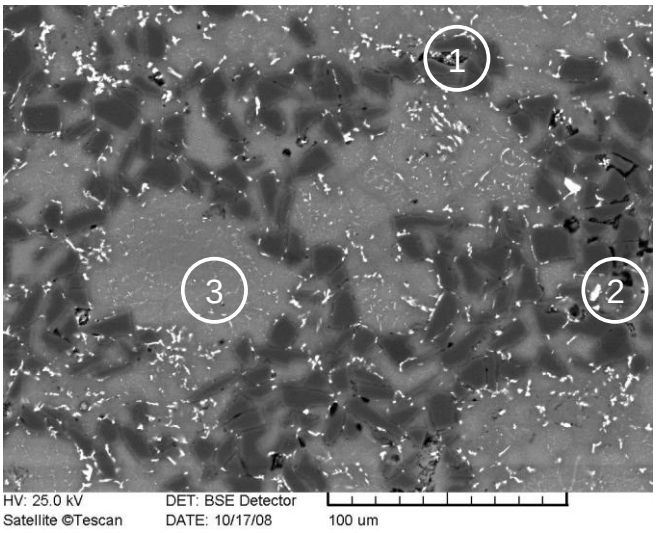


Figure 8 Typical porosity locations

Table 8 Frequency of porosity detection according to SiC composition

Matrix	**SiC amount, wt%**		
	10	**20**	**30**
AlCu	random	random	1
AlSi	3	2, 3	1, 2
AlZn	2, 3	1	1, 2

3.4.2. Distribution of the SiC

SiC area fraction obtained from the microscopic images of composite samples states valuable information for the reinforcement distribution. The area fraction of the SiC particles has been determined by Leica Qwin image analyzer software. COVT parameter was used to characterize the distribution of the particles:

$$COV_T = \frac{\sigma_T}{T}$$

COV_T variation coefficient

$\sigma_T{}^2$ standard deviation

T mean area fraction

It is known that the most homogeneous reinforcement distribution could be obtained with the reduction of standard deviation and COV_T. In Table 9, the COV_T values of the composites were presented. It was seen that COV_T increases with the increase of SiC amount (except AlSi+30%SiC). Also the variation of SiC area fraction in the composites was illustrated in Figure 9.

Table 9. COV_T parameters of the hot-pressed composites

Matrix	**Al-Cu**			**Al-Si**			**Al-Zn**		
SiC, wt%	**10**	**20**	**30**	**10**	**20**	**30**	**10**	**20**	**30**
COV_T	2.2	4.3	7.1	4.5	5.9	3.2	2.4	3.2	5.3

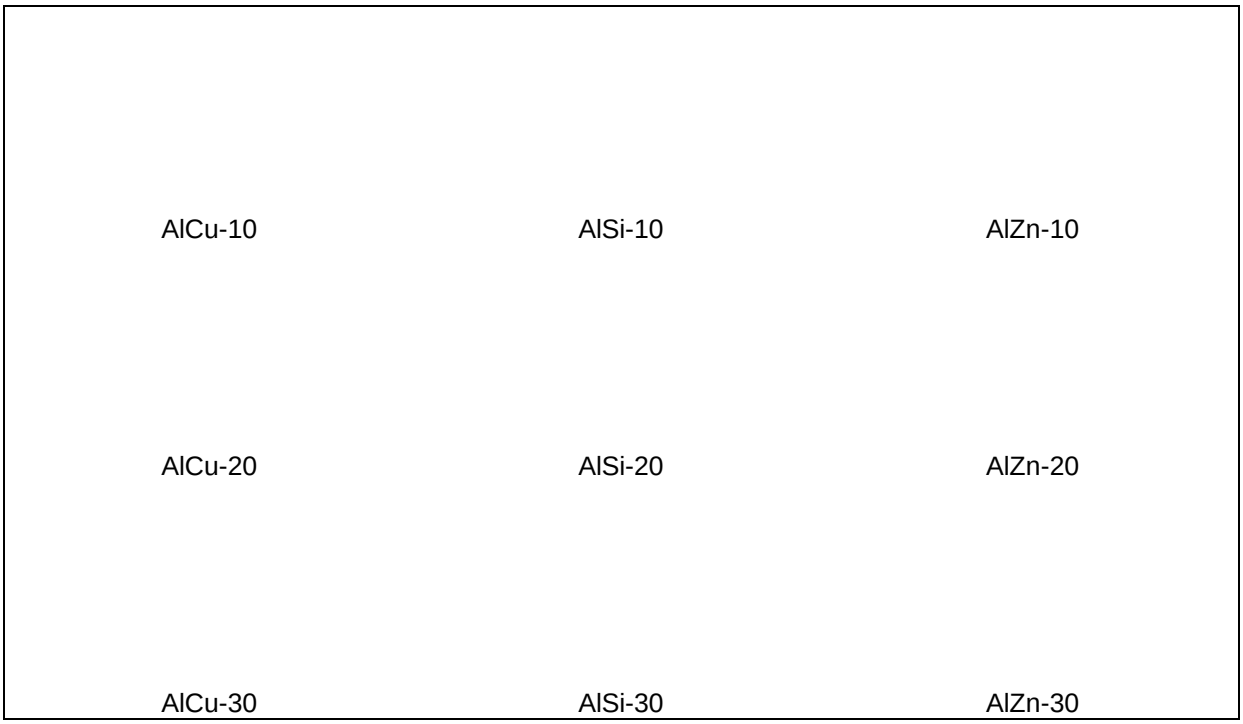


Figure 9 Variation of SiC area fraction

For optimizing the composition and production parameters of particle reinforced composites it is very useful to describe their structure quantitatively. For this purpose, it seems to be effective to generate micrographs based on a real microstructure by different computer algorithms. Micrographs (Figure 10) made by Ghosh et al. [18] were used in the course of the current measurements. Micrographs were prepared to perform measurements by implying magnification of 500x500 pixel followed by removing the boundary lines of Voronoi cells. The latter was done using the open and close operation of Cprob (Cluster Probability) software developed at the Department of Physical Metallurgy and Metalforming of Univ. of Miskolc. The Cprob “cluster close” sub-program was chosen to characterize the effect of SiC amount on the reinforcement clustering. Here the area of particles was increased by 1 pixel in each step by means of dilatation, until they grow into one particle. In this method; clustering could be indicated as a significant decrease in the number of particles. Consequently, clustering could be detected by plotting the decrease of particles number (ΔN,%) as a function of the number of dilatation

steps (NoD). In Figure 10, the relationships between particle-number (ΔN,%) and dilatation steps (NoD) for computer generated images (Gosh, 1997) was presented.

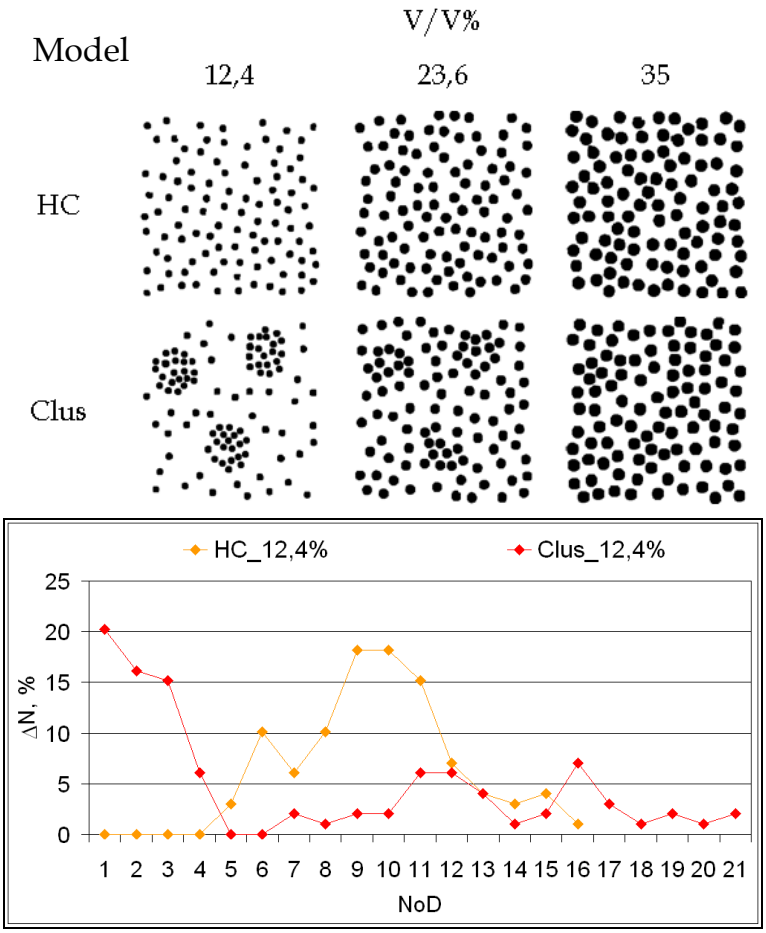


Figure 10 Relationships between particle-number (ΔN,%) and dilatation steps (NoD) for computer generated images [18]

Two types of curves could be obtained from generated microstructures. In case of a random distribution (HC-12.4%, the number shows the area fraction of particles), a characteristic distribution was observed. A sudden decrease was experienced within some dilatation steps in case of the cluster containing distribution (Clus-12.4%). This type was the indication of clusters and the distance of interfaces of particles located within the cluster could be calculated via:

$$\lambda = 2D$$

λ : distance between the interfaces (pixel)
D : number of dilatation steps

Figure 11, 12 and 13 shows the (ΔN,%)-(NoD) curves for AlCu matrix, AlSi matrix and AlZn matrix composites respectively. The curves for all composites had a tendency representing the cluster containing (Clus-12.4%) type. It has been observed that, the slope of a curve has been increased with an increase in the SiC content (except AlCu matrix composites with 10 and 20% SiC). From the results above, it was concluded that the homogeneity of the reinforcements were decreased by increasing the SiC content.

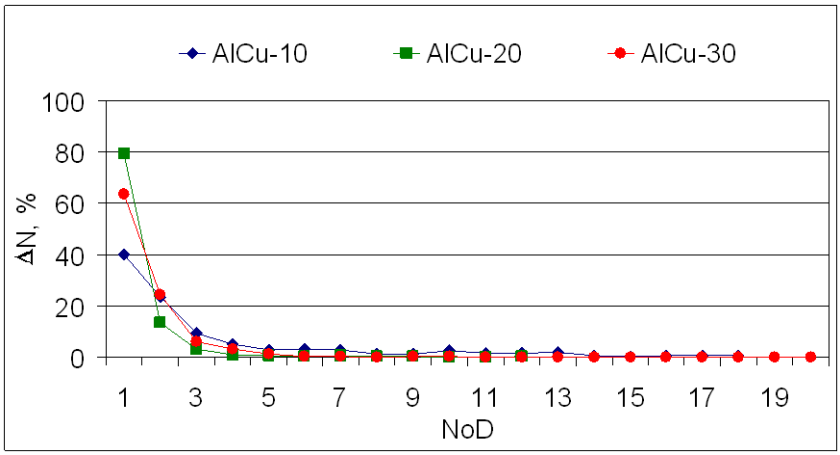


Figure 11 Particle-number (ΔN,%) - dilatation steps (NoD) relationship for AlCu matrix composites

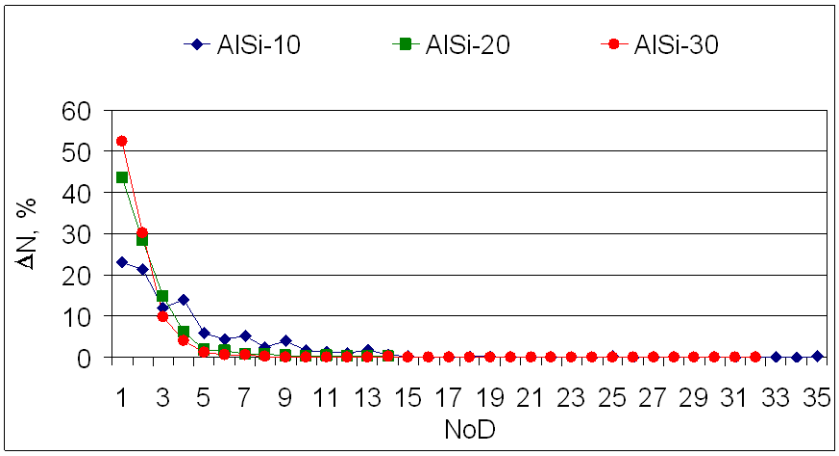


Figure 12 Particle-number (ΔN,%) - dilatation steps (NoD) relationship for AlSi matrix composites

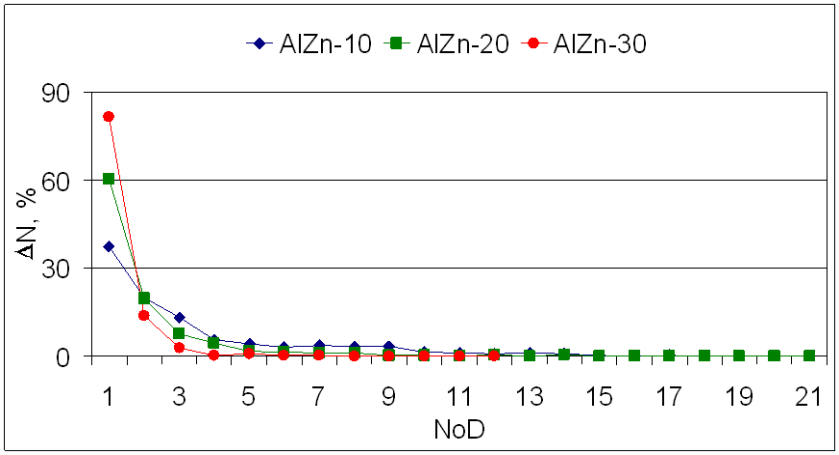


Figure 13 Particle-number (ΔN,%) - dilatation steps (NoD) relationship for AlZn matrix composites

When SiC distribution was investigated with the Quadrat method [19]; it was seen that in most cases (10-20% second phase), the distribution was exponential with positive skewness meaning most of the datas are bigger than the mean. The proportion of empty cells was smaller in case of random distribution (HC) than in clustered (Clus); and decreasing when the area fraction of the particles was increased (Figure 14). Three areas in the function of the second phase content: random, transitional, clustered zones were determined. The composite samples with 10-20% SiC content were in the random area; while the 30% SiC samples was belong to transitional or clustered zones.

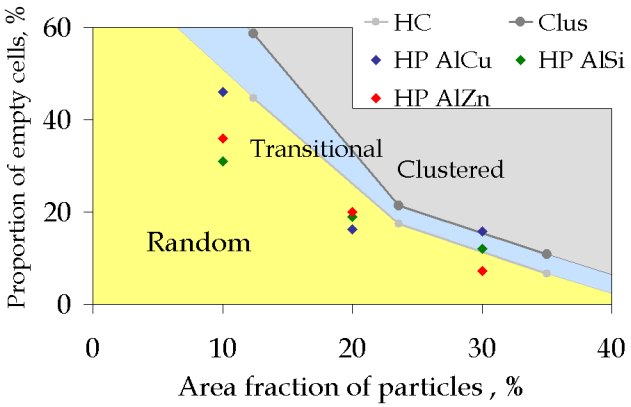


Figure 14 Classification of composites according to reinforcement distribution

3.5. Hardness

Vickers indentation was performed on the sample surface. Average hardness according to HV30 standard was measured and shown in Figure 15.

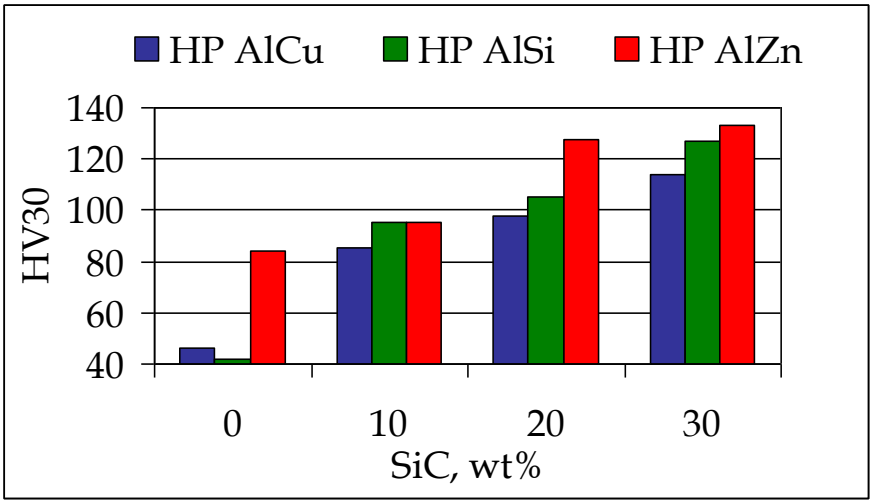


Figure 15 Macrohardness of the samples

As seen from Fig.15, the hardness of the composite increases with the increase of SiC content. It can also be concluded that the age-hardenable AlZn alloy proved to be the best in case of the macro-hardness.

Similar results were obtained in case of micro-hardness tests. The change of hardness with SiC amount was demonstrated in Figure 16. According to figure; the matrix showing the highest hardness was AlZn. As for the AlCu and AlSi matrix composites, the microhardness of the matrix increases by increasing the SiC content.

In Figure 17; the hardness values obtained from the matrix-reinforcement interface was presented. In the AlCu based samples, the interface zone was harder than that of AlZn and AlSi based ones. During liquid phase sintering, the copper in the AlCu based composites had an effective role by forming appropriate bond structure and efficient sintering. Also; it was known that copper limits the formation of porosity by activating the Al-SiC reaction and developing eutectic mixture.

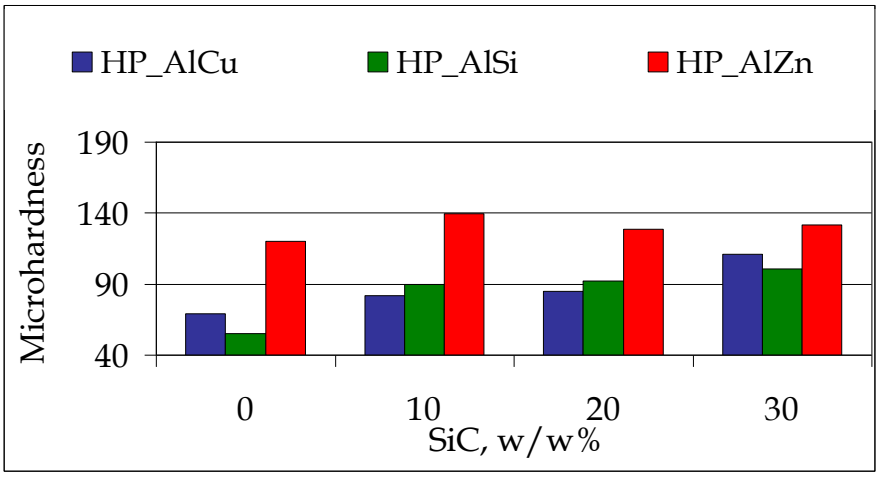


Figure 16 Change in matrix micro-hardness values

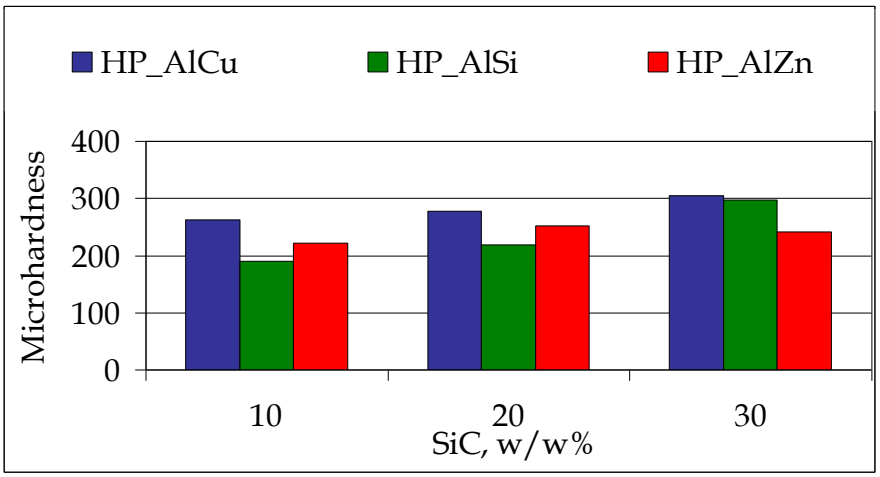


Figure 17 Change in Al-SiC interface micro-hardness values

3.6. Wear tests

One of the advantage of particle reinforced composites were its resistance to wear. In Figure 18; the worn surfaces of the composites were presented. Wear mechanisms observed were:

- Al-Cu & AlZn: abrasive wear, delamination
- Al-Si: delamination
- 30 w/w% SiC: abrasive wear (independently from the matrix material)

The wear rate of the samples decreases by adding SiC. The highest differences were between the unreinforced matrix and samples with 10% SiC content (Figure 19).

Figure 18 Worn surfaces of a) HP AlCu b) HP AlCu-30 c) HP AlSi d) HP AlSi-30 e) HP AlZn f) HP samples

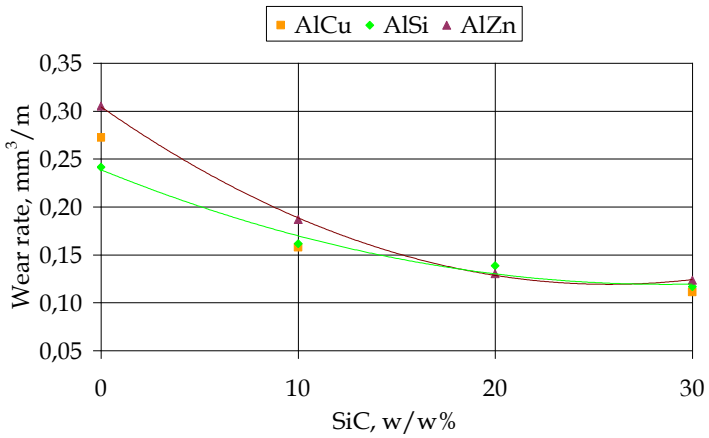


Figure 19 Wear rate of the samples

4. CONCLUSIONS

From this study; the following conclusions were obtained:

- □ Hot pressing produces compact structure having less porosity.
- □ Sintering shrinkage depends strongly on the composition of the matrix (AlCu, AlSi, AlZn), and the amount of the SiC. Formation of binary/ternary eutectic influences the shrinkage, hence the porosity.
- □ Quantity and mean size of the pores are determined by the production method and the SiC content.
- □ Based on the SiC area fraction measurements, the inhomogeneity of the hot pressed ones increases with the SiC content.
- □ Proportion of empty cells determines three area in the function of the area fraction of the particles: Random – transitional – clustered zones.
- □ The clustering can be indicated by binary morphology because a significant decrease of the number of particles can be experienced in case if the specimen contains clustering.
- □ Hardness and wear properties are influenced by the production method, the SiC content and the composition of the matrix.
- □ Hardness is increasing with the SiC content.
- □ Hot pressed samples without SiC reinforcement have smaller wear loss. At 30 w/w% SiC content, hot pressed samples have smaller wear loss.
- □ Dominant wear mechanisms are: abrasive wear and delamination.
- □ After determining the factors causing porosity with Ishikawa diagram, the amount of pores can be controlled.
- □ Pores developing in the matrix are caused by the not adequate pressing or sintering parameters.
- □ Pores caused by non-uniform batching are not distributed uniformly in the cross section.
- □ Pores between the SiC particles caused by wrong RPS ratio or mixing.
- □ Pores developing at the SiC-Al interface if the metal matrix doesn't compass round the SiC particles. In case of adequate sintering, the formation of eutectics eliminates these pores.

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TÜBİTAK
PROJE ÖZET BİLGİ FORMU

Proje No: 106M328

Proje Başlığı: Microstructural Characterization of SiC Reinforced Aluminum Matrix Composites

Proje Yürütücüsü ve Araştırmacılar: Prof. Dr. C. Hakan GÜR (Yürütücü), Araş. Gör. Evren TAN,
Araş.Gör. H. İlker Yelbay (*Mart 2008'den itibaren Kemal Davut yerine*), Araş.Gör. Pınar Karpuz (*Nisan 2008'den itibaren Evren Güneş Sonat yerine*).

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Destekleyen Kuruluş(ların) Adı ve Adresi:

Projenin Başlangıç ve Bitiş Tarihleri: 15.05.2007 – 30.10.2009

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Öz (en çok 70 kelime)

SiC takviyeli alüminyum matris kompozitler; seramik ve metallerin üstün özelliklerini birleştirerek mekanik özelliklerin iyileştirildiği malzemelerdir. Uygulama için gereken özellikler ancak mikroyapının sistematik tasarımı sonucunda elde edilebilmektedir. Mikroyapı parametrelerinin kontrolünün ve tahribatsız muayenenin, işlem parametrelerinin ve kompozitin nihai özelliklerinin iyileştirilmesinde etken role sahip olduğu düşünülmektedir. Bu bağlamda SiC ve Al tozlarından sıcak presleme yöntemiyle numuneler üretilmiştir. Partikül büyüklüğünün takviye dağılımına etkisi ve anizotropi analizleri taramalı elektron mikroskopisi ve görüntü analiz yazılımlarıyla incelenmiştir. Geliştirilen yazılımla (CProbe) takviyenin matristeki dağılım yöntemleri tespit edilmiştir. Mikroyapılar ayrıca ultrasonik ses hızı yöntemiyle karakterize edilmiştir.

Anahtar Kelimeler:

Al-SiCp, metal matrisli kompozit, mikroyapı, görüntü analizi, ses hızı

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* A.Makszimus, T.Pieczonka, C.H.Gür, E.Tan, Z. Gacsi: Microstructural Characteization of SiC Reinforced Aluminum Matrix Composite, Euro PM2009 Congress and Exhibition, 12-14 Oct 2009, Copenhagen, Denmark, Proceedings

* A.Makszimus, Z.Gacsi, C.H.Gür, Investigation of the microstructure and hardness of SiCp reinforced aluminum matrix composites, Materials Science Forum Vol. 589 (2008) 239-244

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