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Engineering Of Hybrid Composite/ Intermetallic Al-Fe-Si Fibers For Structural Materials Applications

By

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Abstract:

A ternary intermetallic compound poses even greater production difficulties, since three elements have to be mixed intimately, instead of two, for the product to be uniform in chemical composition. Furthermore, the three elements must react simultaneously to prevent binary intermetallics forming by reaction between only two of the elements. This paper presents some interesting results obtained in making a ternary Al–Fe–Si intermetallic by exothermic synthesis from its elements. The production method used is a direct metal oxidation, DIMOX, technique. Liquid state processing technique is applied to generate metal matrix composite materials utilizing Al-Si-Mg alloy. Al-Si-Mg alloy is direct metal oxidized, DIMOX, in oxidizing atmosphere at different oxidizing temperatures from 750 ^{o}C to 1100 ^{o}C. The commencement of oxidized aluminum established with the formation of aluminum oxide Al_2O_3 at 920 ^{o}C. The effect of □-Fe addition in Al-Si-Mg alloy is confirmed within aluminum solid solution as well as within alumina matrix at different ranges of oxidizing temperatures. It is shown that however the technique established with aluminum alloy reinforced with alumina, a new emerging ternary Al-Fe-Si intermetallic established in short fibers. Randomly aligned fibers, self-propagating high temperature synthesis of Al-Fe-Si intermetallic compound established in aluminum matrix as well as in alumina matrix. Exothermic reaction established close to 750 °C onset temperature for $\tilde{\alpha}$Fe powder and aluminum mixes. The effect of iron content ranging from 1 to 5 volume % dominates the occurrence of Al–Fe–Si intermetallic fibers in aluminum alloy direct oxidized at different temperatures and holding times. The results investigated in the as-cast state using scanning electricity microscope (SEM) and energy-dispersive x-ray spectroscopy (EDX). A hybrid composite established with aluminum metal matrix reinforced with alumina particulate as well as short randomly aligned intermetallic fibers.

Keywords:

Intermetallic, composite, micro and microstructure, Direct Metal Oxidation [DIMOX]

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1. Introduction:

The investigation has been carried out on a raw material of commercial AlSi1MgMn aluminum alloy. The raw material was melted in an electrical resistance furnace at different temperatures and cast in sand mould. Alloying element are added to the molten materials in the furnace with slightly increased percent to that presented in table 1. Aluminum alloy is directly oxidized in oxidizing atmosphere at different oxidizing temperature from 750^{o}C to 1100^{o}C. The process has been used with alloying elements addition ($\tilde{\alpha}$Fe powder) ranging from 1 to 5 volume % for new intermetallic and a hybrid composite formation. The commencement of oxidized alumina is achieved at 920^{o}C using aluminum Si-Mg-Fe alloy. Holding time as well as alloying elements can be controlled to achieve alumina percent [1].

This process forms an intermetallic compound (IMC) between the solid steel and the liquid aluminum. The phase of the IMC varies according to the melt composition, dipping time and temperature [2-6]. The type of IMC and its thickness are important parameters to obtain an improved bonding strength between the steel and the aluminum melt in HAD [3,8]. The IMC is analyzed by energy dispersive X-ray spectroscopy (EDS). But, in the case of EDS analysis, measuring the exact content of phases with only a slight difference in composition might cause some errors when approaching the resolution limit of the instruments. When the energetic electrons in the microscope strike the sample, the interaction volume that can also make the error in the analysis examining thick or bulk specimens can occur inside the specimen. In the Al-Fe-Si system it is not evident to determine the exact composition and phase even with the help of several established analysis technology such as EDS. Morphology of specimens was made in the scanning electron microscope HITACHI S-3400 (SEM). Phase identification of the IMC correlated with G. Mrówka-Nowotnik et. Al [9].

Additions of α-Fe powder as well as zinc powder to the liquid solution are established with dominant effect in creating new morphology of intermetallic formation. These intermetallic particles had different morphologies, such as platelet, rod, polyhedron or “Chinese script” as well as faceted structure morphology. The fibers, however they are randomly oriented at the surface of test sample, fracture surface revealed square elongated fibers in a faceted structure. Ferritic steel powder is added with stirring at different percentages from 1 to 5 wt%. The DIMOX process is generally carried out and controlled by three parameters: oxidizing temperatures, holding time and alloying element effects. α-Fe is added to state its effect intrinsically and extrinsically on Al-MMC/alumina particulate. However, the thickness of α-Fe (Al) saturated over a diffusion time longer than 30 minutes at 950°C with concentration of voids in oxidized aluminum alloy. Fiber diameter detected within 10 μm width and of 500-800 μm length at the tested sample surface. Fracture surface, in addition revealed square faceted structure. The kinetics of intermetallic formations as well as hyberdization of composite materials established in low cost light weight raw materials recycled in a DIMOX process.

2. Experimental Results and Discussion

Figure 1 presents as received material, AlSiMgMn alloy, in high magnification with low percent (~1%) of α-Fe addition for 15 minute holding time. Grain boundaries revealed microvoids with different morphology as well as clear grain refinement. Figure 2 presents the same as-cast microstructures of AlSiMgMn alloy with slight increase (~2%) of α-Fe additions and for 30 minute holding time. It presents different oriented whiskers of fibers aligned and randomly oriented in aluminum matrix along with grain boundaries at 950^{o}C. There is a correlation between grain boundary voids and crack shape with intermetallic fibers shape and orientation along grain boundaries. Addition of (~3%) α-Fe steel at 1000^{o}C for 30 min, revealed intermetallic dendritic fibers Al-Si-Fe aligned in aluminum matrix within grains as presented in Figure 3. Figure 4 presents agglomeration and growth of intermetallic fibers Al-Si-Fe aligned in aluminum matrix DIMOX with addition of (~4%) α-Fe steel at 1000^{o}C for 60 at min. holding time.

Increasing α-Fe % (~5%) as well as increasing temperature 1050^{o}C established with new morphology as well as alloy segregation Figure 5. Figures 5-8 presents different types of intermetallic compounds correlated with G. Mrówka-Nowotnik et all [9]. These images show different types of intermetallic compounds located within grain, in contrast to Mrówka-Nowotnik et all [x] where, it located at grain boundaries. These intermetallic particles had different morphologies, such as platelet, rod, polyhedron or “Chinese script” as well as whishes. The more addition of α-Fe % has an effect of growth kinetics of localized and new morphology of intermetallic formations. Figure 9 presents fracture surface reveals the commencement of aluminum oxide, alumina, formation as well as intermetallic with □-Fe additions at 950 ^{o}C for 30 minutes. In addition, Figure 10 presents segregation of alloying elements and the kinetics of Al-Si-Fe formations with □-Fe additions at 950 ^{o}C for 30 min.

Figures 11, 12 and 13 present SEM with different localized zone of interest. Figure 11 and 12 simultaneously present fracture surface of alumina with fine grain structure with residual aluminum, short squared intermetallic fibers embedded in alumina with residual aluminum. Figure 13 reveals SEM of delocalized zones of residual aluminum, alumina and embedded intermetallic whiskers within structure. The addition of low melting depressant, zinc, with □-Fe addition established with hybrid structure. Figure 13 presents hybridization of fibers and faceted intermetallic Al-Si-Fe in aluminum matrix DIMOX merged with alumina at 1050^{o}C for 60 at minutes holding time. Not only a hybrid structure established, but also delocalized zone of interest; alumina, aluminum with alumina, aluminum with intermetallic or purely intermetallic zones as presented in Figures from 14 to 20.

EDX established to reveal intermetallic and zone of interest for Al, Si, and Fe distribution along line detection within those zones Figs 14 to 16 respectively. Figure 17 and 18 revealed transition from metallic structure to intermetallic structure with EDX analysis at point detection. It revealed bulk aluminum dominant with (~47%), however a segregation established within Fe % and Si %. Intermetallic and faceted structure established with the effect of α-Fe % as well as holding time effect. Figure 19 reveals delocalized aluminum oxide zone where it resembled ceramic foam materials. Figure 20 also presents delocalized zone of interest of intermetallic zone in a faceted structure.

Kinetic of intermetallic formation passes through different steps from Figure 1 till Figure 8. However, there are clear correlations between Figure 1 and Figure 2, related to the commencement of intermetallic formation along grain boundaries, Figure 3 and Figure 5 reveal clear dendritic and delocalized zones of interest within grains. Increase of temperature, with α-Fe % addition resolve more delocalized zone of interest Figure 5-8, where there is a mutual interaction between direct oxidation of aluminum at this temperature that revealed alumina (Figure 9) along with intermetallic fibers formations within fracture surface. Widmanstäten structure established (Figure 6) along with alloying element segregation (Figure 7) resolve within induced thermal or stress mismatch to reveal slip bands (Figure 8).

On the other hands, the kinetic of formation of alumina and/or intermetallic formations established within fracture surfaces and presented in Figure 9. It reveals the commencement of alumina as well as intermetallic fiber formation with α-Fe additions at 950 ^{o}C for 30 min. More evidence of alumina formation as well as segregation channels revealed aluminum and silicon effect presented in Figure 10. More segregations established with delocalized zones of alumina (Figure 11), alumina with embedded short fibers of intermetallic (Figure 12), or delocalized zones of intermetallic faceted structure (figure 13). Figure 13 presents the three different zones of interest at 1050^{o}C for 60 minutes holding time in furnace. Not only fracture surfaces reveal these delocalized zones of interest, SEM also established to reveal these evidences (Figure 14-15-16). EDS established to reveal the dominant alloying element effect on intermetallic formation in a hybrid composite structure. Aluminum has normal stable level reveal alumina (at right) and aluminum (residual at left) presented in Figure 14. Figure 15 and 16 reveal silicon (Si) and ferritic steel (α-Fe) distribution simultaneously. Low level established in alumina zone (at right), while slight peaks established within residual alumina zone in intermetallic whiskers. Figure 17 presents intermetallic fibers Al-Si-Fe aligned in aluminum matrix DIMOX at 1000^{o}C for 30 min. holding time with the following composition in a weight percent; 47.084 % Al, 20.226% Si and 32.690 % Fe. Figure 18 presents intermetallic fibers Al-Si-Fe aligned in aluminum matrix DIMOX at 1050C for 60 min. holding time with the following composition in a weight percent; 47.036 % Al, 31.346% Si and 21.617 % Fe. Slight differences appear in both Si and Fe % due to delocalization in delocalized zones.

Figure 19 present alumina particulates porous structure with no residual aluminum matrix as a new emerged ceramic foam. Figure 20 presents intermetallic delocalized zone in a faceted structure with more DIMOX temperature 1100^{o}C and more holding time 90 minutes. Temperature and holding time are dominant in more segregation and intermetallic formation (47.036 % Al, 31.346% Si and 21.617 % Fe). The results of EDS analysis made on the coarse intermetallic phases are present in Fig. 14, 15 and 16 for intermetallic constituents' distribution, Fig. 17, 18 and 20 for intermetallic constituents' compositions and Table 2. Comparison of selected reference data to the results obtained by EDS scans showed good agreement between them (Table 2).

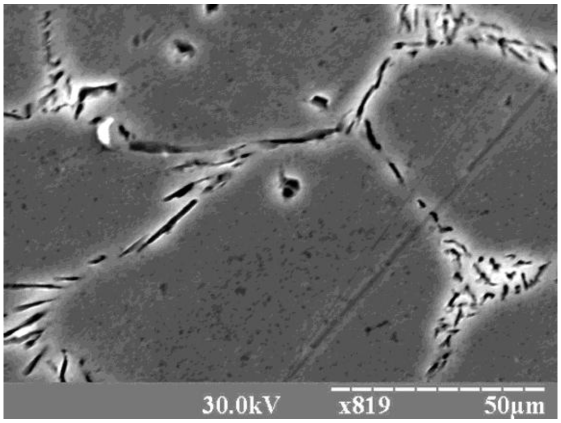

Figure (1): *Aluminum alloy directly oxidized at 950°C in with steel additions for 15 min. holding time in Al-Si-Mg alloy with 1% steel additions.[high mag.].*

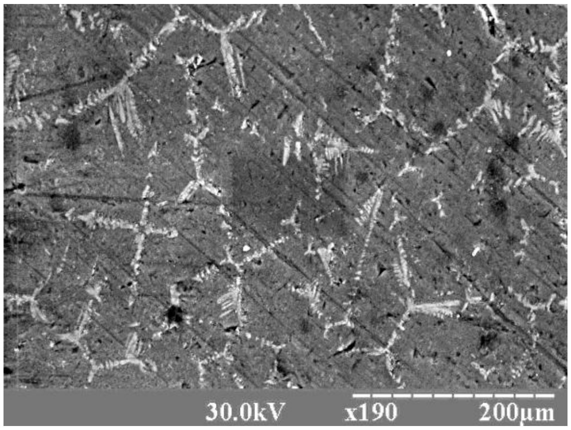

***Figure (2)*:** Intermetallic fibers Al-Si-Fe aligned in aluminum matrix DIMOX at 950°C for 30 min. in Al-Si-Mg alloy with 2% steel additions.[low mag.].

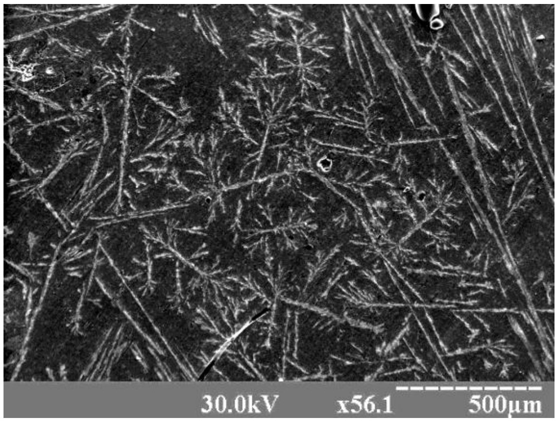

Figure (3): *Intermetallic fibers Al-Si-Fe aligned in aluminum matrix DIMOX at 1000°C for 30 min. holding time (Al-Si-Mg alloy with (~3%) α-Fe steel additions).*

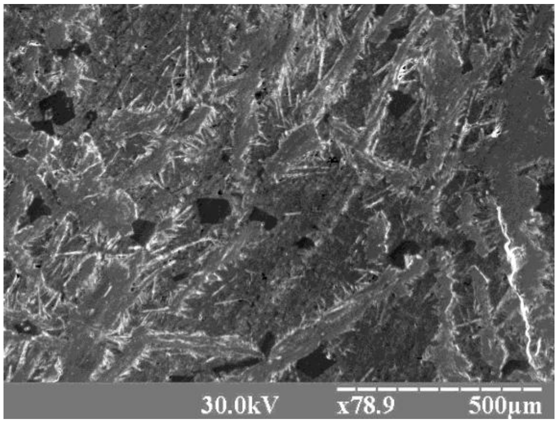

Figure (4): *Intermetallic fibers Al-Si-Fe aligned in aluminum matrix DIMOX at 1000^{o}C for 30 min. holding time (Al-Si-Mg alloy with (~3%) α-Fe steel additions).*

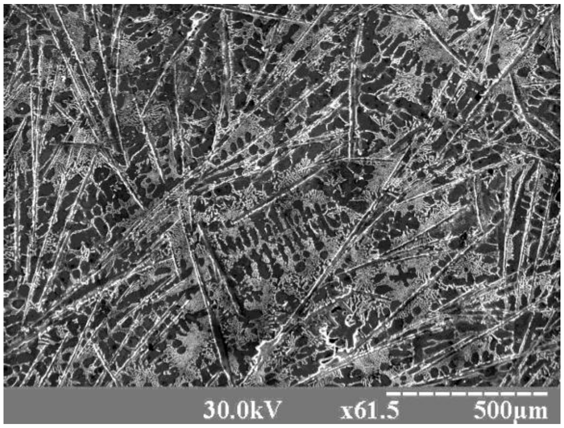

Figure (5)**:** Intermetallic Al-Si-Fe morphology in Al-Si-Mg alloy DIMOX at 1050 for 30 min. with α-Fe steel additions.[low mag.]

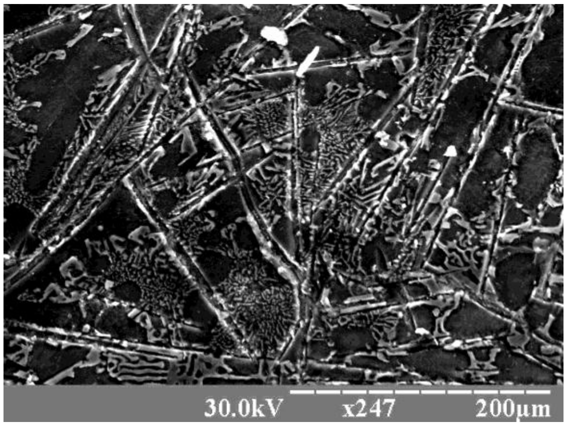

Figure (6): Intermetallic Al-Si-Fe morphology in As-Si-Mg alloy DIMOX at 1050 for 30 min. with α-Fe steel additions.[low mag.]

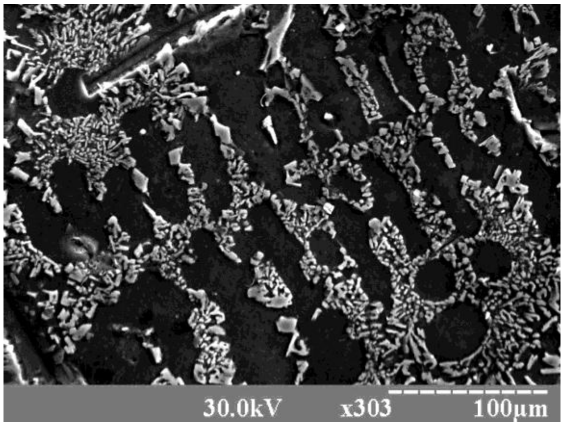

Figure (7): Intermetallic Al-Si-Fe morphology in As-Si-Mg alloy DIMOX at 1050 for 30 min. with α-Fe steel additions.[high mag.]

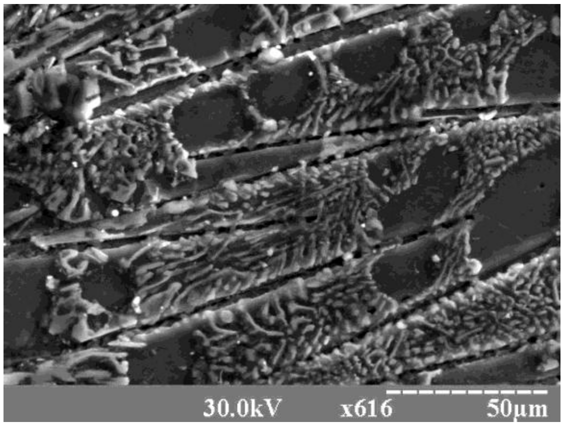

***Figure (8)*:** Intermetallic Al-Si-Fe morphology in As-Si-Mg alloy DIMOX at 1050 for 30 min. with α-Fe steel additions. [High mag.]

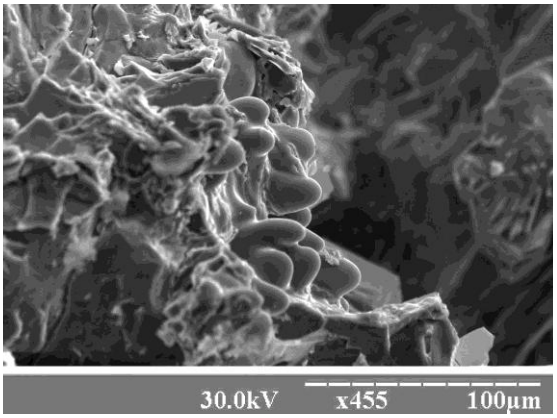

Figure (9): Fracture surface with the commencement of alumina as well as intermetallic with α-Fe additions at at 950 °C for 30 min

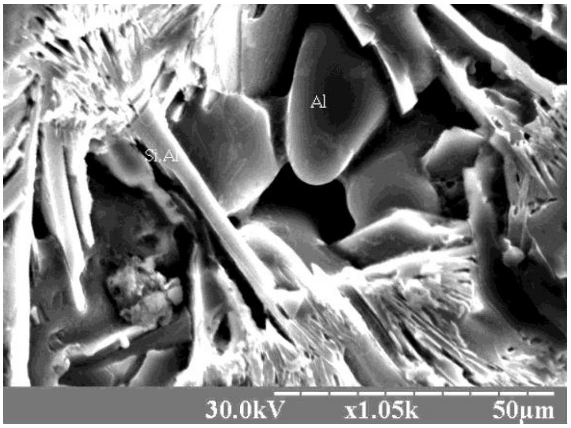

Figure (10): Segregation of alloying elements revealed kinetics of Al-Si-Mg- with Fe additions at oxidized at 950 °C for 30 min.

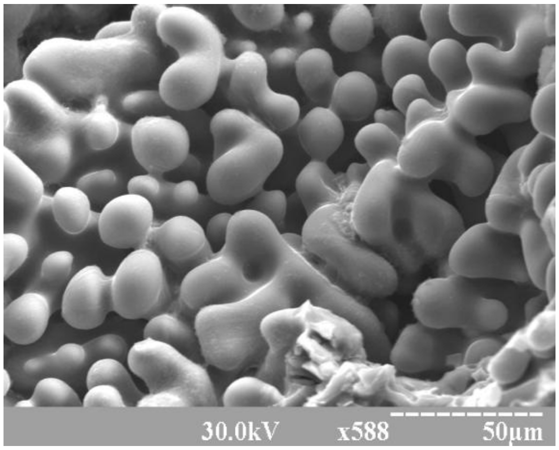

***Figure (11)*:** Alumina particulate synthesis by DIMOX with residual aluminum matrix at 1000°C with 30 min. holding time.

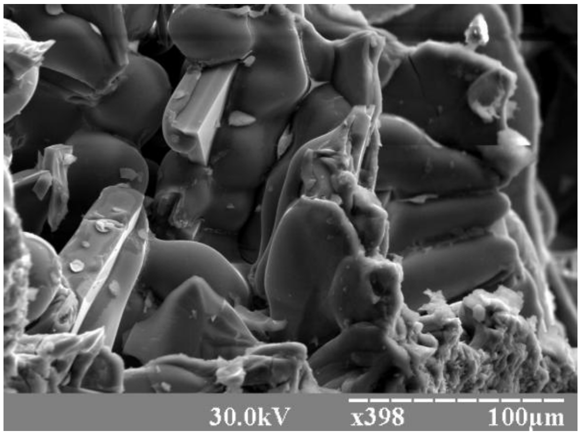

Figure (12): Intermetallic fibers Al-Si-Fe in Alumina particulate delocalized in alumina with residual aluminum matrix oxidized at 1000°C with 30 min. holding time.

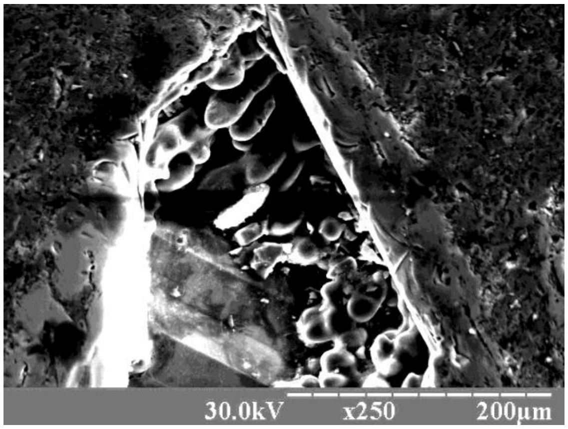

Figure (13): Hybridization of fibers and faceted intermetallic Al-Si-Fe in aluminum matrix DIMOX merged with alumina at 1050^{o}C for 60 at min. holding time.

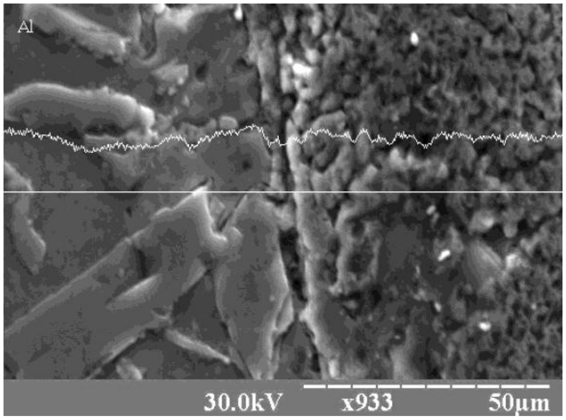

***Figure (14)*:** Aluminum distribution in aluminum matrix DIMOX at 1100^{o}C for 90 min. holding time.[High mag.].

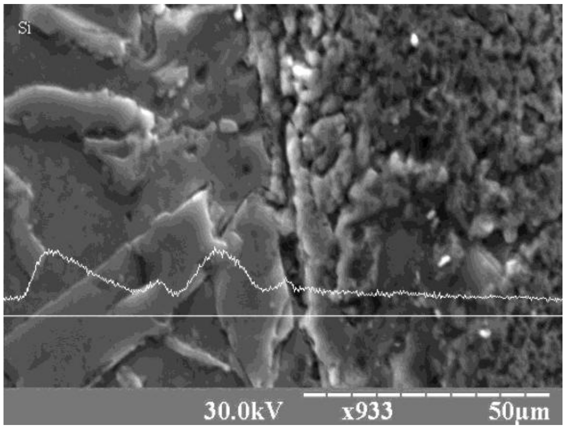

Figure (15): Silicon distribution in aluminum matrix DIMOX at 1100^{o}C for 90 min. holding time.[High mag.].

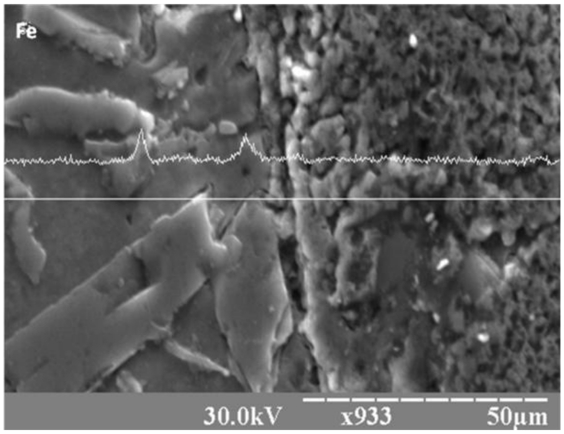

Figure (16): Ferrous distribution in aluminum matrix DIMOX at 1100°C for 90 min. holding time.[High mag.].

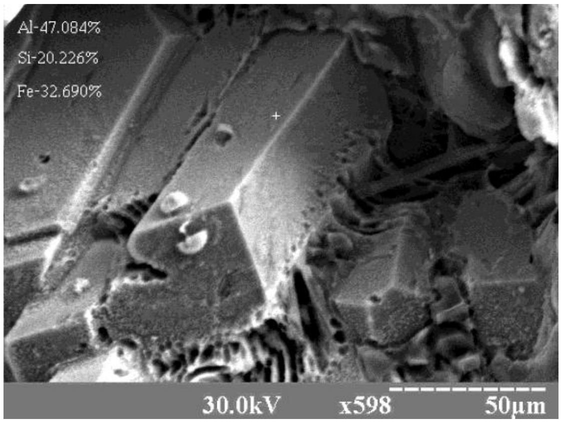

Figure (17)**:** Intermetallic fibers Al-Si-Fe aligned in aluminum matrix DIMOX at 1000°C for 30 min. holding time.

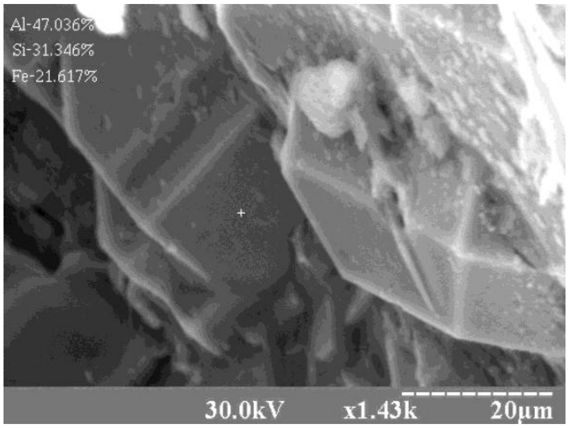

Figure (18): Intermetallic fibers Al-Si-Fe aligned in aluminum matrix DIMOX at 1050C for 60 min. holding time.

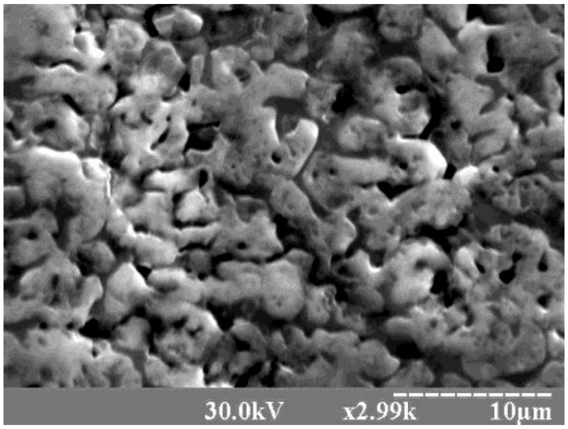

Figure (19): delocalized zone of alumina matrix from Al-DIMOX at 1100°C for 90 min. holding time with Zn additions. [High mag.]

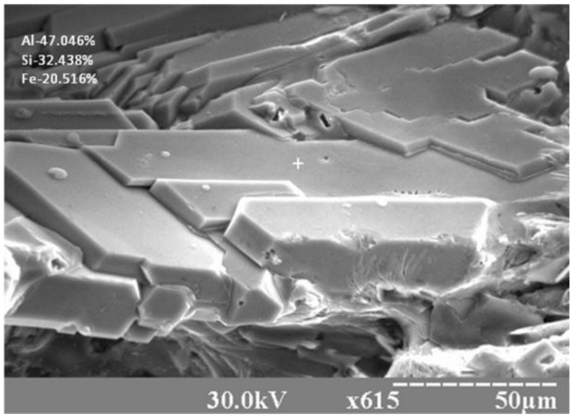

***Figure (20)*: Agglomeration of Intermetallic Al-Si-Fe faceted in aluminum matrix DIMOX at 1100°C for 90 min. holding time with Zn additions. [High mag.]

Table 1. Chemical composition of as received used raw materials

Elements	Si	Mg	Fe	Ni	Cu	Mn	Ti	Zr	V	Zn	Sr	Al
From	11.0	0.5	0.0	0.05	0.06	0.0	0.05	0.12	0.05	0.05	0.001	Balance Wt%
To	14.0	1.5	0.8	0.9	0.8	1.0	1.2	1.2	1.2	0.9	0.1	

Table 2. Table 2. Composition of the observed intermetallic phases (%wt).

Phase	Al%	Si%	Fe%	Reference
From	47.036	20.226	20.32	This work
To	47.084	32.438	32.690	
From	balance	12	25	5, 9
To	balance	15	30	

3. Conclusions:

The alloy used in this study possessed a complex as-cast microstructure. New emerging process established for a hybrid composite processing. DIMOX introduced not only for composite processing, but also for intermetallic processing. The kinetics if intermetallic fibers at micro scale by adding solid α-Fe powder to liquid aluminum alloy 6xxx introduced. Intermetallic mechanism of formation as well as alumina in a single phase or in a bulk merged phases established. Effect of DIMOX temperature as well as holding time with the addition of α-Fe powder established with localized different zone.

After different parametric study of DIMOX process, engineering of intermetallic shape and morphology, as well as hybrid composite materials established with more correlation with the conventional process used before. Depending on the composition and DIMOX parameters, the complicity of interaction between aluminum, aluminum oxide and intermetallic fibers are established. A clear delocalized zone of interest and reinforcement behavior from short whiskers, fibers, faceted structures to a hybrid composite are also observed. Scanning electron microscopy, SEM and energy dispersive X-ray spectroscopy, EDS, established with a wide range of intermetallics identified phases.

Synergetic effects of alloying elements as well as DIMOX parametric induced different intermetallic kinetics as well as delocalized zone of interests. DIMOX processing induced Engineering of new materials with different functions. A hybrid composite of aluminum metal matric reinforced with alumina particulate as well as intermetallic whiskers and/or fibers are introduced. In addition delocalized zone of interests may also introduced with a new porous material (metallic foam)

References:

[1] B. M. Rabeeh, A.M.Elmahallawy, S. H. Haddad, and M. A. Ibrahim “Development and Processing of a functionally Gradient Ceramic/Metal Materials ", Proceeding of the 9th Applied Mechanics and Mechanical Engineering Conference, AMME Conf. 16-18 May, 2000, Cairo, Egypt.

[2] C.D. Marioara, S.J. Andersen, J. Jansen, H.W. Zandbergen, The influence of temperature and storage time at RT on nucleation of the □ ’’ phase in a 6082 Al-Mg-Si alloy, Acta Mater 51 (2003) 789-796.

[3] N.C.W. Kuijpers, W.H. Kool, P.T.G. Koenis, K.E. Nilsen, I.Todd, S. van der Zwaag, Assessment of diffrent techniques for quantification of □-Al(FeMn)Si and □-AlFeSi intermetallics in AA 6xxx alloys, Materials Characterization 49 (2003) 409-420.

[4] G. Mrówka-Nowotnik, J. Sieniawski, Influence of heat treatment on the micrustructure and mechanical properties of 6005 and 6082 aluminium alloys, Journal of Materials Processing Technology 162-163 (2005) 367-372.

[5] L.F. Mondolfo, Aluminium Alloys, Structure and Properties. Butterworths, London-Boston, 1976.

[6] M. Warmuzek,K. Rabczak, J. Sieniawski, The course of the peritectic transformation in the Al-rich Al–Fe–Mn–Si alloys, Journal of Materials Processing Technology 162-163 (2005) 422-428.

[7] M. Warmuzek, J. Sieniawski, K. Wicher, G. Mrówka- Nowotnik, The study of distribution of the transition metals and Si during primary precipitation of the intermetallic phases in Al-Mn-Si alloys, Journal of Materials Processing Technology 175 1-3 (2006) 421-426.

[8] L.A. Dobrzanski, W. Borek, R. Maniara, Influence of the crystallization condition on Al–Si–Cu casting alloys structure, Journal of of Achievements in Materials and Manufacturing Engineering 18, 1-2 (2006) 211-214.

[9] G. Mrówka-Nowotnik, J. Sieniawski, M. Wierzbińska, Analysis of intermetallic particles in alsi1mgmn aluminium alloy, Journal of of Achievements in Materials and Manufacturing Engineering, Volume 20, issues 1-2, january-february 2007.155-158.

Nomenclatures:

α …	ferrite
DIMOX	Direct Metal Oxidation
SEM	Scanning Electron Microscopy
EDS	Energy Dispersive X-Ray Spectroscopy