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Alloy Segregation and Nano Structural Al-Composite Intrinsically Processing Via DIMOX, Rheocasting and Thixocasting

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ABSTRACT

Alloy segregation along with delocalized zone of interest is achieved in direct metal oxidation, DIMOX. Recycling of automotive aluminum piston scrap, alloy 6xxx, is achieved at temperature 1050ºC for different holding time 10, and 60 minute. Alloying element, Boron, is added via the addition the boric acid or borax. Liquid state processing, DIMOX, along with semisolid processing, Rheocasting and Thixocasting, are introduced for the control structural micro constituents. The novel technique, DIMOX, induce in situ aluminum metal matrix Nano-composite intrinsically established. The control of composite micro-constituents location, shape and size is also established via semisolid process, Rheocasting and Thixocasting, each at 800ºC for 10 minute, holding time. Microstructural analysis and mechanical characterization are conducted. Rheocasting, induce alloy segregation more dominated into intermetallic and or ceramic alumina phases in a bulk hybrid composite matrix. In contrast, semisolid, Thixocasting, processing dominate more surface coating rather than bulk processing. Boron has a distinguishable effect as an inoculant, induce borate glass in dispersed phases, bulk whiskers or as coating phase. The application of liquid state processing, DIMOX, with the control of micro constituents with the synergetic effect of boron is introduced for new nontraditional Nano-structural hybrid materials.

Keywords
Alloy segregation, Boric acid, Borax, DIMOX, Hybrid, Nano-composite, Recycle, Rheocasting, and Thixocasting.

1. INTRODUCTION

Metal matrix composites is introduced intrinsically via liquid state processing. Directly-Metal-Oxidation (DIMOX) [1]. Aluminum alloy matrix composites attract much attention due to their lightness, high thermal conductivity, moderate casting temperature and others. Various kinds of ceramic materials, e.g. SiC, Al_2O_3, MgO and B_4C are extensively used to reinforce aluminum alloy matrices [2-3]. Automobile scrap, aluminum piston parts, 6xxx alloy, is recycled via DIMOX for the objective of light weight, low cost, domestically and high performance (LLDH) materials. Parametric study is achieved at temperature of 1050ºC, with extremities holding time 10 and 60 minute [1-2]. No clear evidence of oxidation before 920ºC, while porosity is dominant around 1100ºC [3].

A semisolid reaction, Rheocasting is introduced at 800ºC for 10 minute to control micro-constituents and porosity shape and size. A new Non-traditional approach based on the synergetic effect of alloying elements, DIMOX and semisolid (Thixocasting) processing is also introduced at 800ºC for 10 minute [3-4]. Boron, is introduced via the addition of 10 wt. %, equal weight fraction mixture, of Borax and Boric acid. The kinetic of processing is in limited understanding. The objective is to introduce new de-localized intermetallic and /or ceramic phases intrinsically and in situ Al-Metal Matrix Composite. One of the major challenges when processing MMCs is achieving a homogeneous distribution of reinforcement in the matrix as it has a strong impact on the properties and the quality of the material [5-6].

The effect of Boron addition via boric acid and borax powder addition resolved with different functions. Synergetic effects of Boron along with liquid state, DIMOX, as well as semi-solid state processing, Rheocasting and Thixocasting have a dominant effect on the morphology of composite constituents. The kinetic of a new hybrid material; aluminum alloy reinforced with ceramic alumina as well as intermetallic delocalized phases (fibers, whiskers, equiaxid grains) in residual aluminum matrix is introduced. Nucleation and growth of reinforcement has an implicit effect of alloying element segregation in bulk aluminum, via liquid state and semisolid state processing [6-7]. Different delocalized zone of microstructures resolve bulk aluminum and porous aluminum matrix [8]. Boron addition has a synergetic new functional hybrid material. Ceramic alumina whiskers, instead of fibers with less percentage of intermetallic fibers are introduced. A 10 wt. % mix of boric acid and borax addition, in equal weights, is also introduced for inducing aluminum borate, intermetallic and /or ceramic oxide material that controlled via semi solid reaction, rheocasting and thixocasting [9].

A clear constituent wettability is obtained with DIMOX and is being controlled in shape and size for better mechanical properties as well as a new emerging process for producing hybrid aluminum. The addition of Boron with both DIMOX and rheocasting process induced different morphology of aluminum oxide, ceramic, reinforcement with intermetallic whiskers in a residual aluminum metal matrix composite. Alloying element segregation has its synergetic effect for introducing a new hybrid composite material intrinsically. The commencement of α-alumina, intermetallic fibers/whiskers, and the segregation, of alloying elements is introduced in a residual bulk aluminum matrix need to be investigated.

2. EXPERIMENTAL WORK

Four sets of samples are prepared to characterize the effect of DIMOX and Rheocasting, and Thixocasting on material attributes, compared to as received (sample recycled at 750^{o}C). The first set of samples DIMOX at 1050^{o}C for 10 and 60 minutes holding time without the addition of alloying elements and poured in a metallic mold. The second set of samples is DIMOX at 1050^{o}C for 10 minutes holding time with the addition of 10 wt. % mix of (Borax + Boric Acid) in a metallic mold. The third set of samples is to characterizing the effect of addition of semi-solid reaction along with Boron effect. Rheocasting is applied at 800^{o}C for 10 minutes, with the addition of 10 wt. % mix of (Borax +Boric Acid) for the DIMOX samples. The fourth set of samples is the application of semi-solid, Thixocasting, on DIMOX samples with 10 wt. % mix of (Borax + Boric Acid).

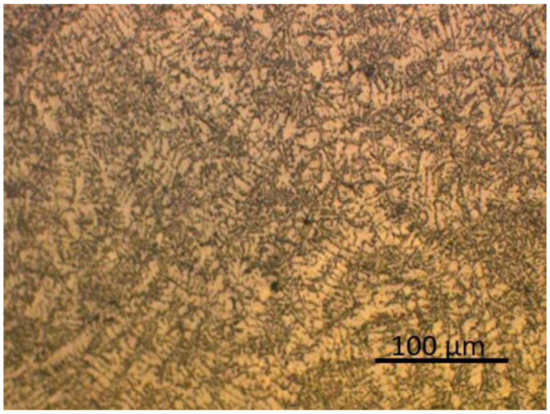
Mechanical characterization is introduced via tensile testing and through 3-point bending testing. Modulus of rupture is calculated for square cross section sample and compared. Modulus of rupture [MOR] is calculated for different samples through DIMOX process and/or DIMOX followed by rheocasting. In addition, scanning electron microscopy and energy dispersive X-ray spectroscopy (EDS) is utilized for both polished and etched surface and fracture surfaces. The control of composite micro-constituents is established by semisolid rheocasting process which tends to reduce internal stress raisers of flake porosity to a controlled shape and size. Rheocasting has a clear evidence of controlling constituents shape and size in bulk material that realized in increasing flexural strength. Thixocasting is introduced for controlling constituent's surface shape and size.

3. RESULTS AND DISCUSSION

Figure 1 presents optical microscopy of a sample (a) DIMOX at 1050^{o}C for 10 minute without addition of alloying elements, (b) DIMOX at 1050^{o}C with 10 wt. % mix addition at 10 minute, and (c) DIMOX at 1050^{o}C without addition at 60 minute. DIMOX has a significant effect of producing in-situ uniformly distributed aluminum metal matrix composite with its clear constituents at 10x magnification (Figure 1 a). Boron has a significant effect of increase the constituent's intensity, shape and size at 10 minute (Figure 1b). Time effect has a significant effect of increase the constituent's intensity, shape and size at 60 minute (Figure 1c).

Optical Microscopy of a samples DIMOX, with 10 wt. % mix addition presented at Figure 2, at 1050^{o}C for 10 minute and (a) Rheocasting at 800^{o}C for 10 minute, (b) Thixocasting at 800^{o}C for 10 minute, and (c) Thixocasting at 800^{o}C for 10 minute. The effect of Boron addition, along with semisolid reaction, reveals that rheocasting has a great significant effect of more control of constituents shape, size, and intensity with dominant uniformity at 10 minute holding time if compared to 60 minute holding time of DIMOX sample (Figure 1c). In addition, Rheocasting has a significant effect of controlling constituents shape and size in a bulk hybrid composite. On the contrary, Thixocasting has only a dominant effect at the sample surface (Figure 2C) compare to Figure 2a, and Figure 2b.

Scanning Electron Microscopy, SEM, of a samples DIMOX at 1050^{o}C for 10 minute is presented at Figure 3 (a) polished and etched surface without addition, (b) fracture surface without addition and (c) polished and etched surface with Rheocasting at 10 minute with 10 wt. % mix addition. Figure 3a reveals that DIMOX induce grain refinement (grain size ~ 100 μm), with dispersed phase of α-alumina along grain boundaries which is more dominant at fracture surface (Figure 3b). Figure 3c reveals the effect of rheocasting, a semi-solid reaction, after DIMOX with the addition of 10 wt. % mix (Borax+ Boric acid). More grain refinement along with more intermetallic fibrous and equiaxid whiskers established (grain size ~ 20 μm), and correlates with Figure 2a. This induce a new hybrid structure with different constituents, fibrous ceramic alumina, fibrous intermetallic along with equiaxid whiskers reinforce residual aluminum matrix with great wettability between composite ingredients.



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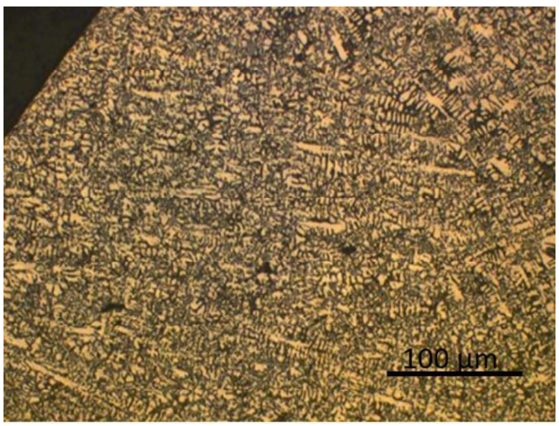

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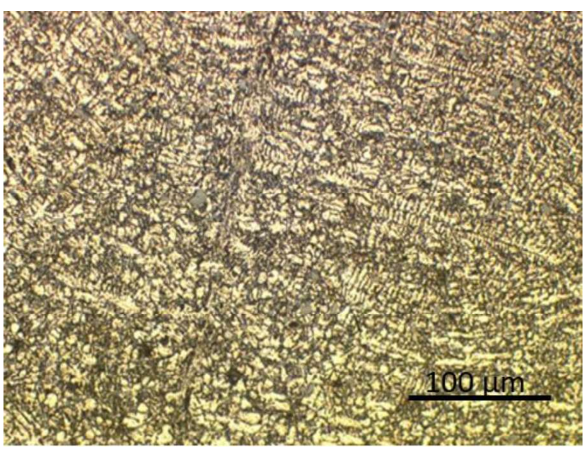
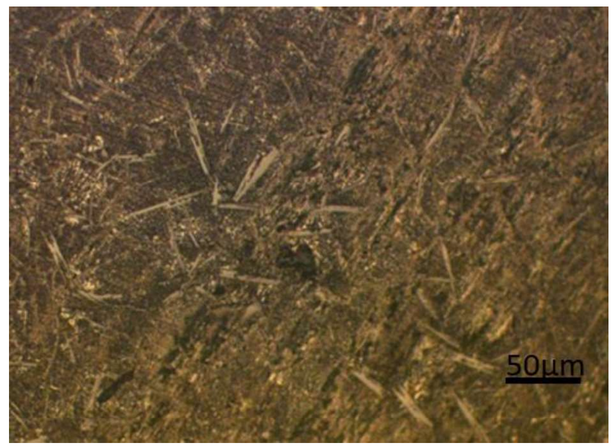


(c)

Figure 1. Optical Microscopy of a sample DIMOX at 1050^{o}C (a) without addition at 10 minute, (b) with 10 wt. % mix addition at 10 minute, and (c) without addition at 60 minute.

(a)



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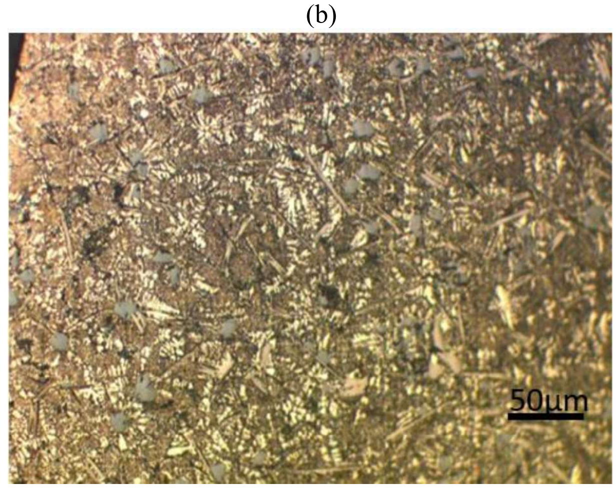
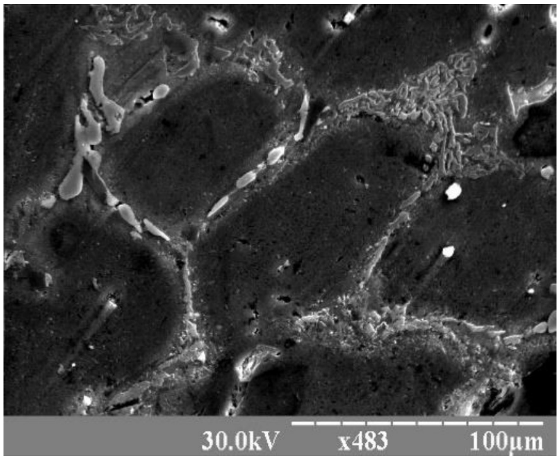


(c)

Figure 2. Optical Microscopy of a sample DIMOX, with 10 wt. % mix addition, at 1050^{o}C for 10 minute and (a) Rheocasting at 800^{o}C for 10 minute (at sample bulk center and surface), (b) Thixocasting at 800^{o}C for 10 minute (at sample center), and (c) Thixocasting at 800^{o}C for 10 minute (at sample surface).



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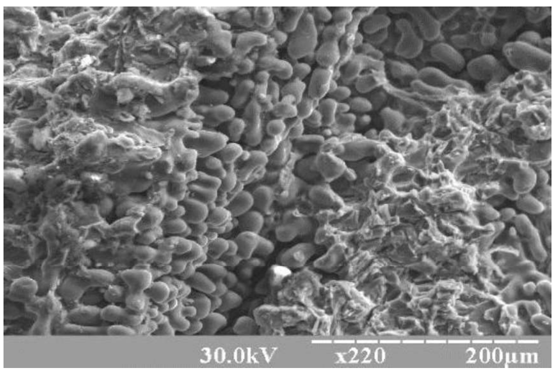

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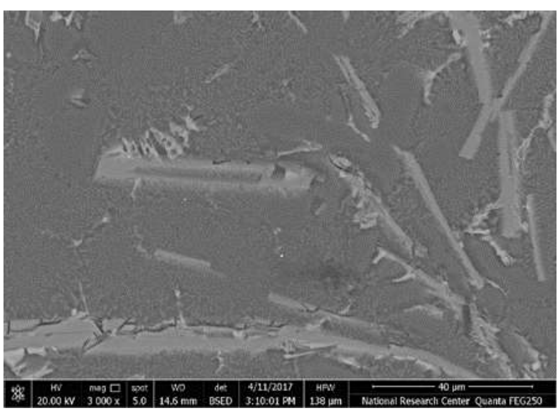


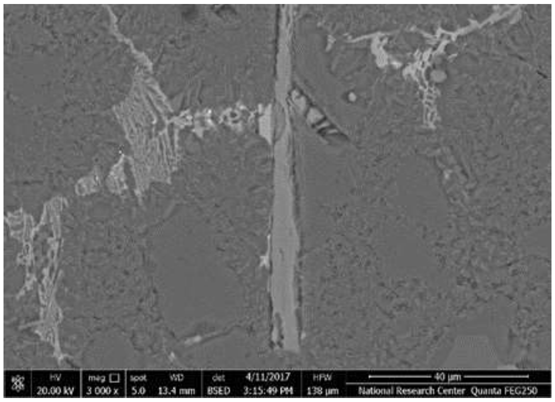
(c)

Figure 3. SEM of a sample DIMOX at 1050oC for 10 minute at (a) polished and etched surface without addition,

(b) Fracture surface without addition and (c) polished and etched surface with Rheocasting at 10 minute with 10 wt. % mixture addition.

Thixocasting is established in another group of samples and presented in Figure 4. Scanning electron microscopy of a sample DIMOX at 1050ºC for 10 minute and Thixocasted at 800ºC for 10 minute is established at (a) the sample center, (b) sample edge, and at (c) fracture surface. Great correlation with Rheocasting after DIMOX induced in less grain refinements (grain size ~ 30-40 μm), along with different composite constituents. Segregation of alloying elements is more dominant at fracture surface delocalized intermetallic phase (Figure 4c). On the contrary to rheocasting, thixocasting reveals less grain size refinements in a bulk composite with new coating morphology.

Fracture surface of the samples DIMOX at 1050ºC for 10 minutes without addition of alloying elements is scanned in SEM, and presented in Figure 5. Figure 5a presents SEM of fracture surface in samples, without addition, that reveals different phase's morphology with cleavage fracture and ductile dimples fracture. Energy dispersive X-Ray spectroscopy is established at two zone of interest A, and B that induce clear alloy segregation (Table 1). Point A has Wt. % of 0.27 O, 0.7 Mg, 23.87 Si and 75.15 residual Al. Point B has wt. % of 0.62 Mg, 8.08 Si, 3.13 Fe, 5.66 Ni, 2.82, and 79.69 residual Al. Figure 5b reveals the effect of rheocasting after DIMOX that induce alloy segregation along with delocalized phase of interests (intermetallic fibrous phase and equiaxid phase). Cleavage fracture of intermetallic reinforcement phase along with ductile dimpled fracture of residual aluminum matrix. Rheocasting induce grain refinement (Figure 5b) if compared to Thixocasting effect (Figure 5c) that induce less reinforcement and clear wettability correspondence.



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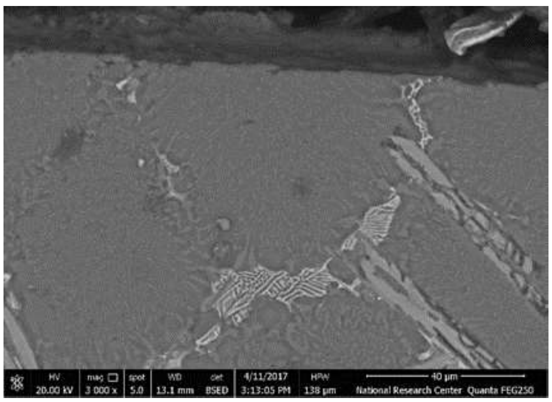

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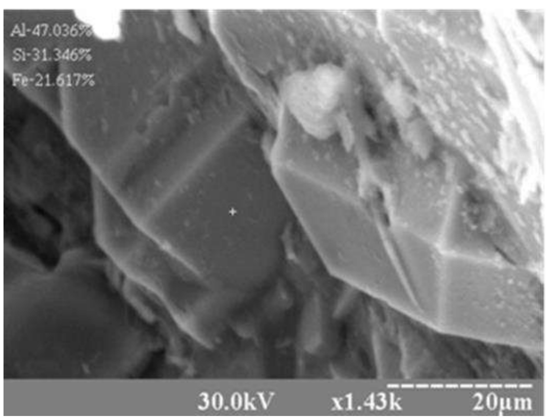

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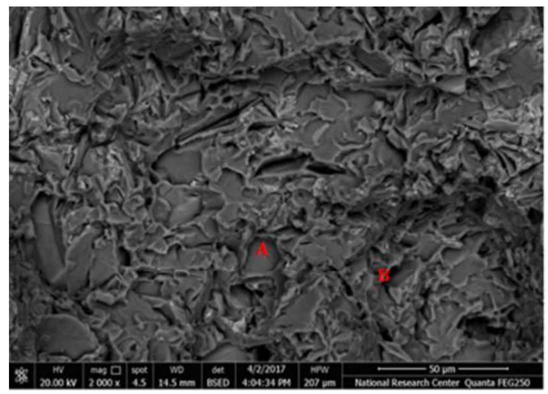
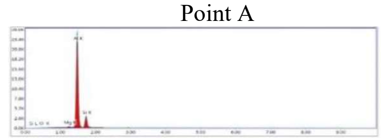
Figure 4. SEM of a sample DIMOX at 1050ºC for 10 minute and Thixocasted at 800ºC for 10 minute at (a) the sample center, (b) sample edge, and (c) fracture surface.

Table 1; EDS reveals alloy segregation of DIMOX sample Figure 5a without B addition.

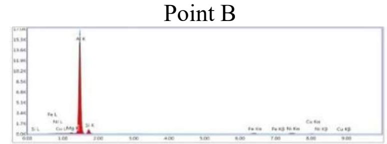
Element Wt. %	Point A	Point B
O	0.27	-
Mg	0.7	0.62
Al	75.15	79.69
Si	23.87	8.08
Fe		3.13
Ni		5.66
Cu		2.82



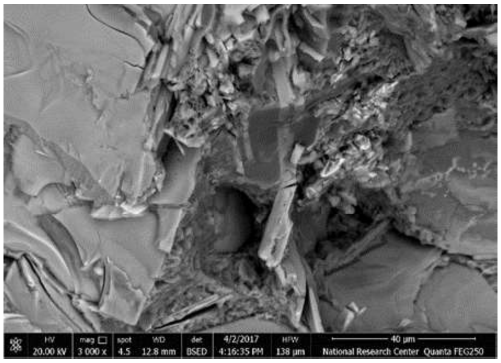
Point A



Point B



(a)



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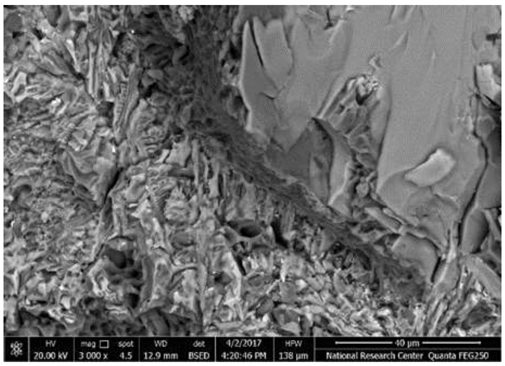


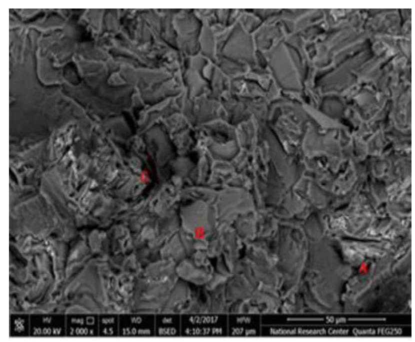
(c)

Figure 5. SEM of fracture surface in samples, without addition, (a) DIMOX at 1050ºC with EDS, (b) DIMOX at 1050ºC for 10 minute and Rheocasted at 800ºC for 10 minute and (c) DIMOX at 1050ºC for 10 minute and Thixocasted at 800ºC for 10 minute.

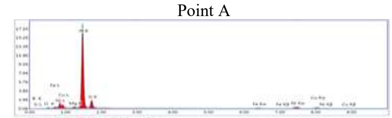
The effect of liquid state, DIMOX, and the semisolid state processing, Rheocasting and Thixocasting, with Boron addition is established in Figure 6a-c and Table 2-4 respectively. Figure 6a presents SEM of a sample DIMOX at 1050ºC for 10 min with the addition element boron via 10% mixture of (Borax + Boric Acid). It reveals the segregation of alloying elements via energy dispersive X-ray spectroscopy, EDS at different points. Delocalized zones of interest with its different fracture morphology reveals the de-localized intermetallic phases in fibrous and/or equiaxid whiskers with its segregated alloying elements (Table 2). Boron has a great effect of accelerate the possibility of inoculation as well as the segregation of alloying elements. Borosilicate along with ceramic aluminum oxide (alumina) dominated at point A, Silicon enriched intermetallic phase dominated at point B (correlated with Fig. 4C), and point C reveals borate glass with residual aluminum.

Figure 6b presents fracture surface with different delocalized phases with different color and morphology that analyzed in Table 3 reveals the effect of Rheocasting with B-addition. At point A, represent dark zone with borosilicate glass, 2.68wt. % B, enriched with 13.96 wt. % C with alumina. At Point B, dark gray, delocalized with alumina and Si enriched zone 18.88 wt. % Si. Point C, gray zone, delocalized with alumina with Fe-enriched, 14.57 wt. % Fe, and Mg enriched, 0.99 wt. % Mg. Point D, Light gray, is enriched with alumina with Fe-enriched (19.74 wt. % Fe), and As-enriched (11.31 Wt. % As).

Figure 6c presents fracture surface with different delocalized phases with different color and morphology that analyzed in Table 4 reveals the effect of Thixocasting with B-addition. At point A, represent light zone with less borosilicate glass, 0.27 wt. % B, enriched with 20.4 wt. % Fe with 0.25 Co. At Point B, dark gray, delocalized with 29.53 wt. % Si, with 69.24 wt. % Al. Point C, more dark gray zone, delocalized with alumina with Si-enriched (17.04 Wt. % Si), Fe-enriched, 10.0 wt. % Fe, and Cu enriched, 10.01 wt. % Cu. Point D, Light gray, is enriched with alumina with Si-enriched (27.55 Wt. % Si), more Fe-enriched (22.4wt. % Fe). Alumina is more dominant in Rheocasting with more enriched Boron and carbon elements in bulk matrix composite and its constituents.

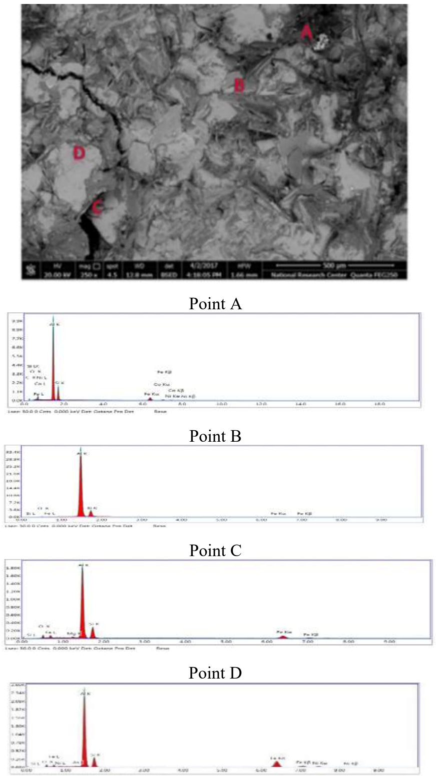


Point A



Point B

(a)



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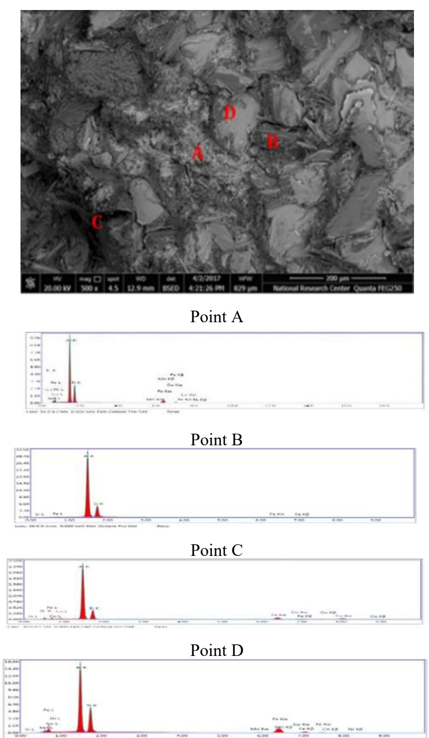

(c)

Figure 6. SEM with EDS of fracture surface of samples (a) DIMOX at 1050°C for 10 minute with 10 wt. % addition of a mix. Of (BA+BX). (b) DIMOX + Rheocasting with addition, and (c) DIMOX + Thixocasting with addition.

Table 2; EDS reveals alloy segregation of DIMOX sample Figure 6a with B addition.

Element Wt. %	Point A	Point B	Point C
B	2.87	-	1.51
O	1.93	-	-
Mg	1.09	-	-
Al	65.11	15.04	54.59
Si	12.45	82.94	42.64
Fe	2.15	-	-
Ni	8.4	-	-
Cu	5.99	-	1.26
Sn		2.02	-

Table 3; EDS reveals alloy segregation of DIMOX+ Rheocasting sample Figure 6b with B addition.

Element Wt. %	Point A	Point B	Point C	Point D
B	2.68	-	-	-
C	13.96		-	-
O	1.47	1.83	7.53	3.89
Mg	-	-	0.99	-
Al	52.13	78.01	59.67	52.21
Si	16.06	18.88	17.23	10.17
Fe	12.01	1.27	14.58	19.74
Ni	1.38	-	-	2.68
Co	0.31	-	-	-
As				11.31

Table 4; EDS reveals alloy segregation of DIMOX+ Thixocasting sample Figure 6c with B addition.

Element Wt. %	Point A	Point B	Point C	Point D
B	0.27	-	-	-
O	-	-	3.95	-
Al	53.51	69.24	57.1	46.9
Si	24.21	29.53	17.04	27.55
Fe	20.4	1.23	10.0	22.4
Ni	1.04	-	-	1.69
Cu	-	-	10.01	-
Co	0.25	-	1.9	0.7
As		-	-	-
Mn	0.32	-	-	0.76

Mechanical characterization is also established via tensile testing of the different group samples and 3-point bending test. Tensile strength with modulus of rupture, MOR, via 3-point bending tests are gathered and presented in table 5. Tensile tests reveals the importance of in-situ DIMOX + Rheocasting or Thixocasting that yields tensile strength of 167.25 MPa (DIMOX + Thixocasting) compared with tensile strength of 116.15 MPa for As- Received sample. MOR is more dominant for the new emerged Al-hybrid materials reveals the effect of semi-solid reaction with boron addition that yield MOR = 371.84 MPa for DIMOX + Thixocasting samples compared with 118.86 MPa for As-Received samples. The application of novel technique, DIMOX, is dominant in-situ hybrid composite processing. The addition of Boron is dominant in formation of borate glass with its magnificent effect on mechanical properties.

Table 5; Mechanical characterization of samples, tensile testing and 3-point testing.

Test Samples	Tensile strength [MPa]	Flexural strength MOR [MPa]
As Received Material at 750°C `	116.15	118.86
Dimox at 1050°C for 10 min without the addition of alloying elements	126.71	234.20
Dimox at 1050°C for 60 min without the addition of alloying elements	115.62	313.86
Dimox at 1050°C for 10 min with the addition of 10% wt. (50% Boron +50% Boric Acid)	176.90	356.87
Dimox at 1050◦C with the addition of(boron+ boric acid) + Rheocasting at 800°C	156.85	369.14
Dimox at 1050◦C with the addition of (boron+ Boric Acid) + Thixocasting at 800°C	167.25	371.84

4. CONCLUSIONS

The kinetic of DIMOX along with alloying elements effects, semisolid rheocasting interaction is also introduced. Alloy segregation along with the control of micro-constituents shape and size is introduced via fibrous intermetallic reinforced ceramic residual aluminum, and the formation of ceramic alumina phase which is more dominant in DIMOX + Rheocasting. The effect of boron addition via 10 Wt. % mixture addition of equal weights borax and boric acid has a significantly effect of accelerate the formation of hybrid composite along with more segregation kinetic of alloying elements delocalization in a semi-solid reaction Rheocasting. The application of in-situ liquid state processing, DIMOX, along with semi-solid processing, Rheocasting and/or Thixocasting is more dominant in hybrid composite processing. Boron has a magnificent effect on ultra-fast processing along with control micro-constituent of composite in a uniformly distributed manner via alloy segregation and the formation of borate glass, borosilicate and aluminum borate. Mechanical properties is enhanced and compared to as received. The control of micro-constituents shape, size and location is controlled via semi-solid rheocasting as bulk matrix control with more oxides. The control of surface is more dominant in Thixocasting processing

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