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# Microstructural characterization of squeeze-cast Al–8Fe–1.4V–8Si

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**Abstract**

This paper investigates the effect of vanadium on the composition and morphology of intermetallics formed during the squeeze casting of Mg-modified Al–8Fe–1.4V–8Si alloy in both monolithic form and as-reinforced with 7.58, 10.52 and 15.68 wt.% SiC particles ($SiC_p$). Iron intermetallics of α-$Al_7(Fe,V)_3Si$ and β-$Al_{18}Fe_{11}Si$ phases were predominantly observed in the alloy and composite. SEM studies and the EDX analyses revealed that refinement of Fe-intermetallics and modification of β-phases to less deleterious morphologies of α-phases has been achieved by vanadium addition of 1.4%. Also, heat treatment enhances V diffusion and SiC particles act as nucleation sites for the formation of finer α-intermetallics. Fractographs exhibited cracking of long β-phases and partial decohesion of $SiC_p$ from the matrix.


*Keywords:* Al–Fe–V–Si alloy; Squeeze casting; Intermetallic phases; Vanadium; SiC particles

## 1. Introduction

In recent years, there have been considerable efforts in the aerospace community to develop high-temperature aluminum alloys capable of competing with titanium alloys. The technological development in both rapid solidification and powder metallurgy over the past decade has led to several candidate materials. Al–Fe–V–Si is a new series of alloys which have the potential for light-weight and high-temperature applications [1–8]. Ceramic particulate reinforcement such as SiC can be a powerful tool for developing this alloy with enhanced strength and stiffness, wear resistance, stability of properties at elevated temperature and reduced density [2,9–12]. Moreover, several attempts have been made to enable conventional casting processes to produce these alloys successfully with optimized mechanical properties. Refining the size and morphology of Fe-containing compounds is one of the most important achievements in this respect [13].

Basically, the as-cast microstructure of the Al–Fe–V–Si alloy system consists of a primary phase, brittle intermetallic silicide compounds, and other eutectic mixtures. It is a well-known fact that the brittle intermetallic compounds have detrimental effects on the mechanical properties of alloys. In the alloy system studied, the β-$Al_{18}Fe_{11}Si$ phase is the most undesirable one, due to its needle shape, which is expected to raise the stress concentration and to result in a lower ductility of the alloy [14,15]. The secondary phases in Al alloys can have significant effects on material properties including strength, toughness, formability and re-crystallization even when their content is less than 5 vol.% [16,17]. Sustained efforts continue in order to both improve the properties of such alloys through the modification of the as-cast structure and also to better comprehend the effects of different elements, such as Mg, Ca, Sr, K, Li and Mn, on the microstructure and morphology of intermetallics [13,14,18,19]. Addition of these elements to Al alloy can reduce the size and amount of platelet β-phase and modify them to Chinese script α-compound with the less deleterious morphology [13,19,20]. The properties of cast Al–Fe–V–Si alloys are determined by the fineness of their microstructures and the distribution of their phases [21]. In this paper, an attempt has been made to understand the role of vanadium in the phase refinement of the Al–8Fe–1.4V–8Si alloy and its $SiC_p$ reinforced composite.

## 2. Experimental procedures

The alloys were prepared by melting Al–Si and Fe–V master alloys in an induction furnace. They were modified by adding nearly 1.5 wt.% Mg. After melting, sufficient time was given for the homogenization of the melt. The molten alloy was degassed

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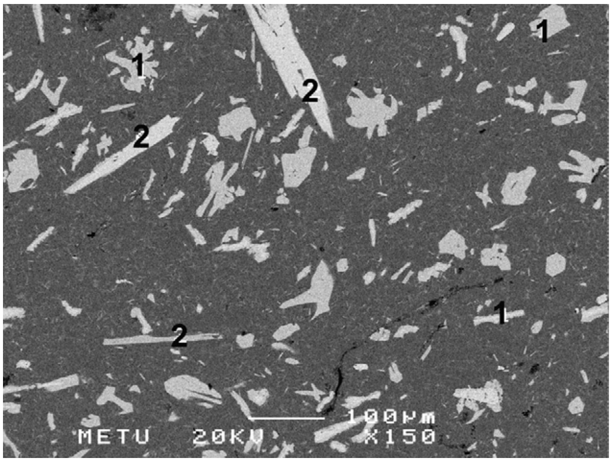


Fig. 1. SEM of the squeeze-cast sample showing phases α (labeled as 1) and β (labeled as 2).

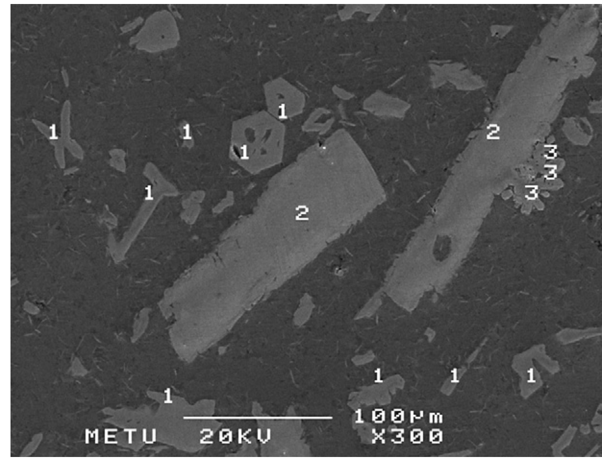


Fig. 2. SEM of the squeeze-cast Al–8Fe–1.4V–8Si sample showing the existence of V-rich phases (labeled as 3) around the needle-like β-phase (labeled as 2).

with the help of hexafloroethane. Molten aluminum alloys were poured into a mold specifically designed for vertically filling the squeeze-casting machine. Bending and tensile test specimens were produced simultaneously in this mold. During squeeze casting, the applied pressure was 80 MPa. The nominal chemical composition (wt.%) of the alloy used in this study is approximately Al–8Fe–1.4V–8Si. The composite of this alloy has been produced by adding particles of SiC as reinforcements with a mean diameter of 29.2 ± 1.5 μm. The composites produced contain 7.58, 10.52 and 15.68 wt.% SiC particles. The alloy and its composites were solutionized at 540 °C for 1 h followed by water quenching and artificial ageing at 190 °C up to 6 h. Scanning electron microscopy (SEM) observations and energy dispersive analysis (EDX) were performed on specimens to investigate the morphology and the chemical composition of the intermetallics. In order to study the effect of V on the mechanical behavior of the composites, they were tested by three-point bending and fracture surfaces were examined by SEM.

## 3. Results and discussion

### 3.1. Microstructures

Fig. 1 shows the microstructure of the squeeze-cast Al–8Fe–1.4V–8Si alloy with the presence of mainly two different Fe-intermetallics; small α-$Al_7(Fe,V)_3Si$ and large β-$Al_{18}Fe_{11}Si$ phases, indicated as 1 and 2, respectively.

The α-phase has two distinct morphologies, "Chinese script" and polygonal form (1 in Figs. 1 and 2), with the composition of $Al_7(Fe,V)_3Si$. The SEM analyses of both the polygonal and the Chinese script phases revealed the presence of aluminum, iron, vanadium and silicon elements with no significant difference in composition between the two morphologies. The plate- or needle-like phases (2 in Figs. 1 and 2) are β-intermetallics with the composition of $Al_{18}Fe_{11}Si$. The longest of them reaches about 250 μm in length. These phases are very hard and brittle with relatively low-bond strength with the matrix. In particular, the needle-like morphology of the β-phase does not favor high ductility and may act as a stress-raiser, thus being detrimental to mechanical properties of the alloy [22,23].

### 3.2. Vanadium effect on microstructure

In Al–Fe–V–Si alloys, compared to Mn, Cr and Mo, vanadium was found to be the most promising element in stabilizing the metastable $Al_{12}Fe_3Si$ phase and increasing the mechanical properties at elevated temperatures, by substituting vanadium for iron [24,25]. Beside this important role of V in Al–Fe–V–Si alloy, in the present study it was also observed that the finer intermetallics contain higher amounts of V compared to coarse intermetallics. Fig. 2 shows three different intermetallics; phases labeled as 1 and 3 with higher V content, have smaller sizes compared to phase labeled as 2. The composition of phase 3 with the highest V amount (about 38.11%) as obtained by the EDX analyses (Fig. 3) is $Al(Fe,V)_4Si_3$ and it is located near to β-$Al_{18}Fe_{11}Si$ phase (phase 2 in Fig. 2). It was observed that as amount of V increases in intermetallics, the amount of Fe decreases and Si increases (Table 1). These observations lead to the conclusion that addition of V promotes refinement of Fe-intermetallics by changing the morphology of large β-platelets to the finer ones by replacing Fe by V.

Allen et al. [16] observed that in 1xxx Al alloys, the formation of $FeAl_m$ containing higher Si than $Fe_4Al_{13}$ have been promoted by raising the level of Si content of the interdendritic liquid during solidification. On the other hand, they argued that it was yet not absolutely clear if V, Zr and grain refiner additions promote formation of intermetallics with higher Si content like $FeAl_m$

Table 1
Microanalysis of phases in the squeeze-cast Al–8Fe–1.4V–8Si alloy

| Phase marked | Figure | Compositional analysis (wt.%) | | | |
|---|---|---|---|---|---|
| | | Al | Fe | V | Si |
| 1 | 1, 2 and 6 | 62.33 | 25.88 | 2.91 | 8.88 |
| 2 | 1, 2 and 6 | 60.55 | 36.24 | 0.00 | 3.21 |
| 3 | 2 | 11.33 | 9.20 | 38.11 | 41.36 |

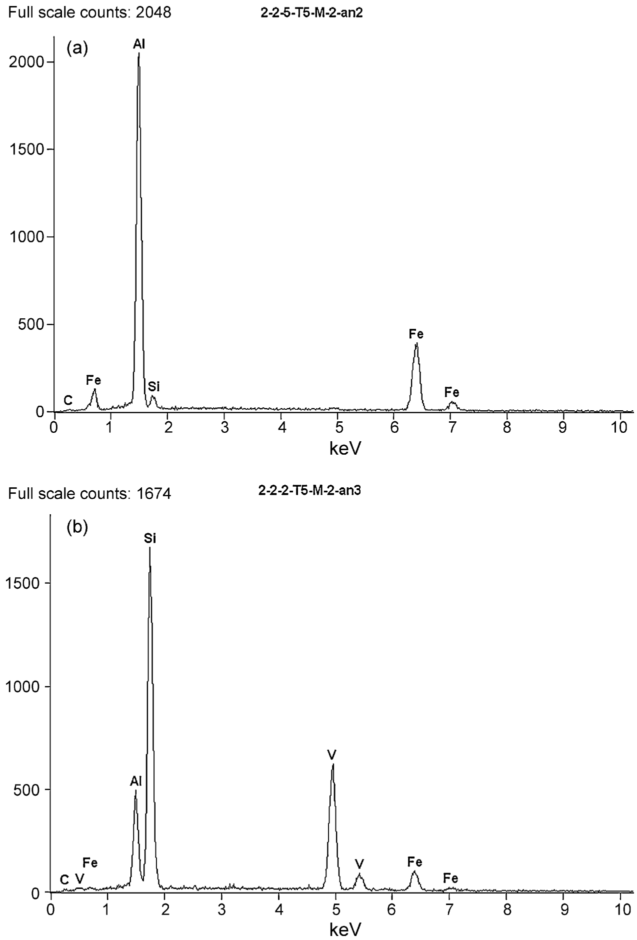


Fig. 3. EDX analysis of the (a) β-phase and (b) V-rich phase 3 forming around it.

by locally raising the Si concentration. They suggested that in commercial direct chill cast alloys, V present either as impurity or grain refiner would play an important role in phase selection during casting processes besides the effects of solidification rate and Si content.

In the present study, EDX analysis showed that $Al(Fe,V)_4Si_3$ intermetallic (phase 3) is richer in Si than β-$Al_{18}Fe_{11}Si$ phase as the amount of V increased to 38.11% in phase 3 (Table 1). Therefore, it seems most likely that increasing the amount of V raises Si locally. On the contrary, it was observed that the amount of Fe in the β-phase has decreased from 36.24 to 9.2 wt.% in phase 3.

Table 2
Microanalysis of β-phase in Fig. 4

| Region marked | Compositional analysis (wt.%) | | | |
|---|---|---|---|---|
| | Al | Fe | V | Si |
| A | 61.32 | 23.96 | 4.61 | 10.11 |
| B | 59.95 | 35.30 | 1.55 | 3.21 |

Si and V-rich intermetallics (phase 3) were mostly found around β-phase (Fig. 2). Allen et al. found that V is partitioning into the Al matrix during solidification away from the Fe aluminides as expected from the nature of the Al–V binary phase diagram [16]. Heat treatment such as solutionizing and ageing can help V to diffuse from matrix to poor V regions (β-phases), thereby locally increasing the amount of Si, and subsequently promoting the formation of small intermetallics with the higher amount of V and Si (phase 3) around β-phase. Samuel et al. [26] reported that in Sr-modified cast 6xxx type aluminum alloys; the formation temperature of β-phase is high enough to enhance the diffusion of Sr into the β-platelets. Similarly, it was observed that V diffused to β-phase and finally caused the formation of phase 3 attached to the β-phase platelets (Fig. 2). Since V diffuses slowly in Al [27], heat treatment provides sufficient time and temperature for slow diffusion of V from matrix to Fe-intermetallics causing V concentration gradient along the β-phase in Al–Fe–V–Si/$SiC_p$ (Fig. 4).

The platelet β-phase has a complex, interconnected network shape, and it appears to grow around a dendrite arm [15]. The EDX analyses revealed that the amounts of V and Si were higher in the region near the interface between the matrix and the β-phase, compared to the middle of the dendrite arm. This difference can be seen by a change in color in Fig. 4. The brighter region in the middle of the β-phase is relatively poorer in V and Si but richer in iron as found in EDX analysis (Fig. 5). Compared to “B”, in region “A” the amount of iron has decreased from approximately 35 to 24 wt.%. On the other hand, the amount of Si and V increased from 3.21 and 1.55 wt.% to 10.11 and 4.61 wt.%, respectively (Table 2). Region “A” has a composition similar to α-phase around SiC particle (Fig. 4).

### 3.3. SiC particles effect on microstructure

Ashtari et al. [13] explained the refinement of β-phase based on its nucleation behavior. They studied the influence of K addi-

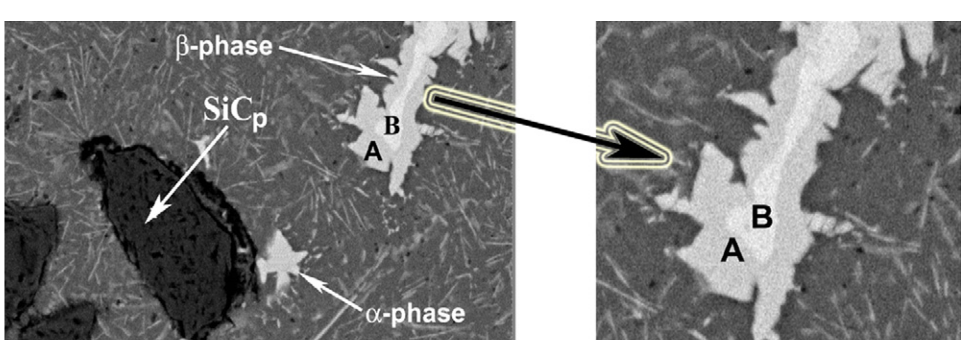


Fig. 4. SEM image of the composite (regions with different Fe, V and Si in the β-phase can be seen in the enlarged view).

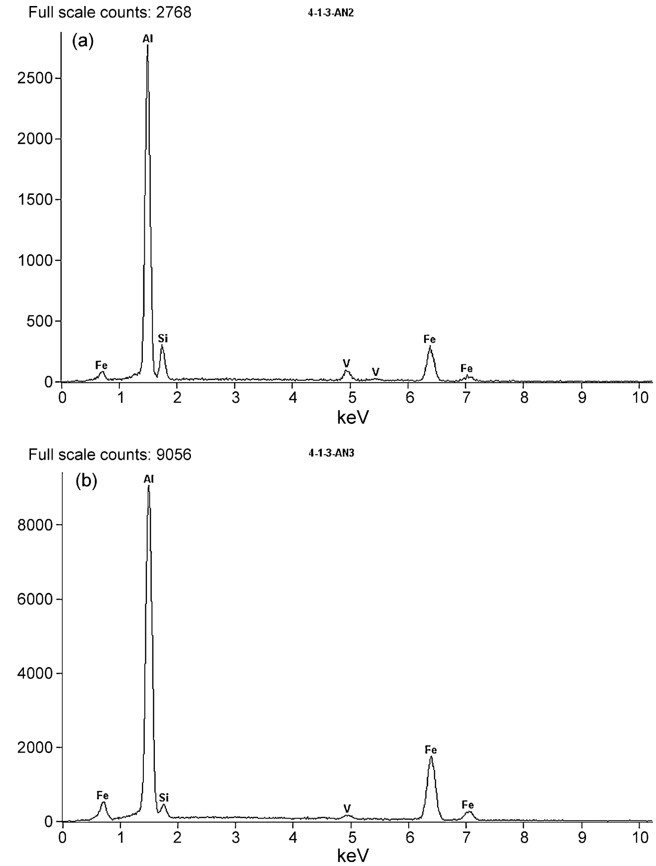


Fig. 5. EDX analysis of (a) "A" and (b) "B" regions of the β-phase showing the gradient of iron, vanadium and Si content.

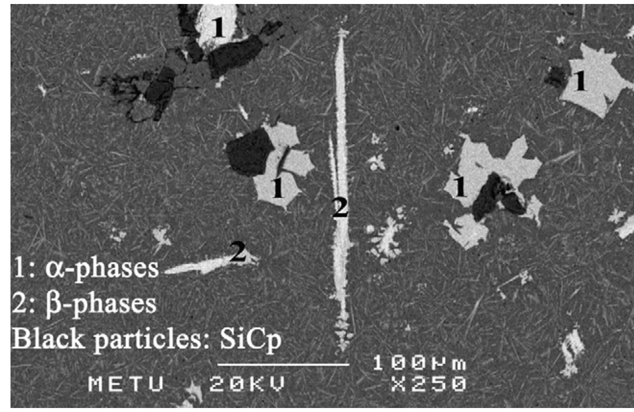


Fig. 6. SEM of the squeeze-cast sample showing α-phases mostly located around $SiC_p$.

tion on the Fe-containing intermetallic compounds in Fe-rich AA319 aluminum alloys and observed that the β-phase crystallization took place after α-phase by K addition. They explained the effect of K by increasing both the liquidus and crystallization temperature of β-phase. In other words, the β-phase crystallization took place under lower undercoolings in the K-containing alloys. The decreased undercooling was considered to be the result of inducing additional nucleation sites such as potassium oxides. The very fine oxides were considered to be the nucleation sites for the α-compounds, which were responsible for the refinement of the Fe compounds in the graphite mold.

However, in our study there was no clear evidence for the effect of V addition on the nucleation sequence of α- and β-phases. Results showed α-phases mostly nucleated and grew around SiC particles indicating that they act as nucleation sites for α-phases (Fig. 6). This heterogeneous nucleation can be explained as the result of enrichment of Si in the melt around the particles based on the thermal lag model [28]. According to this model, SiC particles have a lower thermal conductivity and heat diffusivity than those of aluminum melt. Thus, SiCp are not able to cool down as fast as the melt after casting. As a result, the temperature of the particles is somewhat higher than the liquid alloy. The hotter particles may heat up the liquid in their immediate surroundings, and thus delay solidification of the surrounding liquid alloy. As it was observed, nucleation of β-phase started in the liquid alloy at a distance away from the particles, where the temperature is lower. Since β-phases are poor in Si and V content, growth of their nuclei will lead to enrichment of Si and V in the remaining melt around SiCp which is adequate condition for formation of α-phases. Another effect of thermal lag is that the melt around the particles will solidify in the last stages and fine phases will form. Introduction of SiC particles in turn promotes the formation of small α-phases rather than large β-phase platelets.

### *3.4. Mechanism of the change of the crystallized Fe-intermetallics*

In the case of higher cooling rate induced by metallic mold casting, in the similar manner that Ashtari et al. [13] observed, the addition of V may allow the α-phase to nucleate in a non-equilibrium state, while the β-phase (equilibrium phase) also nucleates. Because of the monoclinic crystal structure of β-phase, slow two-dimensional plate-like growth is expected. Unlike β-phase, α-phase with hexagonal crystal structure implies the fast growth of three-dimensional Chinese script of this phase in any direction [13]. The α-phase which grows quickly on the interconnected network branch of dendrite arm of β-phase (Fig. 4), consumes iron. Decreased Fe in the liquid is not adequate for further growth of β-phase and it cannot grow anymore. EDX analysis showed that the amount of iron present in β-phase is about 35 wt.% whereas it is about 24 wt.% in α-phase (Table 2). By this way, the α-phase becomes the dominant intermetallic. Therefore, addition of V modifies the size and amount of β-phase by preventing its further growth and promoting the growth of α-phase preferably.

As it can be concluded, V addition is playing a significant role on the formation of V and Si-rich intermetallics of $Al(Fe,V)_4Si_3$ (phase 3) and α-$Al_7(Fe,V)_3Si$ phase. The results reveal that the latter forms either near to SiC particles or in the outer shell of β-phase where V raises the amount of Si locally. It implies that in regions with high Si content (i.e. near SiC particles), V is not the only prerequisite for α-phase formation. However, it refines the morphology of β-phase platelets to α-phase by locally raising Si content from 3.21 to about 10.11 wt.% (Table 2). In conclusion,

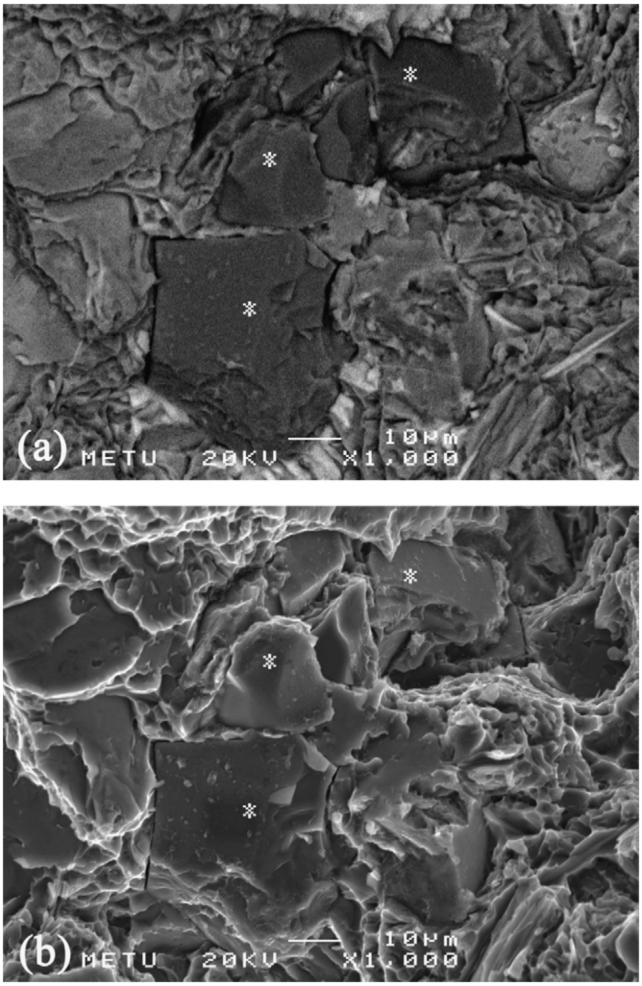


Fig. 7. (a) Back-scatter electron image of Al–Fe–V–Si/$SiC_p$ composite showing decohesion of SiC particles and (b) SEM showing small dimples around $SiC_p$.

control of V addition and SiC content in this alloy can provide a powerful means to influence secondary-phase content, and ultimately material properties.

### *3.5. Fractography*

Since the mechanical properties of cast Al–Fe–Si alloys are mostly affected by intermetallics, several attempts have been made to improve them by refining the size and morphology of Fe-containing compounds in this respect. Tash et al. [29] reported that high Mn/Fe levels in heat-treated 356 and 319 aluminum alloys promote the formation of the α-Fe scripts rather than the β-phase platelets and improve the alloys machinability and decrease the drilling force. Three fracture modes have been usually observed in metal matrix composites: nucleation and growth of voids in the matrix, particle cracking and debonding between a particle and the matrix [30–34]. With an increase in reinforcement content in the composite, fracture has been reported to dominate by cracking of particles. Broken SiC particles in composites have not been detected since their amount was not so high, but partial decohesion of SiC particles from matrix was observed (Fig. 7).

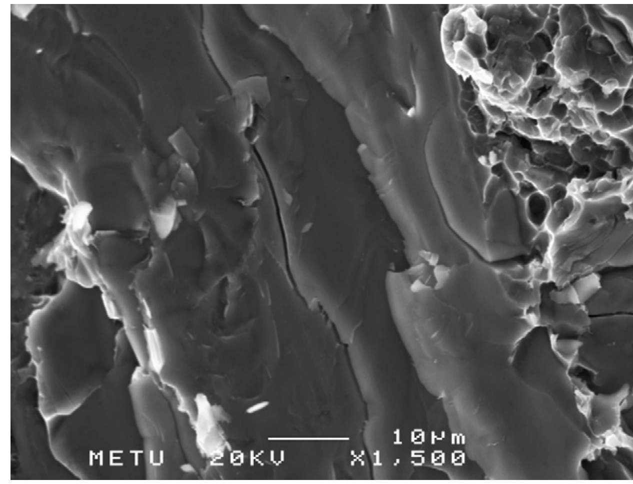


Fig. 8. Scanning electron micrograph of Al–Fe–V–Si/$SiC_p$ composite showing cracking of the β-phase.

The small dimples have been usually observed on the fracture surface of metal matrix composites when decohesion is dominant [34]. The fractograph of the system studied showing small dimples around SiC particles and β-phase platelets visualized this fact (Figs. 7b and 8). β-Intermetallics existing in Al–Fe–V–Si system are hard and brittle and may easily fracture (Fig. 8). Therefore, fracture characteristics of the alloys are dictated by their behavior.

In this study, large β-phases showed long cracks (Fig. 8), whereas no crack has been observed along α-phases. As a result, long β-phases are more harmful than α-phases for mechanical property of this alloy. On the other hand, as smaller phases are known to provide much less hindrance to feeding during casting, the massively large β-phase in Al–Fe–Si systems is expected to decrease the effective feeding during casting process, resulting in micro-pores that deteriorate the mechanical properties [22]. The maximum strength of composite at fracture is absolutely sensitive to the presence of micro-pores and porosity inherently present in the as-cast samples. Addition of V causes phase refinement of intermetallics and modifies β-phases to the less deleterious morphologies of α-compounds and enhances their formation. Thus, the mechanical properties of the studied alloy can be improved by V addition, in association with heat treatment and SiCp as a nucleation sites for finer intermetallics.

## 4. Conclusions

1. Mostly coarse intermetallic phases have been observed to form in the squeeze-cast Al–8Fe–1.4V–8Si alloy. The α-$Al_7(Fe,V)_3Si$ and β-$Al_{18}Fe_{11}Si$ phases were the major intermetallics found in the system studied.
2. The α-phases showed two distinct morphologies; namely, the “Chinese script” and the polygonal shape. β-Phases were the most undesirable phases due to their large needle shapes.
3. Vanadium addition refined the morphology of β-phase platelets to α-phase by locally raising Si.

4. SiC particles acted as nucleation sites for finer α-intermetallics.
5. Decohesion of $SiC_p$ and cracking of long β-platelets are the most important factors to control the fracture behavior of the cast composite of Al–Fe–V–Si alloys reinforced with SiC particles.

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