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Tensile fracture behavior of spray-deposited SiC_P/Al–Fe–V–Si composite sheet

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SiC_P/Al–Fe–V–Si (Al–Fe–V–Si reinforced with SiC particles) composite prepared by spray deposition was densified by hot pressing and then rolled into sheets. Microstructure of the composite was observed, and fracture properties and fractographies of the composite at different tensile temperatures were investigated. The results show that uniform distribution of SiC particles and strong bonding between the particles and the matrix are obtained by rolling after hot pressing. It is found that fracture properties and fractographies of the composite are affected by the distribution and orientation of SiC particles. The composite is characterized by the fractographies of the composite varying with the elevation of temperature. Cracking of SiC particles is the dominant rupture mode because of the strong interface bonding with the tensile temperature below 300 °C. Debonding at SiC/Al matrix interfaces becomes the dominant rupture mode with tensile temperature above 300 °C, particles breakage reduces sharply as the tensile temperature is elevated, while tensile strength and elongation of the composite decreases rapidly as the tensile temperature increases.

Keywords: multi-layer spray deposition; fracture behavior; SiC reinforcement; microstructure; SiC_P/Al–Fe–V–Si composite

1. Introduction

Al–Fe–V–Si system heat-resistant aluminum alloy has attracted considerable attention during the past few years with excellent characteristics, such as low density, high specific strength, stiffness, thermal stability, and suitable for applications at high-temperature environments. It was developed by Skinner by plate fluid cast technology,[1] and has been believed to be a substitute for titanium alloy for applications at an elevated temperature. Considerable research efforts [2–4] have been expended in the development of rapid solidified-Al–Fe–V–Si system heat-resistant aluminum alloy. The Al–Fe–V–Si alloys have been believed to be promising candidates for high-temperature application, especially those reinforced with ceramic particulates (e.g. SiC particulates), short fibers, or whiskers that are more attractive.[5–9]

A combination of multilayer spray deposition technology and co-deposition of ceramic particulates is a powerful tool for developing high performance composites.

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Many attempts [7–14] have been made on microstructure, strength, creep behavior, and thermostability of SiC_P/Al–Fe–V–Si, but fracture behavior of the materials has hardly been investigated systemically.

Although there are many advantages of SiC_P/Al–Fe–V–Si, the application of the materials is restricted by their low ductility and fracture toughness properties. Especially for the SiC_P/Al–Fe–V–Si with SiC particles aggregation, abrupt break (brittle breakage of SiC particles, or detachment of SiC/Al matrix interface) at the early stage of loading restricts the improvement of mechanical properties and expansion of application of the composite. The SiC/Al matrix interfaces play a critical role in determining properties of metal–matrix composite. Variation of the interfacial strength relies on the deformation temperature. As a result, a clear understanding of the characteristics of the interfaces is central in optimizing the response of the composite to stress arising from an applied load at different tensile temperatures. So, it is necessary to develop a method to improve SiC particle distribution and bonding strength between SiC particle and the matrix.

The present study has been focused on SiC reinforcement distribution improvement by hot pressing, rupture modes at different tensile temperatures, and their inherent mechanisms.

2. Experimental procedure

2.1. *Materials preparation*

In this study, the nominal composition of Al–Fe–V–Si alloy is Al-11. Seven Fe-1.15 V-2.4Si β-SiC particles of a volume fraction of 15% and a mean size of about 10 μm with irregular shape and cubic structure produced by Lianyungang Dongdu Silicon Carbide Co. Ltd were selected as the reinforcement phase. The composite preforms were firstly fabricated by a multilayer spray deposition equipment. The processing parameters of the spray deposition experiments are given in Table 1.

2.2. *Working processes and parameters*

Hot pressing was used for the composite preform densification. The preforms as-deposited were cylinders with 300 mm in diameter and 400 mm in height. Then they were turned to 155 mm in diameter before being hot pressed. The as-turned billets were heated up to 450 °C, then hot pressed to a column with the 165 mm diameter. The hot pressing mould was heated up to 410 °C. The as-pressed columns were turned to 155 mm in diameter again and then hot pressed again.

Table 1. Process parameters of the spray deposition experiments.

Process parameters	Values
Atomization temperature (K)	1223–1373
Diameter of the melt stream (mm)	3.2–3.6
Spray height (mm)	200–350
Rotate speed of the substrate ($r\,\mathrm{min}^{-1}$)	100–350
Scan velocity of the nozzle (s)	10–30
Pressure of the atomization gas (MPa)	0.7–0.9

Before being hot rolled, the columns were sawed into plates and the plates were heated at 490 °C for 1 h. The pass reduction was about 10%. The billet was heated for 10 min/pass. The obtained composite sheets were about 1.0–1.2 mm thick.

2.3. Materials characterization and testing

Tensile specimens with the tensile axis parallel to the longitudinal direction were machined from sheets of the as-rolled composite. Mechanical properties of the composite were examined by means of tensile testing with a tensile strain rate of 5×10^{-4}.

Microstructures of the composite were examined by means of optical microscopy (OM) and scanning electron microscope (SEM).

3. Experimental results and discussion

3.1. Microstructure at different working states

SiC particles are characterized with high melting point, stiffness, and Young's modulus. In recent years, metal matrix composites reinforced with SiC particles have been broadly investigated.[15–17] The shape and distribution of SiC particles in the matrix vary with working process. SiC particles in the composite are broken mechanically with out-of-shape. Shape and distribution in the matrix hence changed with processing technique and deformation.

Figure 1 shows the shape and distribution of SiC particles and fracture surfaces of the composite in different states. Presence of weak structure (porosity, oxide films, and insufficient metallurgical bonding between deposited particles and weak bonding between SiC particles and the matrix) in the as-deposited material is shown in Figure 1 (a). Pores in the spray-deposited composite exhibit a diversity in morphology and their sizes are distributed in the range 10–100 μm. Moreover, the smaller pores are more dispersedly spaced throughout the sample. The primary sources of porosity in deposited materials generally, include entrapped gas, solidification shrinkage, and interstitial porosity.[18] It is shown in Figure 1(b) that the microcracks easily initiate at the pre-existing defects located between deposited powders and between SiC particles and the Al matrix in the deposits, and propagate rapidly along the brittle interfaces between the particles due to local stress concentration. Therefore, the low ductility and poor strength of the as-deposited composite can be attributed to the porosity and the weak interface bonding strength between the particles. Hot pressing can reduce the porosity obviously as shown in Figure 1(c) but cannot eliminate the lamellar structure and the boundaries formed between powder particles formed during spray deposition. The fracture surface of the tensile samples as-pressed presents a great deal of equiaxed dimples resulting from the extraction of SiC particles in Figure 1(d), which indicates that the interfaces between SiC particles and the matrix become a predominant crack source. The primary advantage of hot pressing in comparison with extrusion is that hot pressing will not lead to SiC particles aggregation to the superficial coat. Furthermore, lamellar structure and the boundaries between powder particles can be eliminated by rolling following, being replaced by homogeneous microstructure (Figure 1(e)). A different behavior is observed in the composite as-rolled after hot pressing in Figure 1(f). The fracture path passed through the particles; SiC particles with flat and smooth surfaces are observed at many locations on the fracture surface. It points to the fact that particle breakage has taken place during the fracture process. Few separations at the interface between the SiC particles and the matrix have been noted.

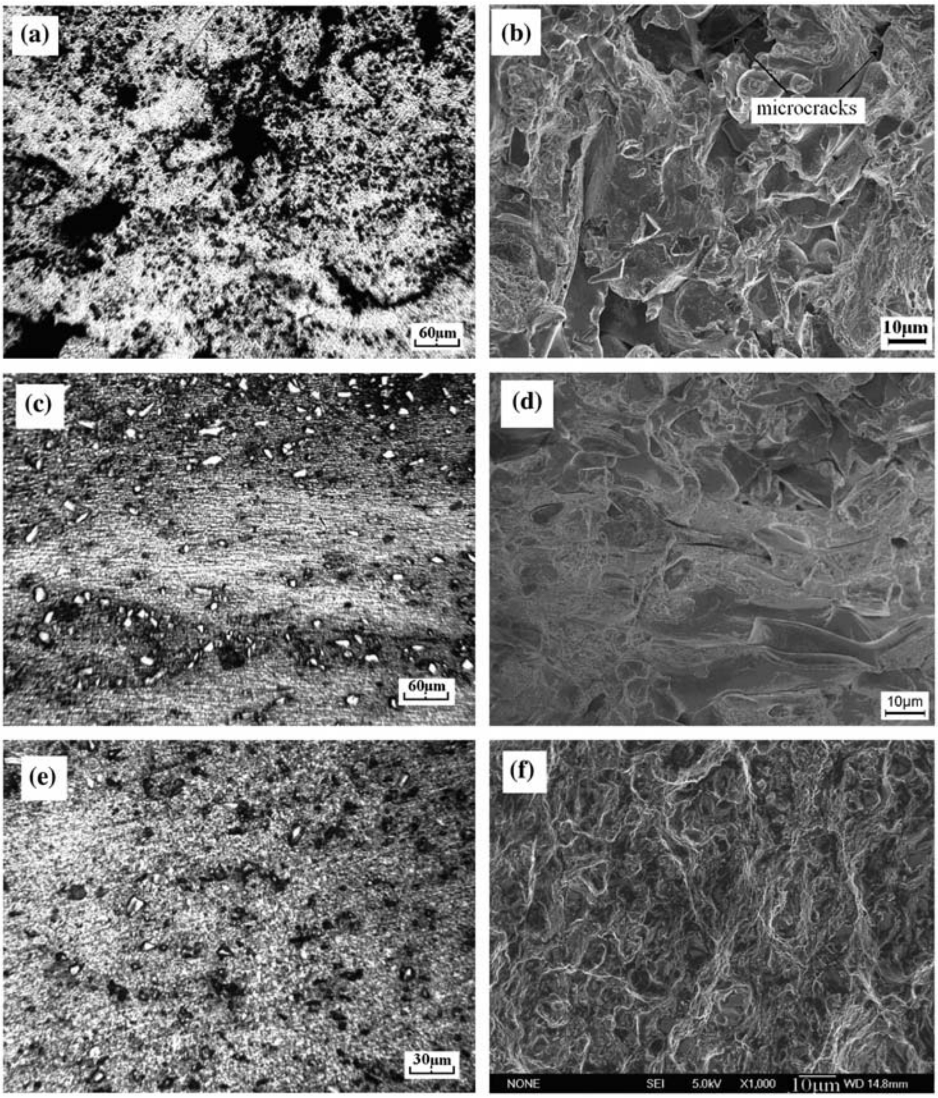

Figure 1. Microstructure (OM: (a), (c), (e), (g)) and fracture surface (SEM: (b), (d), (f), (h)) evolution during densification: (a), (b) as-deposited, (c), (d) as-hot pressed, and (e), (f) as-rolled after hot pressing.

The atoms with long-range disorder of interface indicate that the interface between the SiC particle and the Al matrix is an amorphous layer formed by the oxidation of SiC as shown in Figure 2(a) and (b). When SiC was exposed at temperatures above 800°C in air, oxidation occurs at the surface and a SiO_2 layer was formed, the thickness of the SiO_2 layer was determined by the temperature and holding time. As mentioned above, most of the SiC particles were inserted on the surfaces of the atomized droplets and the SiC particles were heated to a temperature higher than 800 °C by the melt droplets at a temperature of about 1030 °C. Part of the SiC particle surface was exposed in the air and a SiO_2 layer with thickness of about 5nm was formed (Figure 2(b)).

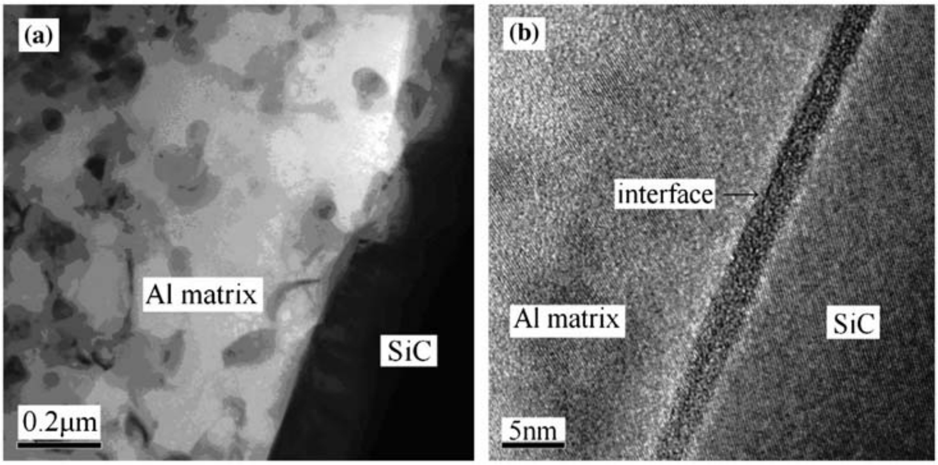

Figure 2. Microstructure near the SiC/Al interface (a) and an amorphous interface between the reinforcing SiC particle and Al matrix (b).

The passive silica layer grown naturally on the SiC particles is expected to act as a protective barrier (diffusion barrier) to prevent the formation of Al_4C_3 by the reaction between Al and SiC as Equation (1) and to improve the wettability of SiC by aluminum, which is a benefit for the mechanical properties of the composite. Good wetting at the interface is required for strong bonding between the SiC particles and the Al matrix, which allows the transfer and distribution of load from the matrix to the reinforcements without fracture failure. Additional advantage from the oxidation of SiC particles at elevated temperatures is the removal of the contaminants, extraneous components, and absorbed water from the surface that help to form a clean and uniform protective interface (Figure 2(a)). The short holding time at elevated temperature (above 800 °C) during spray deposition process results in a thin SiO_2 layer protecting the SiO_2 layer from reacting with Al as in Equation (1).

$$3SiC + 4Al \rightarrow Al_4C_3 + 3Si \text{ (in Al matrix)} \quad (1)$$

3.2. Fractographies at different tensile temperatures

Fractography (SEM) and side view of tensile fracture (OM) of the composite sheets as-rolled at different tensile temperatures after being hot pressed are shown in Figure 3. *The broken particles can be distinguished from the detached particles by making a difference between smooth fracture surface of the broken particles and dimples forming because of the detached particles. The ratio of the broken particles can be evaluated by the number of broken particles devided by the number of the particles that are both broken and detached from the fracture surface*. It can be seen that interfaces between SiC particles and the matrix are relatively reliable at low tensile temperatures. When the sheet is being loaded, the stress assembling along the SiC particles is much higher than that of the matrix. Then the particles are drawn to breakage. Because of particle separation from the matrix during tensile straining, smooth fracture surfaces and few dimples are formed which can be seen in Figure 3(a) and (c). Broken particles can be seen on

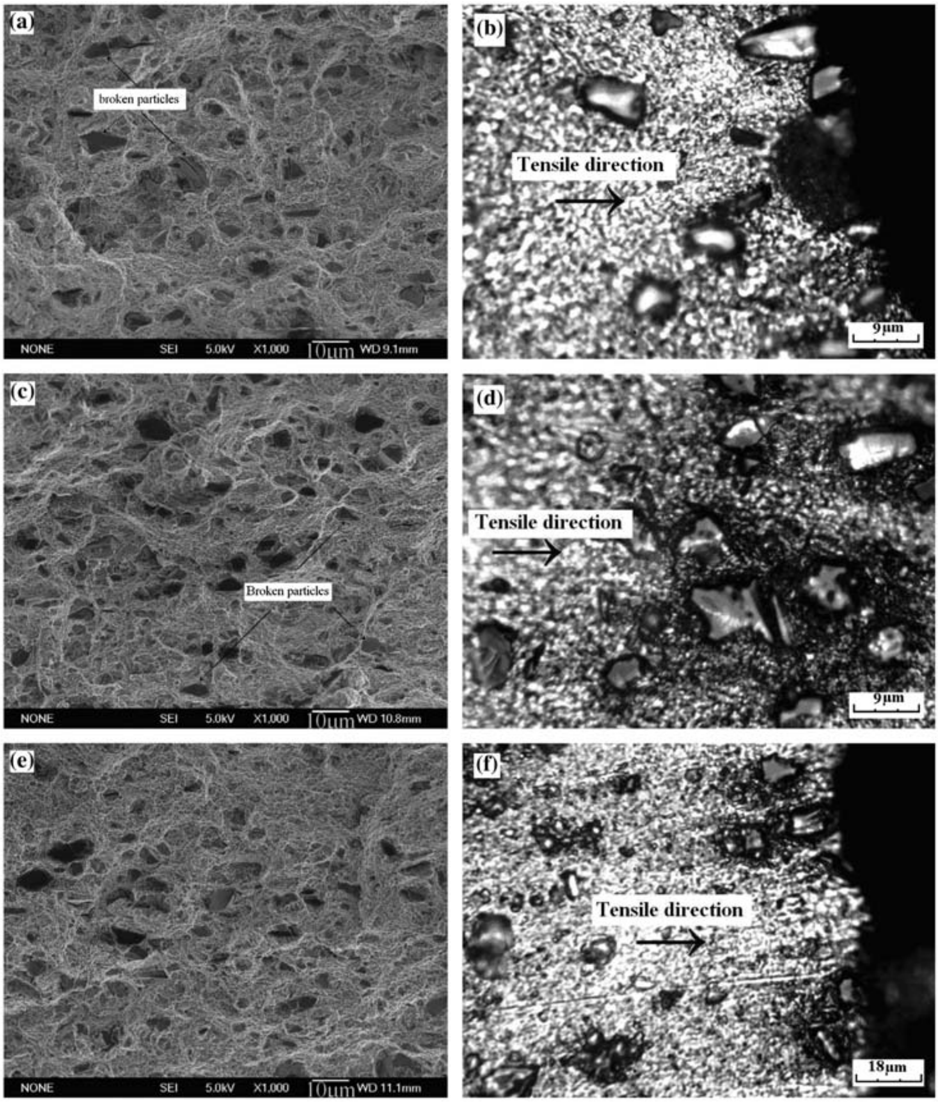

Figure 3. Fracture surface (SEM: (a), (c), (e), (g), (i), (k)) and side face near fracture surface (OM: (b), (d), (f), (h), (j), (l)) of the composite at different tensile temperatures: (a), (b) room temperature, (c), (d) 100 °C, (e), (f) 200 °C, (g), (h) 300 °C, (i), (j) 400 °C, and (k), (l) 450 °C.

the fracture surface as shown in Figure 3(a) at ambient temperature, and the cracked particles can be found in the matrix near the fracture surface with a tensile temperature of 100 °C. Breakage of the most SiC particles and detachment of few particles from the matrix suggests a strong bonding between the SiC particles and the matrix at low tensile temperatures (room temperature and 100 °C as shown in Figure 3(b) and (d), respectively). As the tensile temperature rises, the broken particles decrease and the detached ones increase. Rising the tensile temperature to 200 °C, the ratio of broken particles is about 60% as shown in Figure 3(e) and the ratio is decreased to about 50 and 40% with the temperature rising to 300 and 400 °C as shown in Figure 3(e) and

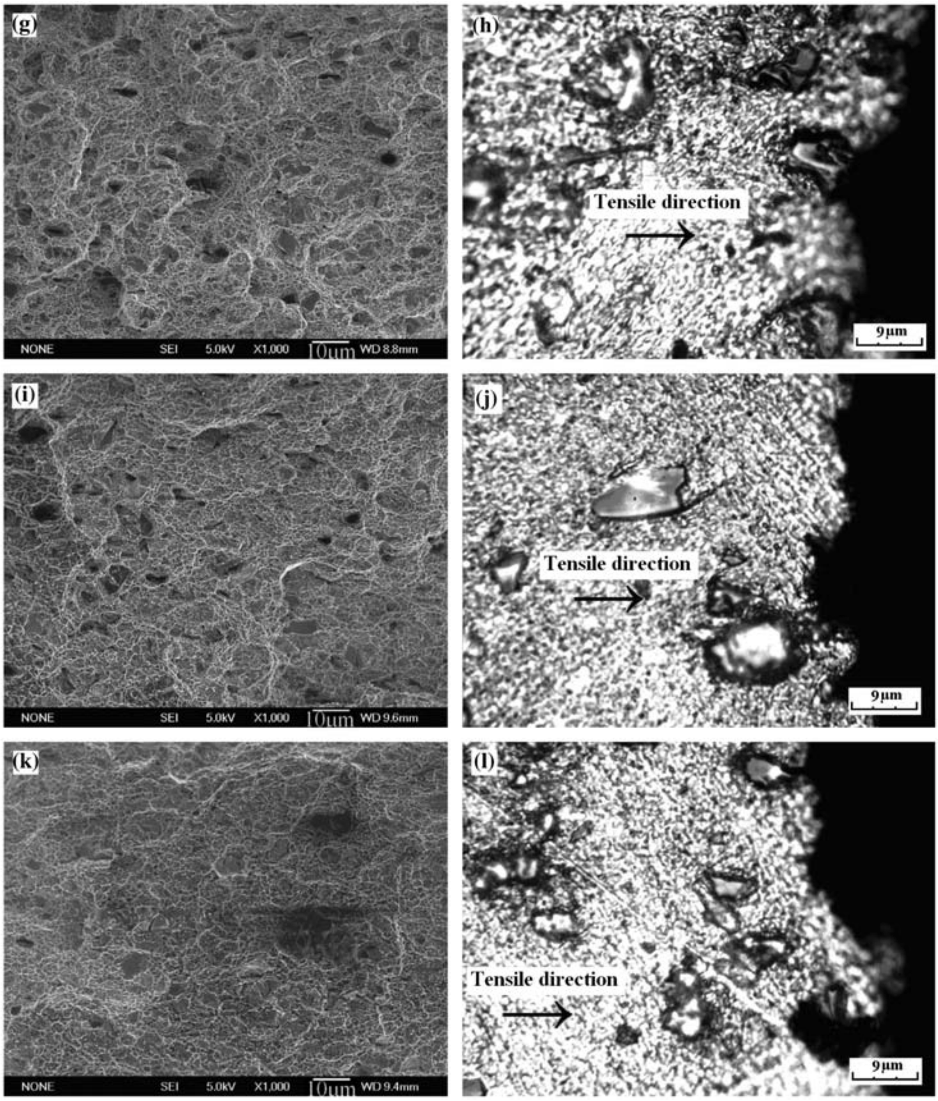

Figure 3. (*Continued*)

(g), respectively. This is consistent with broken particles as well as some dimples resulting from particle detachment in the side views shown in Figure 3(f) and (h). As the temperature elevated to 400 °C, a few particles crack when most of them are pulled out (Figure 3(i)). Accordingly, a crack appears between the SiC particle and the matrix as shown in Figure 3(j) which indicates that the bonding between the SiC particles and the matrix becomes weak with the rise in tensile temperature. With the temperature going up to 450 °C, few broken particles can be seen as in Figure 3(k); crack is formed around the particle in the matrix and cavities appears on the fracture surface in Figure 3 (i). More debonding of the SiC/Al matrix interfaces suggests that the stress concentration has exceeded the bonding strength between SiC particles and Al matrix during tensile testing as the temperature was increased. The expansion of a crack near the area of the SiC/Al matrix interface can be attributed to the lowering of the interfacial strength as a result of temperature elevation.

There are three types of damage modes in the SiC_P/Al–Fe–V–Si composite: cracking of the reinforcing SiC particles, debonding at the matrix–particle interfaces, and cracking in the matrix. It was found that debonding at the matrix–particle interfaces increases as the tensile temperature elevates in Figure 3. At the temperature below 400 ° C, SiC particles crack to a different extent. A combination of thermally induced stress, local stress concentration around the particles, and the natural brittleness of the SiC particle is responsible for the cracking. For the SiC particles cracked, they must be loaded to their fracture strength. This can be achieved generally by the tensile stress and local shear loading through the SiC–matrix interfaces. The extent of particle cracking by the shear mechanism depends on the aspect ratio of the SiC particle. For the case of symmetrically packed particles in a metal matrix, the aspect ratio (S_S) for maximum loading is [19]:

$$S_s = \sigma_{SiC}/\tau_i \tag{2}$$

where σ_{SiC} is the strength of the particle and τ_i is the interfacial shear strength. This indicates that not all the SiC particles are loaded to their fracture strength suggesting the importance of SiC particle size and orientation with respect to the load axis and their distribution in the metal matrix. During tensile deformation at a relative low temperature, SiC particles with larger size fracture first followed by the fracture of smaller sized particles; moreover, the aspect ratio of the cracked particle will increase as the interfacial strength decreases. This suggests that the extent of particle cracking will fall down as the interface weakening. It should be noted that the reduction in the interfacial strength may play a pivotal role in the crack nucleation and propagation as the tensile temperature is being elevated. As the temperature rises, the falling of interfacial strength between SiC particles and Al matrix that is being responsible for debonding at the interfaces increases. At temperatures above 400 °C, debonding at the interfaces becomes the dominant fracture mechanism of the composite.

3.3. Fracture behaviors

Deformation behaviors of SiC_P/Al–Fe–V–Si vary as the tensile temperature changes. Stress–strain curves of SiC_P/Al-11.7Fe-1.15 V-2.4Si sheets at different temperatures with strain rate of 5×10^{-4} are shown in Figure 4. It can be found that both tensile strength and rupture strain of the composite sheets decrease as the tensile temperature rises, which is different from those of Al–Fe–V–Si alloy without particle reinforcement with a brittle zone for the alloy tensed at temperature from 100 to 150 °C. [20] The rupture strain of the composite is 7.7% at room temperature, then, decreases to 6.4% gradually when rising the temperature to 300 °C. The strain decreases to 5.1% sharply when rising the temperature up to 400 °C. It can be found that the rupture elongation of the composite acts consistently with the effect of rupture strain as the tensile temperature elevated. The elongation decreases from 4.5 to 3.0% gradually when the temperature rises from room temperature to 300 °C. In accordance with the effect of rupture strain at 400 °C, the rupture elongation of the composite decreases to 1.4% rapidly.

In accordance with Figure 4, tensile strength (σ_b), yield strength, ($\sigma_{0.2}$) and rupture elongation(δ) of the composite decreases with the tensile temperature elevated as shown in Table 2. Thetensile strength and yield strength decreases from 581.2 and 518.4 MPa

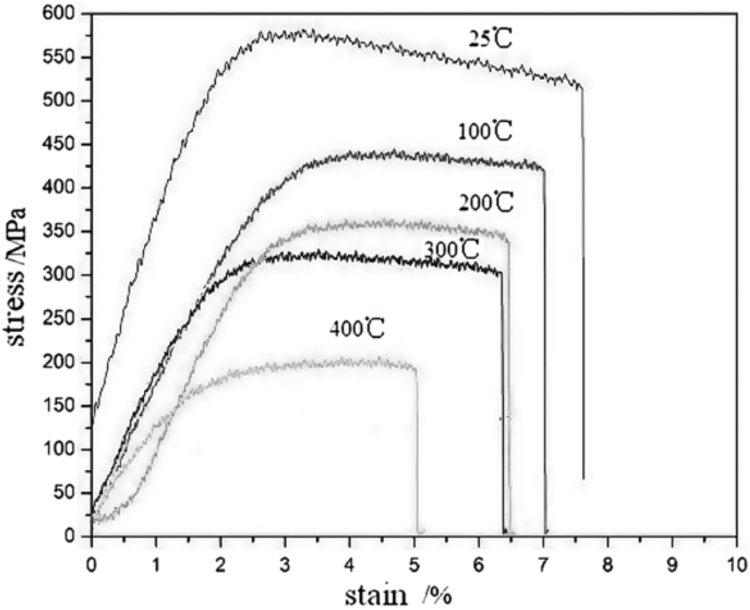

Figure 4. Stress–strain curves of SiC_P/Al-11.7Fe-1.15 V-2.4Si sheets at different tensile temperatures (for stain rate of 5×10^{-4}).

Table 2. Mechanical properties of composite sheets at different tensile temperatures.

Tensile temperature (°C)	σ_b (MPa)	$\sigma_{0.2}$ (MPa)	δ (%)
25	581.2	518.4	4.5
100	461.5	362.7	4.2
200	372.2	221.2	3.3
300	315.8	196.6	3.0
400	232.6	127.7	1.4

to 315.8 and 196.6 MPa, respectively. As the temperature elevates to 400 °C, both the strength and elongation drop sharply.

As mentioned above, the process of rolling after hot pressing can densify the porous composite and improve SiC particle distribution effectively. Cracking of the reinforcing SiC particles and debonding at the matrix–particle interfaces are the two main fracture modes in the SiC_P/Al-11.7Fe-1.15 V-2.4Si composite, and the dominant fracture mode depends on SiC/Al matrix interfacial strength. At the temperature below 300 °C, cracking of SiC particles is the dominant fracture mode because of the strong bonding between the reinforcing particles and the matrix, and mechanical properties of the composite sheets stay on a relatively high level. At temperatures above 400 °C, debonding at the matrix–particle interfaces becomes the dominant cracking mode as a result of bonding weakening sharply between the particles and the matrix.

4. Conclusions

The results of an investigation on the fracture behavior of SiC_P/Al–Fe–V–Si provide the following key observations:

(1) Homogenous SiC particle distribution can be obtained by the process of rolling after hot pressing. Hot pressing does not lead to SiC particles aggregation. This is the most important factor contributing to the homogeneous microstructure.
(2) An amorphous SiO_2 interface of 5 nm in thickness is observed on the surface of the SiC_P/Al–Fe–V–Si composites as-rolled. The amorphous SiO_2 layer improves the wettability of SiC by the aluminum matrix and results in a high strength bond. Strong bond interfaces contribute to improve the mechanical properties.
(3) Fracture modes of the composite depend on the tensile temperature. Cracking of SiC particles is the dominant fracture mode because of the strong interface bonding with the tensile temperature below 300 °C. While the temperature being above 300 °C, debonding at the matrix–particle interfaces becomes the dominant cracking mode as a result of the bond's sharp weakening between the particles and the matrix.
(4) Both of strength and plasticity of the composite sheets deteriorate as the tensile temperature elevates. The tensile strength, yield strength, rupture strain, and rupture elongation are 581.2, 518.4 MPa, 7.7, and 4.5% at ambient temperature. And, they decrease to 315.8, 196.6 MPa, 6.4, and 3.0% gradually when the tensile temperature increases to 300 °C; they then drop to 232.6, 127.7 MPa, 5.1, and 1.4% markedly because of interfacial bond's sharp weakening at 400 °C.

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