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Effect of Multidirectional Forging and Heat Treatment on Mechanical Properties of *In Situ* ZrB₂p/6061Al Composites

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Abstract: Aluminum matrix composites that were reinforced by *in situ* zirconium diboride nanoparticles were fabricated from an aluminum-potassium tetrafluoroborate-potassium hexafluorozirconate system via a direct-melt reaction. The morphologies of the *in situ* particles and the mechanical properties of the composite were investigated by scanning electron microscopy, energy-dispersive X-ray spectroscopy, X-ray diffractometry, optical microscopy and tensile tests. X-ray diffractometry and energy-dispersive X-ray spectroscopy showed the existence of zirconium diboride in the composite. Multidirectional forging and heat treatment were used to determine the effect of plastic deformation and heat treatment for the highest tensile strength applied to the composite. Multidirectional forging and heat treatment have a positive influence on the composite microstructure and tensile strength, which increase by 21.81% and 13.43%, respectively. The mechanism of multidirectional forging and heat treatment that affects the composite mechanical properties has been discussed. The heat-treatment parameters that affect the tensile strength include the solution-treatment temperature, aging temperature and aging time. The highest tensile strength with moderate extensibility loss was achieved for a specimen that was solution treated for 4 h at 793 K and with aging at 423 K for 4 h.

Keywords: composite, multidirectional forging, heat treatment, zirconium diboride

Introduction

Particulate-reinforced aluminum matrix composites (PRAMCs) display advantages such as a simple synthesis, relative inflexibility, strength and lower cost. They have been used widely in aerospace, high-speed railway systems and the automobile industry and have attracted interest from scientific researchers [1, 2, 5]. Zirconium, which can form a zirconium-diboride (ZrB₂) phase, is one of the most common aluminum-alloy additives because of its excellent properties, such as a higher hardness, modulus and melting point [6]. Thus, researchers have focused on aluminum matrix composites that are reinforced with ZrB₂. Gaurav et al. [7] found that the presence of nanoZrB₂ particles can restrict the solidification front and act as nucleation sites for the AA5052 matrix to refine the cast structure. Dinaharan et al. [8] found that *in situ*-formed ZrB₂ particles refined the microstructure significantly and enhanced the mechanical properties of the aluminum alloy, which include hardness, ultimate tensile strength and wear properties.

Among the PRAMC fabrication techniques, the direct-melt reaction (DMR) in which inorganic salt reactants are added to the aluminum melt, is one of the most commonly used techniques to fabricate PRAMCs because of its versatile particle size, economic viability and strong interfacial bonding. However, for PRAMCs that are synthesized by direct melt reaction (DMR), reinforcing particles with a volume fraction above 8 vol.% tend to aggregate as clusters, which reduces their strengthening effect and results in a decrease in tensile ductility [9, 10]. Extensive coarse grains and defects exist after DMR. Thus, a variety of metallurgical and material processing technologies have been investigated to prevent nanoparticle aggregation during DMR to improve the PRAMC mechanical properties. Jiao et al. [11] indicated that ultrasonic and magnetic-couple fields may refine the nanoparticles to 80–100 nm during DMR. In addition to metallurgical methods, material-plastic processing has been applied to solve the drawbacks of as-cast composites as discussed above. Plastic

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deformation may cause work hardening and change matrix alloy microstructures, which may improve the reinforcement distribution state. Heat treatment generates solution strengthening and a dispersed network phase, which can also influence the mechanical properties of the aluminum matrix composites. Plastic deformation and heat-treatment methods may improve the mechanical properties of the aluminum matrix composites and has also been researched in relation to PRAMCs. Zhao et al. [12] found that the tensile strength and hardness of aluminum alloy AA6063 increase with an increase in extrusion speed and temperature. Hanim et al. [13] analyzed the influence of heat treatment on the mechanical properties of an aluminum alloy with heat treatment and studied the microstructure. Until recently, many reports have been provided regarding the preparation and mechanical properties of particle-reinforced aluminum matrix composites. Relatively few reports exist on the microstructure and strengthening effects of multidirectional forged *in situ* ZrB₂p/aluminum alloy matrix composite AA6061 (6061Al) composites and their mechanical properties after solution treatment and artificial aging with different heat treatment parameters.

This work focuses mainly on the effect of multidirectional forging on composites and the optimization of heat-treatment parameters for a forged ZrB₂p/6061Al composite. Scanning electron microscopy (SEM) and optical microscopy (OM) were used to scan and analyze the microstructure of the as-cast, forged and heat-treated composites, respectively. An orthogonal experimental design was performed to study the tensile strength of the forged ZrB₂p/6061 Al composite regarding changes of the solution-treatment temperature, the aging temperature, and the aging time. The best heat-treatment solution and the influencing sequence of the heat treatment parameters on tensile strength were identified and verified by an orthogonal experiment. These results may provide a theoretical foundation for further research and the development of high-performance PRAMCs.

Experimental

Composites and multidirectional forging

Aluminum alloy matrix composite AA6061, which has excellent casting properties, was used. Its chemical composition is listed in Table 1. Inorganic salt potassium hexafluorozirconate (K₂ZrF₆, purity > 99 %) and potassium tetrafluoroborate (KBF₄, purity > 99 %) powder were used.

Table 1: Chemical composition of AA6061 alloy (mass fraction, %).

Si	Mg	Fe	Cu	Mn	Al
0.42	0.62	0.195	0.015	0.015	Bal

The volume fraction of the generated ZrB₂ particles was 10 %. The K₂ZrF₆ and KBF₄ powders (with a mass ratio of 1:1) were dried in an electronic oven for 2 h at 473 K to remove water, cooled to room temperature, mixed and preserved in aluminum foil. AA6061 (1300 g) was heated to 1123 K and melted in a resistance furnace. A thermocouple was used to detect the melt temperature in real time. The K₂ZrF₆ and KBF₄ powders were added to the aluminum melt with mechanical agitation for 10 min with a 50-mm diameter three-bladed mechanical impeller at 150 rpm. When the reaction was complete, a hexachloroethane degasifying agent was used to degas and remove slag in the melt. The melt was poured into a copper mold at 993 K. After being cooled to room temperature, an aluminum matrix composite ingot resulted.

The ingot was machined to 70×60×110 mm and was multidirectional forged with a self-made instrument. To reduce friction, the specimen and anvil interface were spread with graphite lubricant as a high-temperature lubricant during the forging process. Multidirectional forging was performed at 720 K and 12.5 mm/s. Figure 1 shows that the specimen was multidirectional forged with a 90° changing direction loading pass by pass. For each pass, the true strain was estimated as:

$$\varepsilon = [\ln(H/h)] \quad (1)$$

where H is the initial and h is the final specimen height. The average strain for each pass is ~0.45. After the seven multidirectional forging passes, the specimens were water-quenched to study the composite microstructure post-processing.

Heat-treatment parameters designed by orthogonal testing

After multidirectional forging, heat treatment was applied to relieve residual stresses, to improve the composite mechanical properties and to obtain the optimal treatment parameters for the highest ultimate tensile strength.

Forged sample (12.06 mg) was sampled from the forged specimen, and thermogravimetry and differential scanning calorimetry/differential thermal analysis (TG-DTA/DSC, Pyris Diamond) was conducted up to

Figure 1: Schematic of multidirectional forging.

1073 K at 15 K/min. The DTA results showed fluctuations at 893 K, which indicates that the sample had a poor thermal stability at this temperature. Therefore, the upper-limit temperature of the solution treatment was set to 893 K. To determine the best heat-treatment solution for the extruded specimen and the primary and secondary factors of heat treatment with fewer tests, an orthogonal experimental was designed with three factors and three levels. Nine orthogonal tests were conducted as shown in Table 2. The solution treatment temperatures were 853 K, 793 K and 733 K, respectively. Three aging temperatures and times of 403 K, 423 K and 443 K and 4 h, 8 h and 12 h, respectively, were tested. A, B and C represent the solution-treatment temperature, the aging time and aging temperature, respectively. The heat treatment scheme $L_9 (3^3)$ is shown in Table 3.

Table 2: Factors and levels.

Levels	Factors of orthogonal test		
	Solution treatment temperature (A)/K	Aging time (B)/h	Aging temperature (C)/K
1	733	4	403
2	793	8	423
3	853	12	443

Table 3: Experimental design with $L_9 (3^3)$ orthogonal table.

Sample number	Factors of orthogonal test			
	Solution treatment time/h	Solution treatment temperature (A)/K, (level)	Aging time (B)/h, (level)	Aging temperature (C)/K, (level)
①	4	853,(3)	4,(1)	403,(1)
②	4	733,(1)	4,(1)	423,(2)
③	4	793,(2)	4,(1)	443,(3)
④	4	733,(1)	8,(2)	403,(1)
⑤	4	793,(2)	8,(2)	423,(2)
⑥	4	853,(3)	8,(2)	443,(3)
⑦	4	793,(2)	12,(3)	403,(1)
⑧	4	853,(3)	12,(3)	423,(2)
⑨	4	733,(1)	12,(3)	443,(3)

Material characterization

Specimens (15 mm × 15 mm × 10 mm) were obtained from the as-prepared composite from each experimental period for SEM, X-ray diffractometry (XRD) and optical microscopy (OM) analysis. The observed surface was ground on emery paper from 400# to 2000# and polished. SEM (Hitachi, Model S4800/Su1510) was used to scan the microstructure and morphology of the *in situ* particles of the composite. The metallurgical structure was studied by OM (Olympus, Model GX51), and XRD (Bruker, Model D8 Advanced) was used to analyze the composite phase.

Specimens for tensile testing (the specimen size is shown in Figure 2) were obtained from the composite for each experimental period. The tensile-test direction of the multidirectional forged specimen is perpendicular to the forging direction. Each tensile test was performed three times and the average value was calculated. A WDSZ-100 universal testing machine was used to measure the specimen tensile strength with a test force of 5 kN at 1 mm/min.

Figure 2: Specimen size for tensile test.

Results and discussion

Microstructure of as-cast ZrB₂/6061Al composites

The probable reaction equations of the Al-KBF₄-K₂ZrF₆ reaction system are shown in Table 4. The value of ΔG

for each reaction equation was calculated, with a reaction temperature of 1123 K. The negative values show that a melt reaction will occur.

Figure 3 shows the SEM microstructures and energy-dispersive X-ray spectroscopy (EDS) spectra of the ZrB₂ phases that were fabricated from the Al-KBF₄-K₂ZrF₆ system. The EDS analysis in Figure 3 (b) shows that elemental Al, Zr and B are present in the composite. The XRD pattern of this specimen depicts diffraction peaks of the Al₃Zr and ZrB₂ phases in Figure 4, with the latter being the reinforcement. A ZrB_{2p}/6061Al composite was prepared via an *in situ* reaction.

Effects of multidirectional forging on ZrB₂/6061Al composites

Figure 5 shows SEM images of the ZrB_{2p}/6061Al composite. Figure 5 (a) shows the as-cast composite specimen before multidirectional forging. Figure 5 (b) shows the SEM image of the central part of the forged ingot after seven passes. Figure 5 (c) shows the ZrB₂ nanoparticle distribution on the edge of the forged specimen after seven passes.

The darker area in Figure 5 (a) is the aluminum matrix, whereas the brighter spots are ZrB₂ particles. Hexagonal ZrB₂ particles are agglomerated. Figure 5 (b)

Table 4: Reaction equations of *in situ* composite fabricated with Al-KBF₄-K₂ZrF₆ system.

	Reaction equation	ΔG _{850 °C} /(kJ · mol ⁻¹)
Molecular equation	2KBF ₄ + 3Al = AlB ₂ + 2KAIF ₄	-392.4
	3K ₂ ZrF ₆ + 13Al = 3Al ₃ Zr + K ₃ AlF ₆ + 3KAIF ₄	-326.4
	AlB ₂ + Al ₃ Zr = ZrB ₂ + 4Al	-57.1
Atomic equation	3Al + [Zr] = Al ₃ Zr	-685.9
	Al + 2[B] = AlB ₂	-157.1
	[Zr] + 2[B] = ZrB ₂	-75.9
Reaction equation	2KBF ₄ + 3K ₂ ZrF ₆ + 12Al = 2Al ₃ Zr + ZrB ₂ + K ₃ AlF ₆ + 5KAIF ₄	-353.1

Figure 3: (a) SEM microstructure and (b) EDS graphs of ZrB₂ fabricated from Al-KBF₄-K₂ZrF₆ system.

Figure 4: XRD pattern of composite reinforced by Al₃Zr and ZrB₂.

shows that after seven multidirectional hot-forging passes, the ZrB₂ particles are distributed better than previously. Figure 5 (c) shows that the ZrB₂ nanoparticles are dispersed directionally and are perpendicular to the last compression axis. A similar result has been obtained for SiC particle-reinforced AZ91D magnesium matrix composites [14, 15]. The improvement in ZrB₂ nanoparticle distribution occurs mainly because of the kneading effect

that is induced by turbulent flow of the AA60601 matrix during the repeated plastic deformation [15]. A single-pass compression produces a greater horizontal displacement near the edge of the specimen than in the center of the specimen so that nanoparticle reinforcement in this region shows a directional distribution.

Fewer ZrB₂ nanoparticle clusters exist in Figures 5 (b) and (c) and these transform to well-distributed tiny particles that are finer than those without multidirectional forging. Some ZrB₂ particle aggregations are still visible after multidirectional forging. However, the average particle-aggregation size also decreases from 20–40 μm to 3–10 μm after multidirectional forging. In this research, the stress around the ZrB₂ nanoparticle clusters cannot be released during multidirectional forging. Because there are comparatively strong forces that act on the larger particle clusters than on the smaller clusters or dispersed nanoparticles, the larger clusters have a higher possibility of fracture [16]. Multi-pass forging may break up larger reinforced particles or clusters and improve the particle size and distribution significantly, which may promote the composite mechanical performance [17]. Because the ZrB₂ particle deformation is less than that of the aluminum matrix, another explanation for the cluster dispersion is that the AA6061 matrix can flow

Figure 5: SEM microstructure of (a) as-cast composite, (b) center and (c) edge of multidirectional forged composite.

into the particle clusters so that the clusters separate and the particle distribution is improved with an increase in the multidirectional forging passes [16].

Figure 6 shows the optical microstructure of the as-cast composites and those before and after multidirectional forging. Figure 7 shows the matrix grain size in different processing periods and sample positions. The average matrix grain size was measured by the mean linear-intercept method. In the as-cast composite, the average matrix grain size was 85 μm , as shown in Figure 6 (a). Mainly fine grains and recrystallization are shown in Figures 6 (b) and (c). The average matrix grain size decreased significantly after multidirectional forging. The sizes of the minority of grains are larger than others in Figure 6 (b). This is attributed to the non-uniform deformation of grains during multidirectional forging [16] and the inhomogeneous nanoparticle distribution during forging: the grain size in regions with fewer ZrB_2 nanoparticles increases because the nanoparticles may constrain matrix grain growth [17]. After heat treatment, the tendency for recrystallization and grain growth is obvious, so the average matrix grain size increases.

Figure 7: Matrix grain size in different processing periods and positions of the composites.

Table 5 shows the density and porosity of as-cast and multidirectional-forged $\text{ZrB}_{2p}/6061\text{Al}$ composites. The density measurement complied with ISO standard (ISO 3369-2006) [18]. The density of the as-cast composites is lower than its theoretical density, and thus the porosity

Figure 6: Optical microstructure of (a) as-cast composite, seven-passes forged $\text{ZrB}_{2p}/6061\text{Al}$ composites: (b) center and (c) edge of specimen and (d) heat-treated (A2B1C2) specimen.

Table 5: Effect of multidirectional forging on composite density and porosity.

Material	Theoretical density / $\text{g}\cdot\text{cm}^{-3}$	Actual density / $\text{g}\cdot\text{cm}^{-3}$	Porosity (vol.) / %
ZrB _{2p} /60601Al composite (as-cast)	3.0295	2.90	4.27
ZrB _{2p} /60601Al composite (multidirectional forged)	3.0295	3.00	0.97

of the as-cast composites is 4.27 %. For multidirectional forged composites, the porosity is considerably lower at 0.97 %. The higher as-cast composite porosity may result because of the difference in coefficient of thermal expansion (CTE) between the matrix and the reinforcement ($\text{CTE}_{\text{AA6061}} = 24.5 \times 10^{-6}/\text{K}$, $\text{CTE}_{\text{ZrB}_2} = 6.8 \times 10^{-6}/\text{K}$), which results in microgap formation during solidification. Second, microgaps may form in the dendritic arm as the feeding process is insufficient during gravity casting. Finally, gas entrapment and hydrogen porosity during casting may lead to porosity formation.

Optimization heat-treatment parameters

To obtain the optimum heat-treatment parameters, orthogonal tests were performed and the orthogonal parameters were determined as shown in Table 3.

Tensile tests were conducted after the orthogonal test with the heat-treated-specimen results shown in Table 6. (The data in Table 6 are averages of three tensile tests for each specimen.)

Table 6: Tensile strength of specimen after multidirectional forging and heat treatment.

Specimen number	①	②	③	④	⑤	⑥	⑦	⑧	⑨
Average tensile strength/MPa	210	212	222	207	227	207	212	210	204

To analyze the orthogonal test results, the sum of the index (K) and the average value of the index (k) of each level were calculated. The range (R) of each level was calculated. The results are shown in Table 7 and $K_{A1} = 623$, $K_{A2} = 661$, $K_{A3} = 627$; $K_{B1} = 644$, $K_{B2} = 641$, $K_{B3} = 626$ and $K_{C1} = 629$, $K_{C2} = 649$, $K_{C3} = 633$ (K_{jm} is the

Table 7: Analysis of orthogonal test.

Test index	Test parameters		
	A (Solution treatment temperature)	B (Aging time)	C (Aging temperature)
K_1	623.00	644.00	629.00
K_2	661.00	641.00	649.00
K_3	627.00	626.00	633.00
k_1	207.67	214.67	209.67
k_2	220.33	213.67	216.33
k_3	209.00	208.67	211.00
Range (R)	12.66	6.00	6.66
The calculated optimal level	A2	B1	C2
Primary and secondary factors	A > C > B		

sum of level m in column j in the orthogonal list in Table 3, and k_{jm} is the average value of K_{jm}). So $R_A = 12.66$, $R_B = 6.00$ and $R_C = 6.66$ and $R_A > R_C > R_B$. This means that factor A, the solution treatment temperature, is the predominant factor that impacts the tensile strength of the ZrB_{2p}/6061Al composite, followed by factor C, which is the aging temperature. Factor B, the aging time, influenced the composite tensile strength less. A comparison of K shows that A2B1C2 was the best theoretical solution in this orthogonal test.

However, the solution of A2B1C2 did not appear in nine of the real heat-treatment experiments in the orthogonal test. Therefore, a contrast test was required to compare the optimal theoretical solution (A2B1C2) with the best solution from the real experiment (A2B2C2). No. 10 specimen of the multidirectional forged composite was prepared and its tensile strength was tested after heat treatment followed by the solution of A2B1C2. The tensile strength of specimen 10 was 228 MPa, which is higher than that of No. 5. Therefore, the best heat-treatment solution for forged ZrB_{2p}/6061Al composite is solution treatment for 4 h at 793 K, and artificial aging for 4 h at 423 K.

Effect of multidirectional forging on composite tensile strength

Figure 8 shows the tensile stress–strain curves of the as-cast, multidirectional forged and heat-treated specimens. The tensile strength increased significantly after forging, whereas the elongation was lower than that of the as-cast specimen. According to the optimum parameter, the

Figure 8: True stress-strain composite curve.

ultimate tensile strength of the specimen after heat treatment decreased slightly, and its elongation improved. The final processed specimen shows an ultimate tensile strength and elongation of 228 MPa and 13.5%, respectively; the ultimate tensile strength is 1.38 times greater than the as-cast $\text{ZrB}_{2p}/6061\text{Al}$ composite.

The OM images of the metallography microstructures of these specimens are shown in Figure 6. Figure 6 (b) and (c) show that the grains of the forged matrix are aligned in a certain direction in the center and on the edge of the forged specimen. After plastic deformation, metal deformations generate a misdistribution of dislocations, which hinders the dislocation movement so that a larger force is required to drive the dislocation movement by overcoming obstacles. This led to the specimen generating work hardening.

The metal yield strength can be estimated from the classic Hall-Petch equation:

$$\sigma_y = \sigma_0 + K_y d^{-1/2} \quad (2)$$

where σ_y is the yield strength, d is the mean grain size and σ_0 and K_y are the material constants. Thus the yield strength of $\text{ZrB}_2/6061\text{Al}$ increases in a certain direction because the dendritic spacing of the aluminum matrix is narrower in a certain direction after seven passes of multidirectional forging.

Multidirectional forging breaks up larger reinforced particle clusters [19] and improves the particle sizes and distribution significantly as mentioned previously. The better distribution of nanoparticles after multidirectional forging may also contribute to the improvement in forged composite (Figures 5 (a) and (b)). Therefore, the mechanical performance of the forged composites has been improved from 165 MPa to 201 MPa (Figures 8).

Mechanism of heat treatment affects composite properties

As shown in Figure 8, the ultimate tensile strength and elongation of heat-treated specimens followed by the A2B1C2 parameter has been improved slightly. Compared with the forged composite, the ultimate tensile strength of the heat-treated specimen increased by 13.43% and the elongation decreased by 9%. The mechanical property of the forged and heated-treated composite improved compared with the as-cast composite.

The effect of heat treatment is reflected mainly in the solid-solution strengthening, artificial aging and thermal-generated dislocation. For solid-solution strengthening, alloy elements have a maximum dissolution in the metal matrix and increase the density of dislocations in the metal matrix after solution treatment. Lattice distortion forms a Cottrell atmosphere [20] and induces solution strengthening, which increases the composite tensile strength. After artificial aging, a tiny dispersed network phase separates from the crystal and distributes along the crystal boundary to form a chain phase. Aging may improve the ultimate tensile strength and reduce the aluminum-matrix elongation.

Particles in the multidirectional forged composite also contribute significantly to the increase in tensile strength during solution treatment. In their research of the thermal expansion of metal-matrix composites, Vaidya et al. [21] discovered that when the temperature changes during the process, the radial stress σ_r and tangential stress σ_θ that are generated at the matrix-particle interface (Figure 9) can be calculated from:

Figure 9: Graph of thermal stress at matrix-particle interface in extruded composite.

$$\sigma_r = P[(a/r)^3 - V_p]/(1 - V_p) \quad (3)$$

$$\sigma_\theta = P[0.5(a/r)^3 + V_p]/(1 - V_p) \quad (4)$$

where P is the interfacial stress and can be calculated from:

$$P = \frac{\Delta\alpha \cdot \Delta T}{\frac{0.5(1+V_m) + (1+2V_m)}{E_m(1-V_p)} + \frac{V_p(1-2V_m)}{E_p}} \quad (5)$$

where r is the distance between the force point in the matrix and the center of the particle, a is the particle radius, V_p is the volume fraction of the reinforcement, $\Delta\alpha$ is the difference in CTE between the matrix and the particles ($\text{CTE}_{\text{AA6063}} = 24.5 \times 10^{-6}/\text{K}$, and $\text{CTE}_{\text{ZrB}_2} = 6.8 \times 10^{-6}/\text{K}$) and ΔT is the temperature change during heat treatment. Li et al. [22] indicated that when the Al-matrix temperature exceeds 573 K, $|\sigma_r - \sigma_\theta|/\sigma_y$ (σ_y is the yield strength of the matrix), plastic deformation may occur at the matrix-particle interface because of the difference in CTE between the matrix and the particle. This leads to the generation of a high density of dislocations at the matrix-particle interface in the composite because of the mismatch in strains between the matrix and the particles. The dislocation density ρ can be estimated from [23]:

$$\rho = \frac{BV_f\varepsilon}{bt(1-f)} \quad (6)$$

where

$$\varepsilon = \Delta\alpha \cdot \Delta T \quad (7)$$

B refers to a geometric constant that is between 4 and 12 in theory, ε is the thermal strain, V_f is the volume fraction of reinforcement, b is the Burgers vector and t is the smallest dimension of the reinforcement. During solution treatment, a high dislocation density is generated as a result of quenching from the solution-treatment temperature (793 K) to room temperature. A high dislocation density that hinders dislocation movement at the matrix-particle interface will also increase the tensile strength of the forged composite after solution treatment.

In situ ZrB₂ particles generate a high dislocation density at the matrix-particle interface. With the effect of solid-solution strengthening and the precipitated phase, heat treatment improves the mechanical properties of the ZrB_{2p}/6061Al composite considerably.

Effect of different heat-treatment parameters on composite mechanical properties

The heat-treatment process is based on atomic diffusion. From Fick's law:

$$J = -D \frac{dC}{dx} \quad (8)$$

where

$$D = D_0 \exp[-Q/(RT)] \quad (9)$$

When the heat-treatment temperature increases slightly, the spreading increases, which implies that the solution and aging treatment of the ZrB_{2p}/6061Al composite is temperature sensitive. A higher heat-treatment temperature equates to a faster spreading rate of solute atoms and a better solid distribution, which increases the amount of precipitated phase after artificial aging. The increasing temperature increases the phase-transformation driving force and the critical nucleus size of the precipitated phase decreases. If the aging temperature is too high, it may cause grain growth and affect the composite mechanical properties negatively. For solute treatment, overheating occurs when the treatment temperature is too high, which leads to material melt along the grain boundary [24], and decreases the mechanical properties significantly.

Heat-treatment time is another factor that may affect the mechanical properties of the composite specimens. For solution treatment, an increase in solution-treatment time will increase the solute atoms, and thereby increase the matrix strength. However, as shown in Figure 7, the average grain size has increased after heat treatment and the matrix grain growth should be controlled to improve the composite properties. Alternatively, the matrix-metal grain may grow significantly when the heat-treatment time is too long [25] and it may reduce the strengthening effect of the heat treatment.

In comparison, artificial aging is not time-sensitive [26]. However, artificial aging still has its heat parameter for treatment time. The ordinary artificial aging time for the 6000-series aluminum alloys to reach maximum strength is at greater than 5 h [27], but the best artificial aging time for the ZrB_{2p}/6061Al composite is shorter. This results mainly from the acceleration of aging kinetics in the composite. The thermal energy generates high-density dislocations during solution treatment at the matrix-particle interface, which are suitable sites for the heterogeneous nucleation of precipitated phase [23]. Under this condition (4 h of aging treatment), the aging kinetic of the ZrB_{2p}/6061Al composite is faster than its matrix alloy. Therefore, 4 h is the optimal artificial aging time over 8 h or 12 h to reach the highest ultimate tensile strength for the ZrB_{2p}/6061Al composite.

Conclusions

- (1) An *in situ* ZrB₂/6061 Al composite was synthesized via an Al-KBF₄-K₂ZrF₆ reaction system by a direct-melt reaction.
- (2) After multidirectional forging, the ZrB₂-reinforced particles have a better distribution in the composite, and the matrix-alloy grain has refined. The composite tensile strength increases by ~22%.
- (3) Heat-treatment parameters have been optimized by orthogonal testing, and the best heat-treatment solution for the highest ultimate tensile strength is solution treatment for 4 h at 793 K and artificial aging for 4 h at 423 K.
- (4) The tensile strength of the composite reaches a maximum of 228 MPa, with an increase of 13.43% relative to that without heat treatment, and with a moderate decrease in extensibility with the optimal heat-treatment parameters.
- (5) The effect of different heat-treatment parameters on the mechanical properties has been discussed.

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