Effects of Ag, Co, and Ge additions on microstructure and mechanical properties of Be-Al alloy fabricated by investment casting

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Abstract: The effects of Ag, Co, and Ge additions on microstructure and mechanical properties of Be-35AI (wt.%) alloys fabricated by investment casting were studied. The results reveal that the trace metals 1.5wt.% Ag, 0.7wt.% Co, and 0.8wt.% Ge additions do not change the nucleation temperature of Be phase. However, the nucleation temperature of the Al phase decreases from 642 °C to 630 °C by DSC due to the Ge addition. The strength of the alloys sharply increases due to the dissolution of the Ag and Ge solutes into the Al phase and the Co into the Be phase characterized by SEM and EDS. Obviously, the strength of Be-Al-Ag-Co-Ge alloy is improved by the solution strengthening. Furthermore, a few Ag₃Al particles contribute to the strength of the Al phase. Be-Fe-Al ternary intermetallic compounds which can effectively reduce the negative effect of an impurity element Fe on the mechanical properties of Be-Al alloys are also found by XRD and EDS.

Keywords: Be-Al alloys; solution strengthening; investment casting; mechanical properties

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1 Introduction

Beryllium (Be) and Be alloys have many excellent properties as aerospace materials, such as low density, high elastic modulus, high stiffness, and low thermal expansion coefficient. However, Be is intrinsically brittle below 200 °C. It is necessary to decrease the essential brittleness of Be for many structural applications in the aerospace industry ^[1]. The beryllium-aluminium (Al) alloys take the advantages of the Al phase's ductility and the Be phase's high modulus ^[2, 3]. Commercial Be-Al alloys with the Be content from 5% to 80% are widely fabricated by investment casting ^[4]. As the solid solubility between Be and Al is very low, Be-Al alloys with properties substantially different from those of pure metals should be defined as a composite material

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when the Be content is below 65wt.%, in which discontinuous granular Be phase reinforces continuous Al phase, the matrix material ^[1]. A very low mutual solubility of the Be-Al alloy is the barrier to creating alloys with properties substantially different from those of pure metals. Adding elements in Be-Al alloys may lead to some changes in the mechanical properties ^[5]. Allowing for the basic principles of alloy strengthening, the elements need to have relatively high solid solubility or be capable of forming intermetallic compounds in Be-Al alloys. Ag, Cr, Cu, Hf, Li, Sc, Zr, and Mg are likely to experience a noticeable solubility in Al; meanwhile, Au, Cu, Fe, Co, Ni, and Pd have a noticeable solubility in Be^[5, 6]. Many researchers have investigated the effect of adding elements on the properties of Be-Al alloy [7-11]. When Ag element is added, the electric charges are redistributed, which increases the density of electric charges across the interface. As a result, the bond between Be and Al that can strengthen the combination of the Be-Al interface is enhanced by the Ag element ^[7]. The Al alloy with up to 55wt.% Ag can be held at an elevated temperature and all of the Ag

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will be in solid solution ^[12, 13], what is more, it is investigated that the Ag_2A1 was formed due to the reaction between Ag and Al with prolonged exposure to elevated temperatures ^[12]. In the previous study, the Ag-rich precipitation strengthening phase in the Al matrix of Be-Al-Ag alloy could be found after heat treatment ^[14-16].

Cobalt (Co) is a stabilizing element of body-centered cubic Be with a maximum solubility of 28wt.%^[1]. Consequently, Co is selected as a Be phase strengthening element^[6]. It is found that the Be phase can be strengthened by Co addition from ~0.1wt.% to 0.75wt.%, and the yield strength of the alloys increased to 25% without a real effect on the ductility^[15].

Germanium (Ge) segregated to the Al matrix improves the mechanical properties of the Be-Al alloy at levels of 0.2wt.% and 5.0wt.% ^[16]. Moreover, the interaction between Ag and Ge in Be-Al alloy can eliminate the dimensional distortion before and after heat treatment ^[16, 17].

In this study, Be-35wt.% Al alloys with Ag, Ge, and Co additions were studied, with an aim of confirming the significantly promoted mechanical properties via solution strengthening. The microstructural evolution was characterized to determine the effect of Ag, Ge and Co additions on microstructure and properties. The strengthening mechanism at room temperature was discussed. These findings will help to understand the Ag, Ge, and Co microalloying mechanism and to develop a new fundamental framework for the material design of advanced Be-Al alloys.

2 Experiment

2.1 Sample preparation

The chemical compositions of Be-Al alloy and Be-Al-Ag-Co-Ge alloy were measured by wavelength dispersive fluorescence spectrometer (ARL PERFORM'X, Switzerland); results are listed in Table 1. The alloys were prepared using pure Be (99.00%), 1.5wt.% Ag (99.99%), 0.7wt.% Co (99.99%), 0.8wt.% Ge (99.99%), and pure Al (99.99%). These metals were abraded to remove superficial oxides, cleaned by sonication in acetone for 30 min, and dried for 60 min under 80 °C, then, melted together via the medium frequency induction melting furnace. The vacuum level in the furnace was kept from 10 Pa to 20 Pa. When the temperature rose to 1,180 °C (close to the melting point), the argon gas was charged through the furnace to prevent the Al elements from volatilization until the temperature rose up to 1,400±10 °C. The molten metal was poured into the Al₂O₃ ceramic shell mold after electromagnetic agitation for 20 min. The ceramic shell was obtained from the wax pattern, as shown in Fig. 1(a). The preheating temperature of the ceramic shell was (600±10) °C to ensure the filling process of molten liquid. When the flow filling process of the molten metal was finished, argon gas was applied as a cooling medium to accelerate the solidification process under 40-70 °C·s⁻¹ measured from an infrared thermometer in the cooling chambers, reducing segregation during the solidification process.

Table 1: Chemical	compositions	of investigated	d allovs	(wt.%)
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Alloy types	Composition	Ag	Co	Ge	AI	Fe	0	Be
Be-Al alloy	62Be-38AI	-	-	-	37.62	0.081	0.085	Bal.
Be-Al-Ag-Co-Ge alloy	62Be-35Al-1.5Ag- 0.8Ge-0.7Co	1.51	0.73	0.72	34.85	0.082	0.089	Bal.

2.2 Differential scanning calorimeter

The thermal analysis of Al (99.99% purity), Be (99.00% purity), Be-Al alloy, and Be-Al alloy with additions of Ag, Co, and Ge was measured by differential scanning calorimetry (DSC, STA449 NETZSCH, Germany) from room temperature to 1,400 °C with a heating rate of 20 °C·min⁻¹. The measurement accuracies of the temperatures and enthalpies were verified by the measurement with high-purity Al cylinders.

2.3 Microstructural characterizations

The X-ray diffraction patterns (XRD, Rigaku Smart lab, 9 kW) of mirror-polished specimens were obtained with the angle of 2θ from 20° to 90° at a scanning rate of $0.05^{\circ} \cdot s^{-1}$ using Cu K α radiation. The distribution of elements in the Be-Al-Ag-Co-Ge alloy was studied by scanning electron microscopy (SEM, ZEISS SUPRA55, Germany) equipped with an energy dispersive spectrometer (EDS, X-Max-N, OXFORD, England).

The 3 dimensional morphology and the volume fraction of the Be phase of Be-Al alloy were analyzed by Xradia Context Micro CT (CARL ZEISS, Germany).

2.4 Measurements of mechanical properties

Uniaxial tensile tests were performed using an Instrontype tester 4505 at the tensile rate of 0.2 mm·min⁻¹ at room temperature. The gauge length of the specimens is 25 mm, the diameter is 6 mm, as shown in Fig. 1(b). The value was the average of 3 measurements. The Vickers hardness of the Be phase and Al phase was determined under a load of 100 g for 10 s, and the value was the average of 5 points.

3 Results and discussion

3.1 Differential scanning calorimetry

The theoretical melting point of pure Be is $1,289 \, ^{\circ}C^{[18]}$, almost twice as much as pure Al, which is only 660 $^{\circ}C$. The



Fig. 1: Three-dimensional model of wax pattern (a), and samples for mechanical properties test (b)

DSC cooling curves of pure Al, pure Be, Be-Al alloy, and Be-Al-Ag-Co-Ge alloy are shown in Fig. 2. There is only one exothermic peak in the solidification process of pure Al and pure Be. The nucleation process is initiated from T_n and finished at T_{re} , as shown in Fig. 2. In particular, the nucleation process of Be is initiated from 1,256 °C because of impurity elements, the nucleation process of Al begins at 656 °C, and the latent heat of solidification ΔH of Be and Al is –614.5 J·g⁻¹ and –341.7 J·g⁻¹, respectively, obtained directly from Fig. 2.

The T_n of the Al in Be-Al alloy is 642 °C measured by DSC, which is close to 644 °C from the Al-Be phase diagram ^[1, 2]. The T_n of Al in Be-Al-Ag-Co-Ge alloy in Fig. 2 is 630 °C, caused by the dispersion of Ge-containing components in Al ^[19]. While, the effect of Ag and Co on T_n of Al in Be-Al-Ag-Co-Ge alloy is negligible ^[20, 21].

The solid solubility of Ag in Be is very small when the temperature is below 1,010 °C ^[22]. Moreover, there does not seem to be any solid solubility between Ge and Be. The T_n of the Be phase in Be-Al-Ag-Co-Ge alloy is not affected by Ag and Ge. The effect of adding an element Co on the T_n of the Be phase in Be-Al-Ag-Co-Ge alloy is also negligible from Be-Co



Fig. 2: DSC cooling curves of pure Be, pure Al, Be-Al alloy, and Be-Al-Ag-Co-Ge alloy

phase diagram ^[1], which is consistent with the testing result in Fig. 2 by DSC cooling curves.

3.2 X-ray diffraction

Figure 3 shows the XRD patterns of the alloys. The Be and Al diffraction peaks are both observed in Be-Al alloy and Be-Al-Ag-Co-Ge alloy. In addition, the intermetallic compound Ag₃Al is found in Be-Al-Ag-Co-Ge alloy. Ge and Ag both have the same face-centered cubic structure as Al. Co is solidly soluble in the Be phase ^[1], and Ge is solidly soluble in the Al phase ^[19]. Part of the Ag is solidly dissolved in the Al phase ^[13], and the other part of Ag may exist as intermetallic compounds Ag₃Al from XRD to reinforce Al matrix due to the precipitation strengthening. Furthermore, ternary intermetallic compounds are formed as AlFeBe₄ and Al₂FeBe₃ from Fig. 3. The Al₂FeBe₃, whose peaks are at 2θ =40.76°, 65.29°, and 78.30°, is found in both Be-Al alloy and Be-Al-Ag-Co-Ge alloy. The diffraction peaks of AlFeBe₄ are at 2θ = 52.55°, 69.58°, moreover, AlFeBe₄ is only found in the Be-Al alloy. The composition and distribution of the ternary intermetallic compounds are related to the distribution and content of Fe, which is from the Be material. When the temperature range is between 650 °C and 850 °C during solidification process, Fe precipitates out of Be, then the following reaction occurs as shown in Eq. (1). This is a process of diffusion control to form the ternary intermetallic compound, which can effectively



Fig. 3: XRD patterns of Be-Al alloy and Be-Al-Ag-Co-Ge alloy

reduce the negative effect of free elements Fe and Al occurring in the grain boundaries on the mechanical properties of Be-Al alloys ^[23].

$$Al+Fe+Be \rightarrow AlFeBe_4(Al_2FeBe_3)$$
(1)

3.3 Microstructure

The special structure shown in Fig. 4 is the three-dimensional network between Be and Al, in which Be (62wt.%) as strengthening phase is grey, and continuous Al phase (38wt.%) as matrix phase is red. The volume fraction of Be phase is 67.36% by the direct measurement.



Fig. 4: Three-dimensional network between Be and Al of Be-Al alloy

The microstructure and the element distributions of Be-Al-Ag-Co-Ge alloy were characterized by SEM and EDS, respectively. The Be as the reinforced phase is columnar dendritic, as shown in Figs. 5(a) and (b), and the Al is a continuous phase, as shown in Fig. 5(c). The Co element as shown in Fig. 5(e) is solidly soluble in Be element. The Goldschmid atomic radius [24] of Co and Be are 0.125 nm and 0.111 nm, respectively. The Goldschmid atomic radius difference Δr is less than 15% between Co and Be. Furthermore, Co and Be both are close-packed hexagonal crystal structure; as a result, Co can be solidly soluble in the Be phase. The Goldschmid atomic radius of Ag and Al atoms are very close to each other, 0.144 nm and 0.143 nm ^[24], respectively. The Δr is also less than 15% between Ag and Al. Similarly, the same situation occurs in Ge and Al, as the Goldschmid atomic radius of Ge is 0.140 nm [24]. It is confirmed that the Ag and Ge elements are solidly soluble in Al phase. The distribution of Ge and Ag elements is consistent with the Al as shown in Figs. 5(f) and (g). The smaller the Δr , the smaller the distortion degree. This is the reason why the XRD diffraction peaks of Al and Be shown in Fig. 3 do not significantly shift after adding trace elements.

The interface between Be and Al phases can be distinctly observed in Fig. 6. There is not a new compound formed between the Be and Al phases ^[25]. The reason is that the solid solubility of Be in Al phase is 0.1 wt.%, and the solid solubility



Fig. 5: SEM images of microstructure (a) and distribution of elements (b-g) of Be-Al-Ag-Co-Ge alloy

of Al in Be phase is only 0.02wt.% ^[26]. The surface energy of a binary Be-Al alloy calculated from a regular solution model using mean-field approximation statistics ^[25] is expressed as

$$\lambda = ZN_{a} \left[\varepsilon_{\text{BeAl}} - (\varepsilon_{\text{BeBe}} + \varepsilon_{\text{AlAl}})/2 \right]$$
(2)

where Z is the mean coordination number in the liquid, $N_{\rm a}$ is



Fig. 6: SEM image of the interface of Be-Al alloy

the Avogadro's number, and $\varepsilon_{\text{BeAI}}$ is the interaction energy of a Be-Al pair. The exchange energy, λ , is 25 kJ·mol⁻¹^[27]. The heats of evaporation for Be $(-ZN_a\varepsilon_{\text{BeBe}}/2)^{[25]}$ and Al $(-ZN_a\varepsilon_{\text{AIAI}}/2)^{[28]}$ are 297 kJ·mol⁻¹ and 294 kJ·mol⁻¹, respectively. Thus, the value of heats of evaporation for Be-Al $(-ZN_a\varepsilon_{\text{BeAI}}/2)$ is only 283 kJ·mol⁻¹ calculated by Eq. (2), which is lower than 297 kJ·mol⁻¹ (Be) and 294 kJ·mol⁻¹ (Al), indicating that strong interactions between Be and Al cannot happen.

Figure 7(a) shows the microstructure of Be-Al-Ag-Co-Ge alloy, in which Be phase is discontinuous particles in the Al matrix. The shrinkage cavities are found in the Al phase and phase boundaries between the Al phase and Be phase. Elemental analysis result in the red colored region by EDS is shown in Fig. 7(b). It is found that the Al₂FeBe₃ is most likely to be formed in the area with the similar weight ratios among Fe, Be, and Al, corresponding to the XRD results in Fig. 3.



Fig. 7: SEM image (a) and analysis of elements in one point marked in red (b) of Be-Al-Ag-Co-Ge alloy

3.4 Mechanical properties

Figure 8 shows the stress-strain curves of one group among the three Be-Al and Be-Al-Ag-Co-Ge alloys samples. The mechanical properties obtained by taking the average value of three groups of Be-Al and Be-Al-Ag-Co-Ge alloys samples are shown in Table 2. The Be-Al alloy exhibits relatively mediocre mechanical properties with a yield strength (YS) of 118.4±10.6 MPa, an ultimate tensile strength (UTS) of 182.0 ± 2.2 MPa, and an elongation (EL) of $(5.0\pm1.0)\%$. However, the mechanical properties of Be-Al-Ag-Co-Ge alloy are significantly improved. The YS, and UTS are increased to 193.0±3.8 MPa and 255.9±1.7 MPa, respectively, but the EL decreases to (4.4 ± 0.5) %. The corresponding UTS×EL value ^[30] of Be-Al-Ag-Co-Ge alloy reaches 1.19 GPa·%, which is much higher than that of Be-Al alloy (0.97 GPa·%). Accordingly, the additions of Ag, Co, and Ge improve not only strength, but also toughness.

Fracture behavior is tightly related to the microstructures of fracture surface. The microstructures of the fracture surface in Be-Al alloy and Be-Al-Ag-Co-Ge alloy are shown in Figs. 9(a) and (b), respectively. Importantly, no separation and de-bonding of the Be and Al phases are observed. One of the fracture morphologies [Fig. 9(a)] is the brittle zone surrounded



Fig. 8: Engineering stress-strain curves of one group among the three Be-Al alloys samples

by smooth tearing edges in Be phase with different cleavage steps and smooth cleavage planes ^[5]. The other [Fig. 9(b)] is a ductile fracture zone consisting of shallow dimples in Al phase.

The mechanical properties of pure Be and pure Al are very different. The discontinuous particle reinforcement model can be applied to Be-Al alloys^[29, 32]. Be is defined as discontinuous particles of Be-Al alloys^[32].

Samples	Yield strength (MPa)	Tensile strength (MPa)	Elongation (%)
Be-Al alloy	118.4±10.6	182.0±2.2	5.0±1.0
Be-Al-Ag-Co-Ge alloy	193.0±3.8	255.9±1.7	4.4±0.5
Be-Al alloy (Sc) [31]	110.7	179.7	2.9
Beralcast 191 alloy (Ag/Si) ^[17, 29]	158.5	213.6	2.2
Beralcast 363 alloy (Ag/Co/Ge) [17]	199.8	254.9	3.5

Table 2: Mechanical properties of Be-Al alloys

Note: Beralcast 191 alloy (Ag/Si) is 65Be-31Al-2Si-2Ag, Beralcast 363 alloy (Ag/Co/Ge) is 65Be-30.25Al-0.75Ge-3Ag-1Co.

At the initial stage of yielding, previous studies have shown that the stress caused by Be phase dislocation stacking at Be/Al interface can be easily released by the plastic deformation of Al phase ^[29]. Moreover, there is no fracture occurred in Al phase before Be phase is broken. Subsequently, the cleavage fracture occurs in Be phase when the stress of Be phase reaches the tensile strength of Be phase enhanced with Co ^[29]. The Co in Fig. 9(b) is solidly dissolved in the Be phase from the EDS results in Fig. 9(c). The Al phase reinforced by Ag and Ge in Fig. 9(d) suffers all the stress after the cleavage fracture of Be phase during the tensile process. As a result, the Al phase has a sharp plastic deformation, because the instantaneous stress is far greater than the ultimate tensile strength of Al phase ^[29].

The columnar Be grain and Al matrix receive a large load under tensile stress in the tensile test. The load is transferred from the softer Al matrix to the hard Be particles in the previous research ^[29, 32]. The composite yield strength σ_{cy} of the Be-Al alloy is controlled by the yield strength σ_{my} of Al matrix according to the following Eq. (3):

$$\sigma_{\rm cy} = \sigma_{\rm my} [0.5 V_{\rm Be} (S+2) + (1 - V_{\rm Be})] = \sigma_{\rm my} [0.5 V_{\rm Be} S+1]$$
(3)

where *S* is the reinforcement aspect ratio as a constant, V_{Be} is the volume fraction of the reinforcing Be phase (62wt.% Be) obtained by the statistics in Fig. 4, and σ_{my} is the yield strength of the Al matrix ^[32, 33]. The volume fraction of the Be is 67.36% of Be-Al alloy, which is close to the result from previous studies ^[34, 35]. The composite yield strength σ_{cy} is directly determined by the yield strength of Al alloy matrix σ_{my} ^[25] from Eq. (3). Therefore, increasing the yield strength of the Al matrix by Ag and Ge can increase the yield strength of composite material significantly, based on the results in Table 2.



Fig. 9: Fracture microstructure of Be-Al alloys: (a) Be-Al alloy; (b) Be-Al-Ag-Co-Ge alloy; (c)-(d) EDS results of Be phase and Al phase in Be-Al-Ag-Co-Ge alloy

Figure 10 shows that the Vickers hardness of Be phase in Be-Al alloy is 185.0 ± 2.9 HV. However, it is obvious that this Vickers hardness value dramatically increases to 209.8 ± 3.8 HV in the case of the Be-Al-Ag-Co-Ge alloy. Similarly, the hardness of Al phase is improved from 85.6 ± 2.5 HV in Be-Al alloy to 93.7 ± 1.5 HV in Be-Al-Ag-Co-Ge alloy which is strengthened by Ag, Ag₃Al and Ge. Moreover, the Vickers hardness of Be phase experiences a larger increase than those of the Al phase, showing that the former phase is more inclined to be strengthened by Co^[17].



Fig. 10: Vickers hardness of Be phase and Al phase in Be-Al alloys

4 Conclusions

The microstructure and mechanical properties of Be-Al alloy with additions of Ag, Co, and Ge fabricated by investment casting were investigated, and the following conclusions can be drawn:

(1) The Ge element in Be-Al alloy reduces the nucleation temperature of Al phase from 642 °C to 630 °C while the nucleation temperature of Be phase almost doesn't change.

(2) The Ag and Ge can be solidly dissolved in the Al phase to realize the solid solution strengthening, and Co can strengthen the Be phase to improve strength of Be-Al alloys. Likewise, the Vickers hardness of both Be phase and Al phase is also improved.

(3) The intermetallic compound Ag_3Al is found in the Be-Al-Ag-Co-Ge alloy to achieve precipitation strengthening. Meanwhile, the ternary phase $AlFeBe_4$ and Al_2FeBe_3 are found in Be-Al and Be-Al-Ag-Co-Ge alloys due to the presence of an impurity Fe element.

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