

Article

Influence of Ni on the Microstructures and Mechanical Properties of Heat-Treated Al-Cu-Ce-Mn-Zr Alloys

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Abstract: In order to enhance the high-temperature mechanical performance to meet service requirements, the microstructures and tensile properties of heat-treated Al-8.4Cu-2.3Ce-1.0Mn-0.2Zr-xNi (x = 0, 0.5, 1.0, 2.0, 4.0 wt.%) were investigated. The metallographic analysis techniques have been used to examine the microstructural changes with different Ni contents. Results show that after adding 0.5% Ni to the Al-8.4Cu-2.3Ce-1.0Mn-0.2Zr alloy, the spheroidized Al₇Cu₄Ni phase is formed. With Ni content further increasing, the Al₈CeCu₄ and Al₂₄MnCu₈Ce₃ phases disappear, and the nano-sized Al₂₀Cu₂Mn₃ and Al₂Cu phases decrease gradually. When Ni content reaches 4.0%, the Al₃CuNi phase appears. It turns out that the addition of 0.5% Ni has significantly improved the tensile properties at 400 °C. The ultimate tensile strength, yield strength, and elongation of Al-8.4Cu-2.3Ce-1.0Mn-0.2Zr-0.5Ni alloy at 400 °C reach 103 MPa, 93 MPa, and 18.0%, respectively, which makes the alloy possible to be employed at 400 °C. The intermetallic micro-skeleton, composed of thermostable Al₈CeCu₄, Al₂₄MnCu₈Ce₃, Al₁₆Cu₄Mn₂Ce, and Al₇Cu₄Ni phases at the grain boundaries as well as nano-sized Al₂₀Cu₂Mn₃ and Al₂Cu precipitates in the grains, contributes to the good elevated-temperature tensile strength. The fracture mechanism is changed from quasi-cleavage at ambient temperature to coexistence of quasi-cleavage and dimple at 400 °C.

Keywords: Al-Cu-Ce-Mn-Zr-(Ni) alloy; heat treatment; intermetallic compound; microstructure; mechanical property



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1. Introduction

Heat-resistant aluminum alloys have been widely applied in automobile and aircraft industries due to their superior strength-to-weight ratio, high elevated-temperature strength, excellent fatigue resistance, low coefficient of thermal expansion, and good wear resistance [1–4]. With the rapid development of industry and people's increasing awareness of the environment and resources, automotive engine efficiency has been improved constantly [5], which has placed greater demands on material performance. As a key component in the combustion chamber of engine, the piston is exposed to an increasingly high-temperature (300–400 °C) and high-pressure (~20 MPa) service environment [6,7]. However, the high-temperature resistance of existing heat-resistant cast aluminum alloys is approaching the limit (~350 °C), making them difficult to meet service requirements [8,9]. Compared with existing cast aluminum alloys, such as Al-Si-Cu-Ni-Mg [10,11], Al-Cu-Mn [12,13], Al-Cu-Ni [14], and Al-RE-Cu-Si [15], Al-Cu-RE-Mn-Zr alloys own higher

thermal strength above 300 °C [16]. The Al_2Cu and $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ phases in the matrix together with RE-containing intermetallic compounds at the grain boundaries play a crucial role in the good thermal strength of the alloys below 350 °C [17].

To further improve the high-temperature strength of the Al-Cu-Ce-Mn-Zr alloys and make them possible to be employed at 400 °C, a thermally stable microstructure is required. It is generally considered that the elevated-temperature strengthening effect of multicomponent aluminum alloys is attributed to the heat-resistant phases with excellent thermal stability, including Si, intermetallic compounds, and ceramic phases [18]. The micro-skeleton consisting of thermostable rigid reinforcing phases exhibits superior load carrying capability at high temperatures [19]. Ashgar et al. [20,21] found that when the volume fraction of rigid reinforcing phases in the cast Al-Si-Cu-Ni piston alloys increased by 25%, the yield strength of the alloys at 300 °C was improved by ~15%. The strength increased due to the fact that higher stress can be transferred from matrix to reinforcing phases [22]. Therefore, it is possible to enhance the elevated-temperature strength of Al-Cu-Ce-Mn-Zr alloys by introducing intermetallic compounds to improve the load-bearing capacity of the micro-skeleton.

Alloying is an easy and effective method to form intermetallic phases [23–35]. Adding Ni, promoting the formation of Ni-containing reinforcing phases including $\epsilon\text{-Al}_3\text{Ni}$, $\delta\text{-Al}_3\text{CuNi}$, and $\gamma\text{-Al}_7\text{Cu}_4\text{Ni}$, can improve the high-temperature performance of heat-resistant aluminum alloys [36]. Previous works [37–40] demonstrated that the heat resistance of aluminum alloys was remarkably improved after introducing Ni-containing intermetallic compounds. Among the three Ni-containing phases, the Al_3CuNi phase was found to have the most efficient volume utilization and possess the most effective contribution to the elevated-temperature strength. However, the evolution of Ni-rich phase is associated with the alloying elements. Yang et al. [41] concluded that with increasing Cu content in Al-Si-Cu-Ni-Mg piston alloys, the Ni-phase was transformed into short strip-like Al_3Ni , reticular-like Al_3CuNi , and annular-shaped or semi-annular-shaped $\text{Al}_7\text{Cu}_4\text{Ni}$ in turn. In addition, the Al-Cu-RE-Mn-Zr alloys usually undergo the T6 heat-treatment to obtain the nano-sized strengthening phases. Despite the thermal stable temperatures of Al_3Ni , Al_3CuNi , and $\text{Al}_7\text{Cu}_4\text{Ni}$ approaching 350–400 °C [42], the solution temperatures (>500 °C) of Al-Cu-Ce-Mn-Zr alloys are substantially higher than their thermal stable temperatures. Zuo et al. [22] found that the strip-shape Al_3CuNi was thermally stable at 350 °C in the piston alloy, while part of thin rod-shape Al_3CuNi is changed to spherical-shape $\text{Al}_7\text{Cu}_4\text{Ni}$ after thermal exposure at 350 °C for 200 h. Fernández-Gutiérrez et al. [43] reported that the solution treatment resulted in partial dissolution or spheroidization of intermetallic compounds. The loss of interconnectivity of the micro-skeleton reduced the load-bearing capability and decreased the creep resistance. Whether the spheroidization or phase transition of Ni-containing phases happens in the heat-treated Al-Cu-Ce-Mn-Zr alloys needs to be clarified.

Apart from the micro-skeleton at grain boundaries, the crystal structures and morphological features of the nano-sized dispersoids (particularly $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ and Al_2Cu) inside grains are also sensitive to the alloying elements. Wang et al. [44,45] discovered that denser precipitation of $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ or Al_2Cu strengthening phases occurred with 0.2 wt.% Ce addition in Al-Cu-Mn alloy, improving the tensile strength. Chen et al. [46] reported that after adding 1.0 wt.% Ce to the Al-Cu-Mn alloy, the formation of $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ phase was retarded, which is detrimental to the tensile strength. However, high-melting point Al_8CeCu_4 phase was formed with massive addition of Ce, which contributed to the good mechanical properties at elevated temperatures [17,47]. Besides, Sun et al. [48,49] discovered that Mn or Sc reduced the interfacial energy of θ' and lowered thermodynamic driving force for coarsening of θ' phase, which improved the high-temperature microstructure stability. Whether Ni influences the precipitation behavior of the dispersoids in grains also needs to be clarified.

As is known to all, the mechanical properties of multicomponent heat-resistant aluminum alloys mainly depend on the thermal stability and morphological features of

intermetallic compounds [50]. Little effort has been focused on the phase evolution after adding Ni and its effect on the ambient and elevated-temperature mechanical properties of heat-treated Al-Cu-Ce-Mn-Zr alloys. ZL206 alloys, the representative cast Al-Cu-Ce-Mn-Zr alloys, have already been used to manufacture aero-engine casings. In order to obtain sufficient strengthening phases, we chose the upper limit of the chemical composition range of alloying elements [16]. Hence, in this work, the microstructures of Al-8.4Cu-2.3Ce-1.0Mn-0.2Zr-xNi alloys were systematically studied by X-ray diffraction (XRD), optical microscope (OM), scanning electron microscope (SEM) equipped with energy dispersive spectrometer (EDS), and transmission electron microscope (TEM). Various characterizations were conducted to clarify the influence of Ni on the microstructural evolution and mechanical properties of heat-treated multicomponent Al-Cu-Ce-Mn-Zr alloys.

2. Materials and Methods

2.1. Alloys Preparation

The experimental alloys were prepared using 99.9% pure Al and 99.5% pure master alloys (Al-50%Cu, Al-10%Mn, Al-20%Ce, Al-5%Zr, Al-10%Ni). All compositions are given in weight percent unless otherwise specified. The alloys were melted using an electric resistance furnace. The melts were isothermally held at 760 °C for about 20 min and stirred well to ensure complete homogenization. Then, the melts were processed using 0.5% C₂Cl₆ at 740 °C for degassing and slag-removing. After 10 min, the 720 °C melts were finally poured into a pre-heated (300 °C) mold to obtain the casting ingots. The chemical compositions of alloys were measured by ICAP7400 inductively coupled plasma-atomic emission spectrometry as shown in Table 1. The alloys with different Ni contents were labelled as 0 Ni, 0.5 Ni, 1.0 Ni, 2.0 Ni, and 4.0 Ni, respectively.

Table 1. Chemical compositions of as-cast Al-8.4Cu-2.3Ce-1.0Mn-0.5Ni-0.2Zr-xNi alloys (weight percent).

Alloys	Cu	Ce	Mn	Ni	Zr	Al
0 Ni	8.46	2.29	0.98	0	0.21	Bal.
0.5 Ni	8.53	2.33	1.03	0.48	0.17	Bal.
1.0 Ni	8.45	2.27	1.02	1.06	0.22	Bal.
2.0 Ni	8.49	2.22	1.05	1.91	0.19	Bal.
4.0 Ni	8.34	2.31	0.96	3.95	0.18	Bal.

2.2. Heat Treatments

To obtain the phase transition temperatures, the thermal analysis tests were conducted by NETZSCH STA 449C differential scanning calorimetry (DSC) on about 5 mg samples cut from cast alloys under an argon atmosphere at a heating rate of 10 K·min^{−1}. The onset melting temperatures of the cast Al-Cu-Ce-Mn-Zr-(Ni) alloys are dependent on the low melting point eutectic mixtures. According to the results of heating curves in Figure 1, the onset melting temperatures of the five as-cast alloys are about 542 °C, 550 °C, 550 °C, 549 °C, and 550 °C, respectively. After adding Ni, the onset melting temperatures increase. According to the parameters of non-variant reactions in the aluminum corner of the Al-Cu-Ni and Al-Cu-Ce systems [1], the temperature of eutectic reaction (1) (546 °C) is higher than that of eutectic reaction (2) (541 °C), and thus, the Al₇Cu₄Ni may be precipitated before Al₈CeCu₄ during solidification. The prior precipitated Al₇Cu₄Ni consumes the alloying elements and can restrain the reaction (2). Accordingly, the lower melting point eutectic mixtures are reduced after Ni alloying. Compared with 0 Ni alloy, the additional endothermic peaks in the Ni-alloyed materials may be attributed to the melting of the Ni-containing phases. To avoid the overburning phenomenon and simultaneously ensure the precipitation strengthening effect, the T6 heat treatments were performed on the cast alloys by the KSL-1100X furnace. Overall, the experimental conditions for the alloys are provided in Table 2.



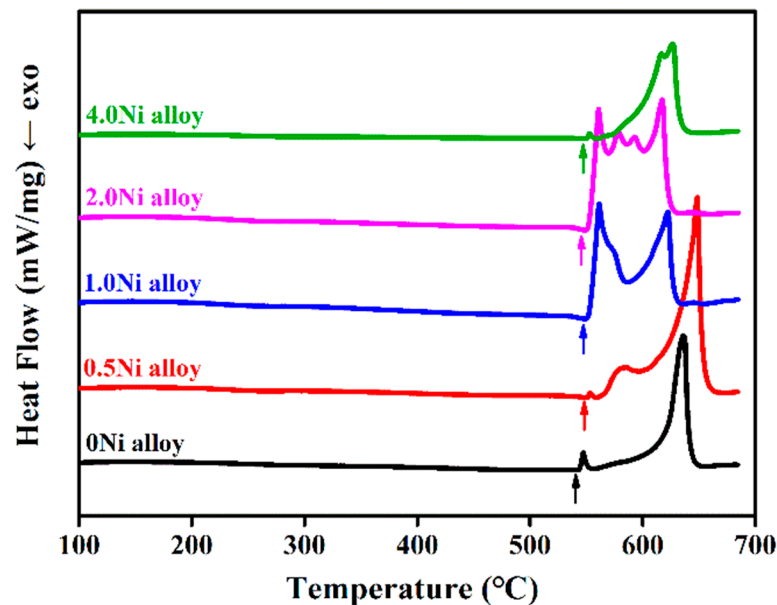
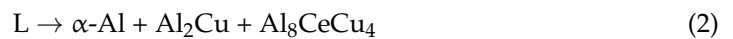


Figure 1. Heating curves of as-cast Al-8.4Cu-2.3Ce-1.0Mn-0.2Zr alloys with different Ni contents. The arrows with different colors indicate the onset melting temperatures of different alloys.

Table 2. Summary of heat treatments performed on the cast alloys.

Alloys	Solution Treatment	Quenching Treatment	Aging Treatment
0 Ni	535 °C/12 h		
0.5 Ni			175 °C/5 h
1.0 Ni	540 °C/12 h	50 °C water	+ air cooling
2.0 Ni			
4.0 Ni			

2.3. Microstructure Analysis

The X-ray diffraction studies were performed on a Bruker-AXS D8 Advance diffractometer with $\text{CuK}\alpha_1$ radiation to identify the phases in the alloys. The metallographic specimens were cut from the same position of the samples, and then mechanically ground and polished in standard routines. The experimental samples were etched using 5 mL HNO_3 + 5 mL HF + 90 mL distilled water for 5 s. After etching, the microstructures were observed using an Olympus GX-41 optical microscope. The Quant 250FEG scanning electron microscopy was used to illustrate microstructures and variations in the fracture behavior of the alloys. The point scanning analyses were performed on an energy dispersive X-ray spectroscopy measurement attached to SEM, operating at 30 kV. The FEI Tecnai G2 20 LaB6 transmission electron microscope was used to examine the thin foil specimens at 200 kV operating voltage to characterize the microstructures. TEM samples were electro-polished, in a 70% ethanol and 30% nitric acid solution at -30 °C, by twin-jet equipment operated at 20 V.

2.4. Mechanical Testing

The dog-bone-shaped tensile specimens with a gauge section of $\Phi 3 \times 15$ mm were prepared from the heat-treated ingots [51]. The tensile tests were performed at 25 °C, 350 °C, and 400 °C at a constant strain rate of about $5 \times 10^{-4} \text{ s}^{-1}$ on an Instron 5968 testing machine. In the high-temperature tests, the specimens were held at the test temperatures for about 20 min before loading to ensure the temperature homogenization across the samples. The mechanical results reported in this study are the mean values of three specimens.

3. Results

3.1. Microstructures of Heat-Treated Alloys

The typical optical microscope photos of heat-treated Al-8.4Cu-2.3Ce-1.0Mn-0.2Zr alloys with different Ni contents are shown in Figure 2. The intermetallic compounds with different morphologies segregate at the α -Al interdendritic regions and grain boundaries. These intermetallic phases tend to increase with Ni content.

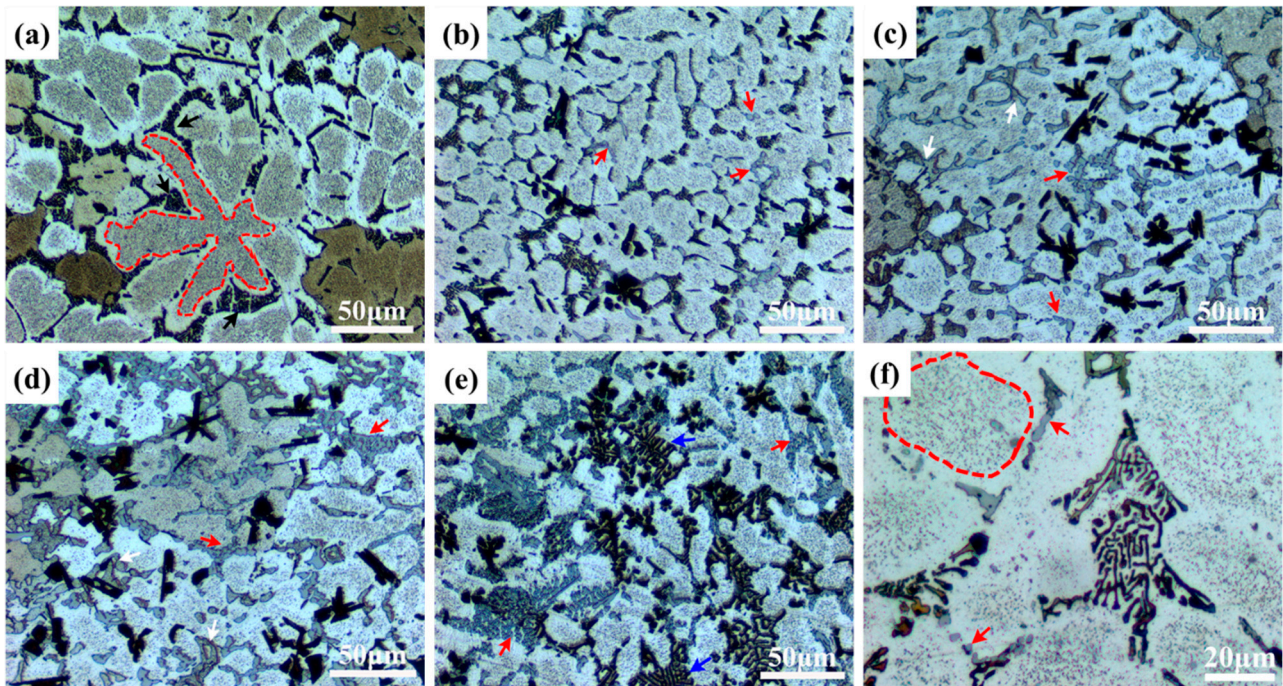


Figure 2. Optical microscope images of heat-treated alloys: (a) 0 Ni alloy, (b) 0.5 Ni alloy, (c) 1.0 Ni alloy, (d) 2.0 Ni alloy, (e) 4.0 Ni alloy, (f) magnified picture of 0.5 Ni alloy. The arrows with different colors indicate different phases.

In the 0 Ni alloy, plenty of black reticular-like phases (black arrows) appear at the interdendritic areas and grain boundaries, and a large number of dispersoids are uniformly distributed in the grains (in the area outlined by the red dotted line). These are consistent with the results of previous works [16,17,47]. In the 0.5 Ni alloy, the secondary dendrite arm spacing (SDAS) of α -Al is obviously reduced. The newly formed white skeleton-like phases (red arrows) are characterized by partial melting and spheroidization. With the increase of Ni content, the black reticular-like phases disappear in the 1.0 Ni and 2.0 Ni alloys, but the white skeleton-like phases (red arrows) and grey net-like phases (white arrows) seem to be broken and spheroidized, and they exhibit spherical chain shapes along the grain boundaries. In the 4.0 Ni alloy, the black coarse fishbone-like phases (blue arrows) were observed, which are similar to the Ni-containing phases in the literature [22].

Figure 3 demonstrates the XRD patterns of the heat-treated Al-8.4Cu-2.3Ce-1.0Mn-0.2Zr alloys with different Ni contents. From the XRD results, there are α -Al, Al_2Cu , $\text{Al}_{24}\text{MnCu}_8\text{Ce}_3$, and Al_8CeCu_4 phases in the 0 Ni alloy. The intermetallic compounds include the Al_2Cu phase with a tetragonal structure, the $\text{Al}_{24}\text{MnCu}_8\text{Ce}_3$ phase with a cubic structure, and the Al_8CeCu_4 phase with a tetragonal structure. However, whether the $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ phase with an orthorhombic structure exists is not very clear. This might be due to the low volume fraction of this phase in the matrix [17]. Compared with the 0 Ni alloy, the $\text{Al}_7\text{Cu}_4\text{Ni}$ phase with a rhombohedral structure appears in the 0.5 Ni alloy, which is consistent with the observed white skeleton-like phases by the optical microscope. With the increase of Ni content, the Al_8CeCu_4 phase disappears in the 1.0 Ni alloy and 2.0 Ni alloy, but the diffraction peak intensity of the $\text{Al}_7\text{Cu}_4\text{Ni}$ phase obviously increases. This is corresponding to the metallographic observations that the black reticular-like phases

disappear and the white skeleton-like phases coarsen. A new Ni-bearing Al_3CuNi phase with a hexagonal structure is formed in the 4.0 Ni alloy, which corresponds to the fact that the observed black fishbone-like precipitates. The SEM equipped with EDS was also used to identify and analyze the different intermetallic compounds in the alloys.

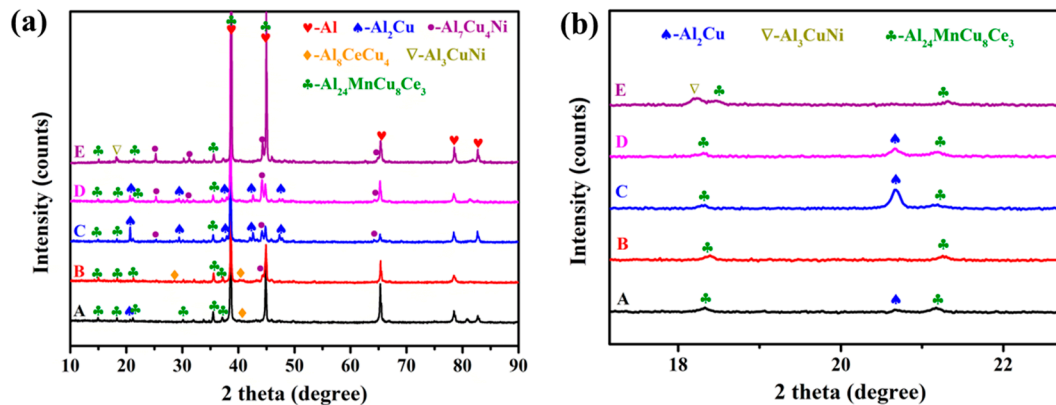


Figure 3. XRD patterns of heat-treated alloys. (b) illustrates the enlarged area of (a). A, B, C, D, and E represent the 0 Ni, 0.5 Ni, 1.0 Ni, 2.0 Ni, and 4.0 Ni alloys, respectively.

Figure 4 shows SEM micrographs of heat-treated $\text{Al-8.4Cu-2.3Ce-1.0Mn-0.2Zr}$ alloys with different Ni contents. The intermetallic phases are labelled and identified by EDS, and their specific compositions are given in Table 3. Four kinds of intermetallic compounds including reticular-like $\text{Al}_{24}\text{MnCu}_8\text{Ce}_3$ (Label 1), reticular-like Al_8CeCu_4 (Label 2), strip-like $\text{Al}_{16}\text{Cu}_4\text{Mn}_2\text{Ce}$ (Label 3), and sphere-like Al_2Cu (Label 4) were detected in the 0 Ni alloy [16,17]. However, apart from $\text{Al}_{24}\text{MnCu}_8\text{Ce}_3$ (Label 5), Al_8CeCu_4 (Label 6), $\text{Al}_{16}\text{Cu}_4\text{Mn}_2\text{Ce}$ (Label 7), and Al_2Cu (Label 8) phases, the skeleton-like $\text{Al}_7\text{Cu}_4\text{Ni}$ (Label 9) phase is formed in the 0.5 Ni alloy. Compared with the as-cast alloys [52,53], the $\text{Al}_7\text{Cu}_4\text{Ni}$ phase is partially spheroidized or even broken after T6 heat treatment, while the Ce-rich intermetallic phases show slight spheroidization. Given that the solution treatment temperature ($>500^\circ\text{C}$) is higher than the thermal stable temperatures of these intermetallics ($350\text{--}400^\circ\text{C}$), this phenomenon occurs. The intermetallics with different morphologies form a nearly continuous network structure along the grain boundaries and interdendritic areas.

Table 3. Compositions of the phases in Figure 4 identified by EDS.

Position	Elements (at.%)						Possible Phase
	Al	Cu	Mn	Ce	Ni	Zr	
1	76.1	17.9	1.2	4.7	0	0.1	$\text{Al}_{24}\text{MnCu}_8\text{Ce}_3$
2	88.4	9.1	0.2	2.3	0	0	Al_8CeCu_4
3	81.0	12.0	4.5	2.4	0	0.1	$\text{Al}_{16}\text{Cu}_4\text{Mn}_2\text{Ce}$
4	73.4	26.2	0.3	0.1	0	0.1	Al_2Cu
5	86.7	10.4	0.6	2.3	0	0	$\text{Al}_{24}\text{MnCu}_8\text{Ce}_3$
6	88.1	9.2	0.3	2.1	0.3	0	Al_8CeCu_4
7	62.5	22.1	9.4	5.6	0.4	0	$\text{Al}_{16}\text{Cu}_4\text{Mn}_2\text{Ce}$
8	71.3	28.3	0.2	0.1	0	0.1	Al_2Cu
9	84.0	12.0	0	0	4.0	0	$\text{Al}_7\text{Cu}_4\text{Ni}$
10	81.7	10.6	4.7	2.6	0.4	0	$\text{Al}_{16}\text{Cu}_4\text{Mn}_2\text{Ce}$
11	84.9	14.7	0	0	0.4	0	Al_2Cu
12	71.1	23.1	0.5	0.7	4.6	0	$\text{Al}_7\text{Cu}_4\text{Ni}$
13	70.7	16.8	8.4	4.1	0	0	$\text{Al}_{16}\text{Cu}_4\text{Mn}_2\text{Ce}$
14	87.5	12.1	0.1	0.1	0.2	0	Al_2Cu
15	89.3	8.3	0	0	2.4	0	$\text{Al}_7\text{Cu}_4\text{Ni}$
16	90.8	1.6	0.8	0	6.8	0	Al_3CuNi
17	73.7	16.2	6.4	3.3	0.3	0.1	$\text{Al}_{16}\text{Cu}_4\text{Mn}_2\text{Ce}$
18	89.7	6.7	0.2	0.5	2.8	0.1	$\text{Al}_7\text{Cu}_4\text{Ni}$

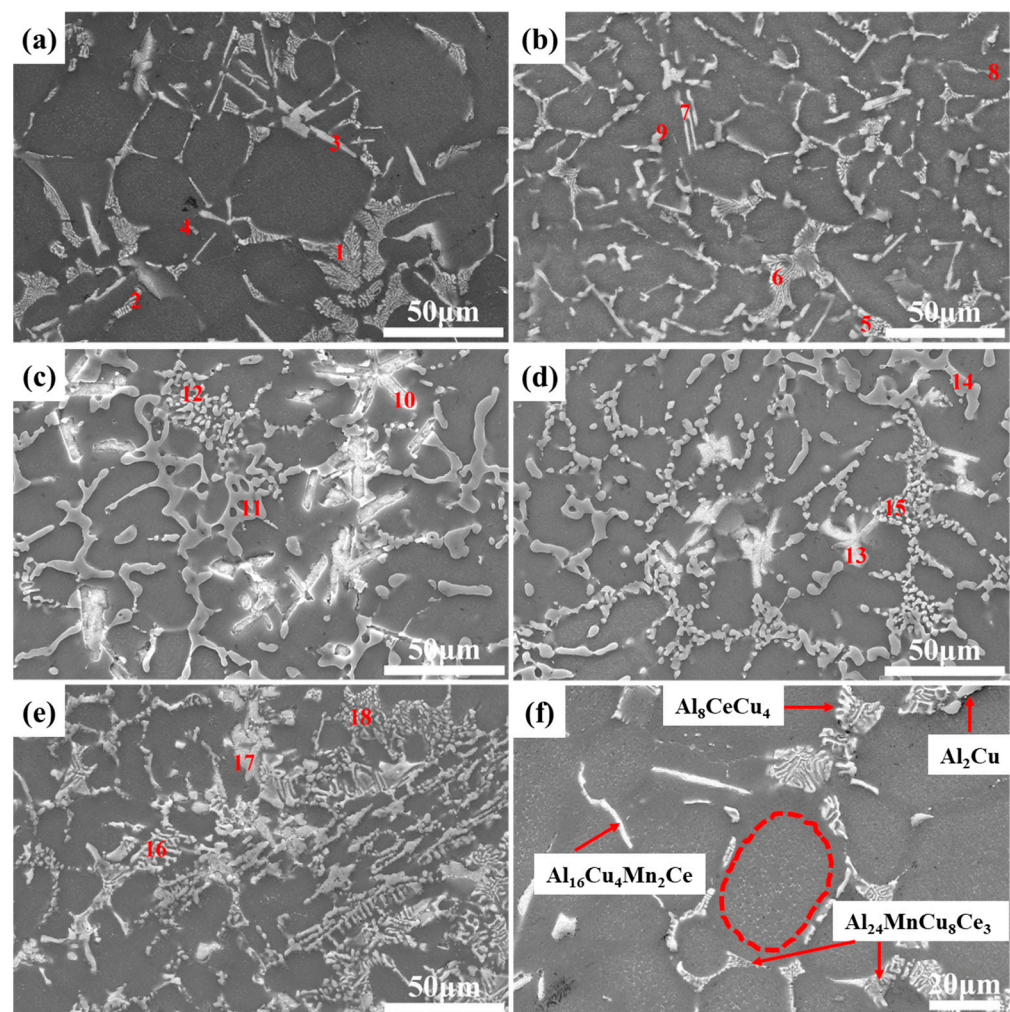


Figure 4. SEM images of heat-treated alloys: (a) 0 Ni alloy, (b) 0.5 Ni alloy, (c) 1.0 Ni alloy, (d) 2.0 Ni alloy, (e) 4.0 Ni alloy, (f) the nano-phases inside grains in the 0 Ni alloy.

In the 1.0 Ni alloy and 2.0 Ni alloy, the coarse blocky-like $\text{Al}_{16}\text{Cu}_4\text{Mn}_2\text{Ce}$ phase appears (Label 10 and 13), but the thermally stable Al_8CeCu_4 and $\text{Al}_{24}\text{MnCu}_8\text{Ce}_3$ phases disappear. It is worth noting that the Al_2Cu phase at the grain boundaries is not completely dissolved into the matrix after heat treatments, and the remaining part is spheroidized and transformed into isolated sphere shape or network shape with smooth surfaces (Label 11 and 14). Moreover, the $\text{Al}_7\text{Cu}_4\text{Ni}$ phase is fragmentized into small spheres with $\sim 5\ \mu\text{m}$ diameter (Label 12 and 15). In the 4.0 Ni alloy, the newly precipitated fishbone-like Al_3CuNi (Label 16) together with $\text{Al}_{16}\text{Cu}_4\text{Mn}_2\text{Ce}$ (Label 17) and $\text{Al}_7\text{Cu}_4\text{Ni}$ (Label 18) forms a coarse network skeleton, which can dis sever matrix. It should be noted that fine phases appear inside the grains as shown in Figure 4, which will be identified and analyzed by TEM in the following.

The TEM images and diffraction patterns of $\text{Al-8.4Cu-2.3Ce-1.0Mn-0.2Zr}$ alloys with different Ni contents are shown in Figure 5. According to the results of selected area electron diffraction patterns parallel to $\langle 110 \rangle$ $\alpha\text{-Al}$ zone axis and previous research results [54–56], the $\text{T-Al}_{20}\text{Cu}_2\text{Mn}_3$ and $\theta'\text{-Al}_2\text{Cu}$ nano-phases were found in the five heat-treated alloys. The coarser rod-shaped phase with 100–2000 nm length and $\leq 300\ \text{nm}$ thickness is $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ as shown in Figure 5a, c, e, g, i, whose long axis direction is parallel to $[100]_{\alpha\text{-Al}}$. The finer needle-phase with 10–200 nm length and $\leq 10\ \text{nm}$ thickness is $\theta'\text{-Al}_2\text{Cu}$, as shown in Figure 5b, d, f, h, j, whose long axis direction is also parallel to $[100]_{\alpha\text{-Al}}$. The sizes and volume fractions of the two nano-sized precipitates display no obvious change with addition of Ni under 1.0%. Otherwise, the number density of the two nanophases decreases significantly with addition of Ni above 1.0%. According to the non-variant reactions in the aluminum

corner of the Al-Cu-Ni and Al-Cu-Mn systems [1], the reaction temperature of reaction (3) (590 °C) is higher than that of eutectic reaction (4) (547 °C). Consequently, the Ni-bearing Al_3CuNi and $\text{Al}_7\text{Cu}_4\text{Ni}$ phases are precipitated earlier than Al_2Cu and $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ phases during solidification.

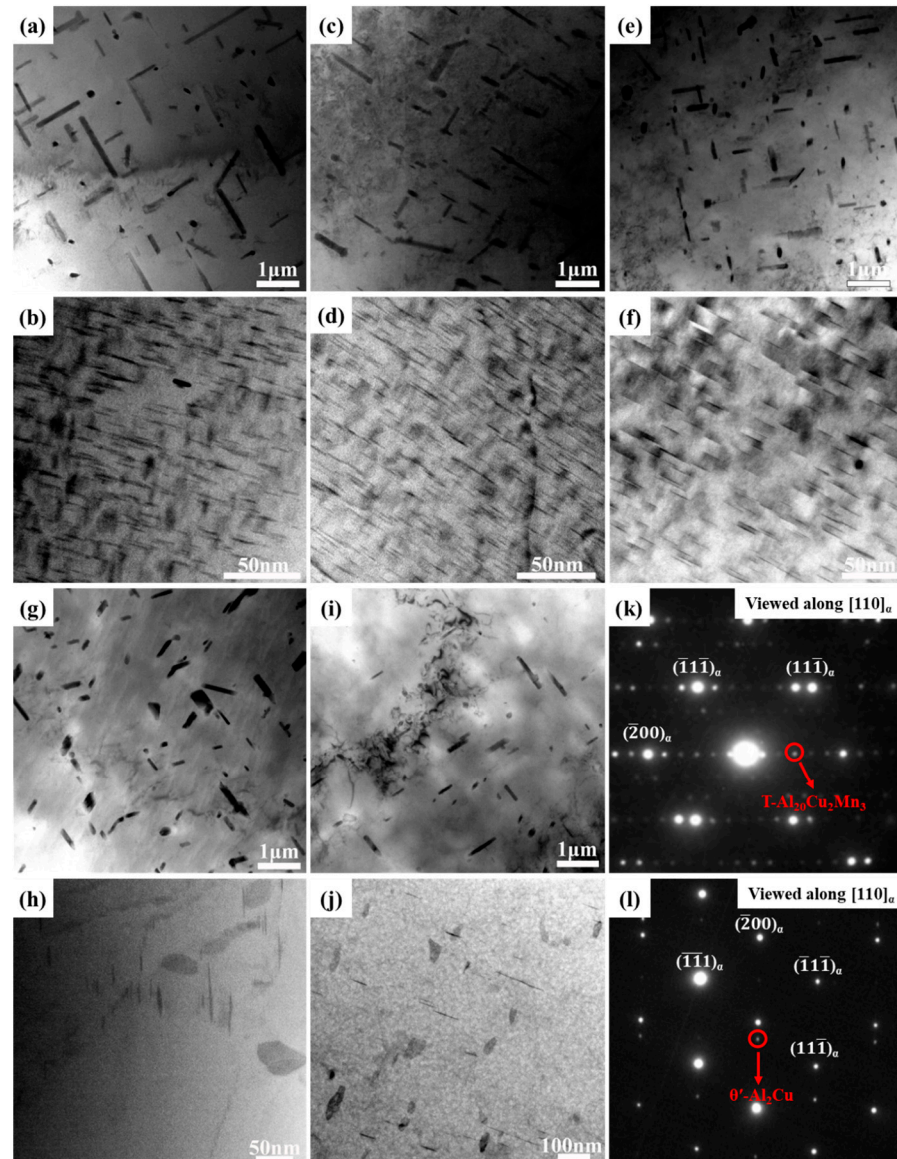
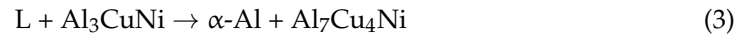


Figure 5. TEM images of nano-phases in heat-treated alloys: (a,b) 0 Ni alloy, (c,d) 0.5 Ni alloy, (e,f) 1.0 Ni alloy, (g,h) 2.0 Ni alloy, (i,j) 4.0 Ni alloy, (k) diffraction pattern of $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ viewed along $[110]_{\alpha\text{-Al}}$, (l) diffraction pattern of Al_2Cu viewed along $[110]_{\alpha\text{-Al}}$, (a,c,e,g,i) microstructures of $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$, (b,d,f,h,j) microstructures of Al_2Cu .

The massive addition of Ni may lead to the prior precipitated Ni-bearing phases consuming the alloying elements and depressing the formation of Al_2Cu and $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ [42].

3.2. Mechanical Properties of Heat-Treated Alloys

Figure 6a shows the effect of Ni content on engineering stress-strain curves of heat-treated Al-8.4Cu-2.3Ce-1.0Mn-0.2Zr alloys at 25 °C, 350 °C, and 400 °C, respectively. The results of the tensile properties (i.e., ultimate tensile strength, yield strength, and elongation)

at room and high temperatures, based on three samples for each chemical composition, are summarized in Figure 6b–d. There is a general tendency towards a decrease in ultimate tensile strength (UTS) and yield strength (YS) and an increase in elongation (EL) with increasing temperature. When the temperature increases, the easy dislocation movement causes the reduction of strength due to the domination of thermally activated cross slip, and the matrix softening causes the increase in elongation [57].

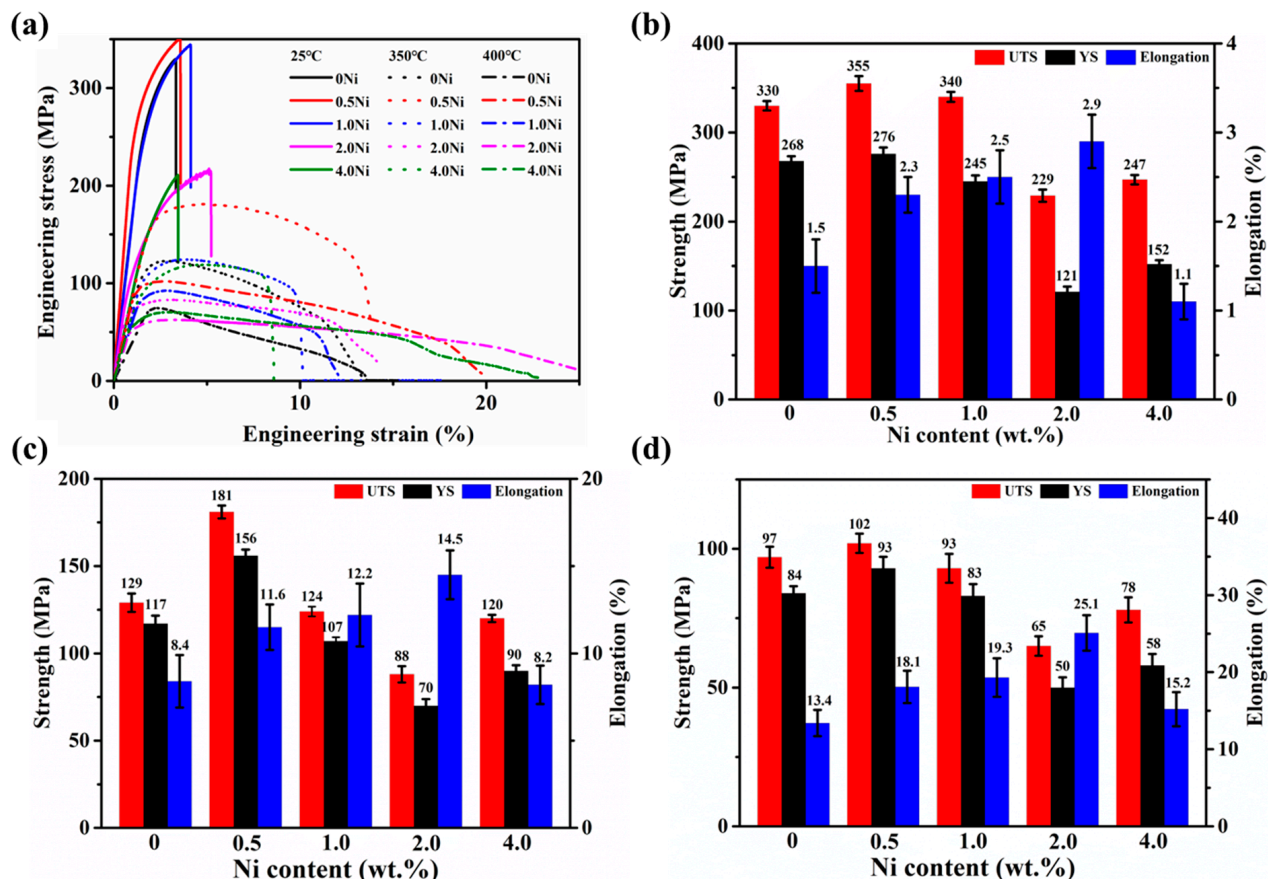


Figure 6. The tensile properties of the heat-treated alloys at different temperatures: (a) the engineering stress-strain curves at different temperatures, (b) 25 °C, (c) 350 °C, (d) 400 °C.

It turns out that adding 0.5% Ni to the alloy can improve the mechanical properties. With the increase of Ni contents, the UTS and YS decrease firstly and then increase, while the EL changes in reverse. The optimal elevated-temperature tensile properties are obtained in the 0.5 Ni alloy. The UTS and YS of the 0.5 Ni alloy at 350 °C reach 181 MPa and 156 MPa, which are ~40% and ~33% higher than that of the 0 Ni alloy. Especially, the 0.5 Ni alloy still possesses a high tensile yield strength of 93 MPa at 400 °C. The yield strength of existing casting aluminum alloys (RR350, ZL208) at their maximum service temperature (350 °C) is about 95 MPa [16,58,59]. By contrast, the 0.5 Ni alloy can maintain this strength up to 400 °C, which exhibits significant potential to raise the service temperature by 50 °C. Therefore, this alloy can be used at 350–400 °C to meet the requirements of advanced engines.

3.3. Fracture Surfaces Characterizations

To better understand the failure modes of the alloys at ambient and elevated temperatures, the fracture surfaces of the testing alloys were examined by SEM. Figure 7 illustrates the fractographs of the tensile specimens and the corresponding fracture surfaces depending on the Ni content at 25 °C and 400 °C.

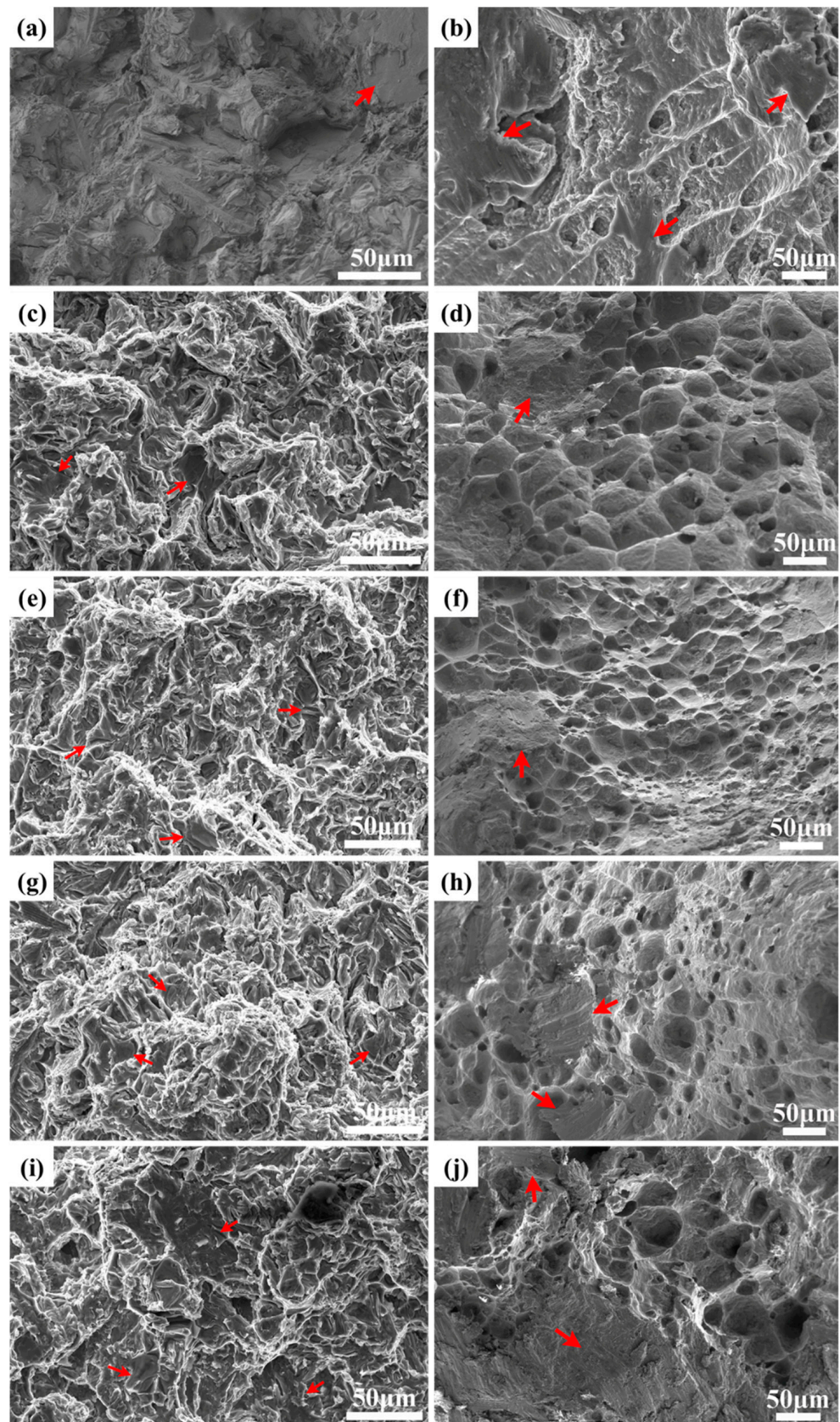


Figure 7. SEM images of fractured surfaces on the tensile specimens of heat-treated alloys at 25 °C and 400 °C: (a,b) 0 Ni alloy, (c,d) 0.5 Ni alloy, (e,f) 1.0 Ni alloy, (g,h) 2.0 Ni alloy, (i,j) 4.0 Ni alloy, (a,c,e,g,i) fractographs at 25 °C, (b,d,f,h,j) fractographs at 400 °C. The red arrows indicate the cleavage planes.

A quasi-cleavage fracture is between a cleavage fracture and a dimple fracture. The cleavage crack nucleus is generated at different parts, and then expands into cleavage facets, which are connected by many tearing edges [60]. The failure of the five alloys is dominated by quasi-cleavage fracture at ambient temperature. It can be observed that the fracture surfaces exhibit many cleavage planes (red arrows) separated by tearing ridges at 25 °C. The cleavage planes, originating from the crystals, breaking strictly along a certain direction under external force, are formed by the fracture of the intermetallic compounds at the interdendritic regions and grain boundaries. The tearing ridges, resulting from local plastic deformation, are produced by the deformed and broken α -Al. It should be noted that the cleavage planes are obviously smaller after adding 0.5% Ni, which is attributed to the fine spheroidized $\text{Al}_7\text{Cu}_4\text{Ni}$ phase substituting for coarse $\text{Al}_{24}\text{MnCu}_8\text{Ce}_3$. However, the coarse $\text{Al}_{16}\text{Cu}_4\text{Mn}_2\text{Ce}$ and Al_3CuNi phases are broken during deformation in the 4.0 Ni alloy, and thus, the size of cleavage planes substantially increases, which is consistent with the reduction in elongation of the alloys varying from 2.0% Ni to 4.0% Ni.

Both cleavage planes and dimples are observed on the fracture surfaces at 400 °C. It indicates that α -Al undergoes a certain amount of plastic strain before fracture, which is consistent with the evolution of elongation to fracture. The evolution of fracture surface morphologies reveals that the fracture mode gradually changes from brittle fracture to a mixed form of brittle and ductile fracture with the increasing of temperature. In addition, the area ratio of the cleavage planes is increased with the appearance of Al_3CuNi , which confirms that the coarse Ni-containing intermetallic phases result in the decreased elongation of the 4.0 Ni alloy.

Longitudinal sections normal to the fracture surfaces of the tensile-tested samples were examined to analyze the crack propagation modes beneath the fracture surface. Figures 8 and 9 show the microstructures near the fracture surface of the samples sectioned from the specimens tested at 25 °C and 400 °C. For the rupture process at room temperature, cracks initiate from intermetallic phases indicated by white arrows and pass through the intermetallic compounds indicated by black arrows. The coarse intermetallic phases in alloys generate a stress concentration in the surrounding matrix and provide easy crack initiation and propagation areas. As a result, many microcracks are observed in the 4.0 Ni alloy at ambient temperature. For the rupture process at 400 °C, the hard and brittle intermetallic phases are broken, and cracks are initiated. The cracks can easily propagate through the interdendritic areas and grain boundaries without restraint in the 0 Ni alloy. After adding Ni, the cracks have to pass through the intermetallics to continue propagating, as indicated by black arrows, which suggests that the intermetallic phases can slow down the propagation of cracks.

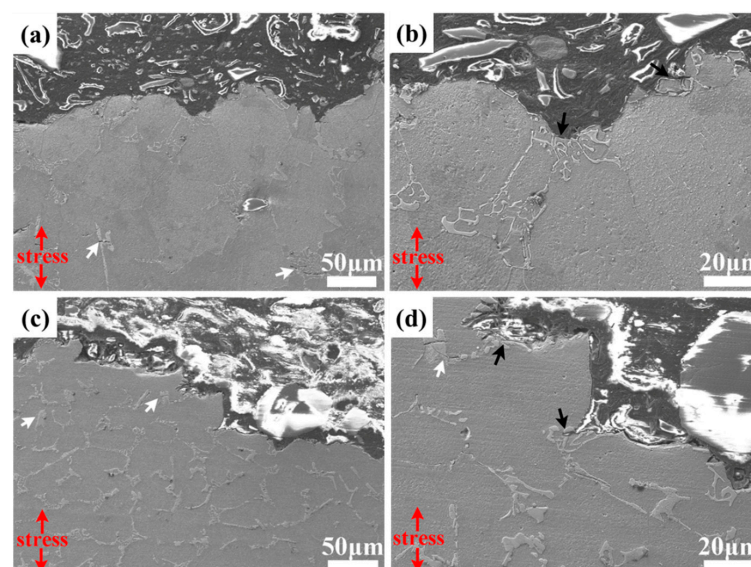


Figure 8. Cont.

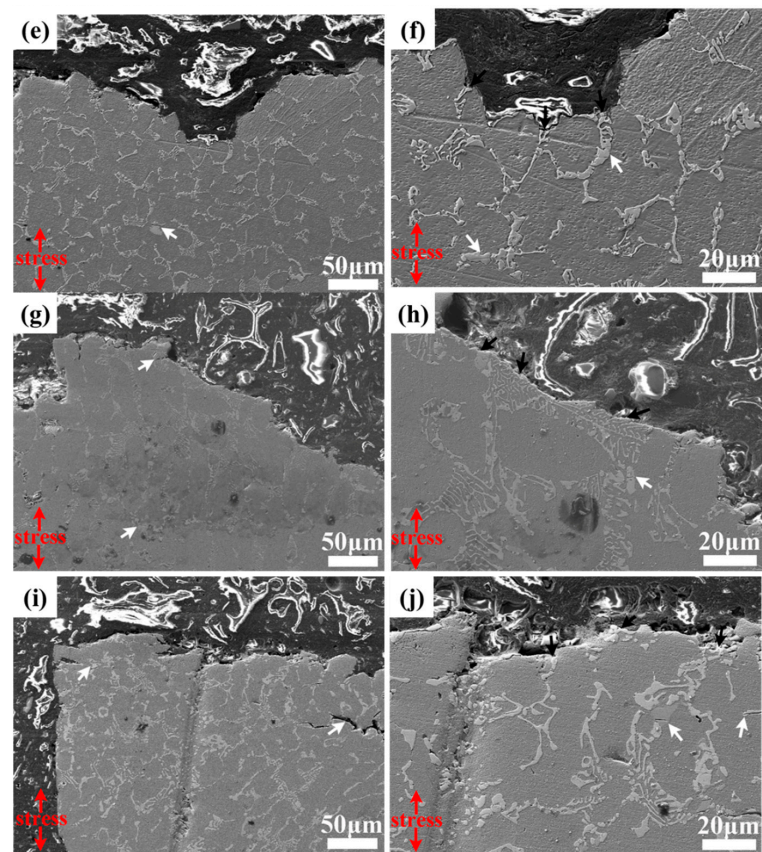


Figure 8. SEM microstructure beneath the fracture surfaces of the tensile specimens of heat-treated alloys tested at 25 °C: (a,b) 0 Ni alloy, (c,d) 0.5 Ni alloy, (e,f) 1.0 Ni alloy, (g,h) 2.0 Ni alloy, (i,j) 4.0 Ni alloy, (b,d,f,h,j) magnified pictures of (a,c,e,g,i), respectively. The white arrows indicate cracks in the intermetallic phases and the black arrows indicate the torn intermetallic phases.

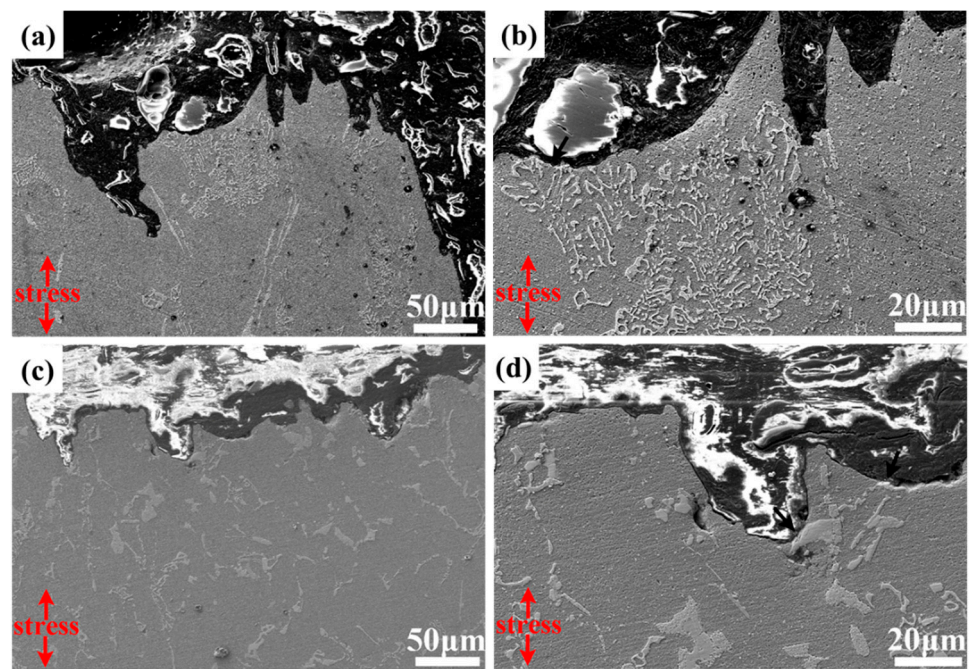


Figure 9. Cont.

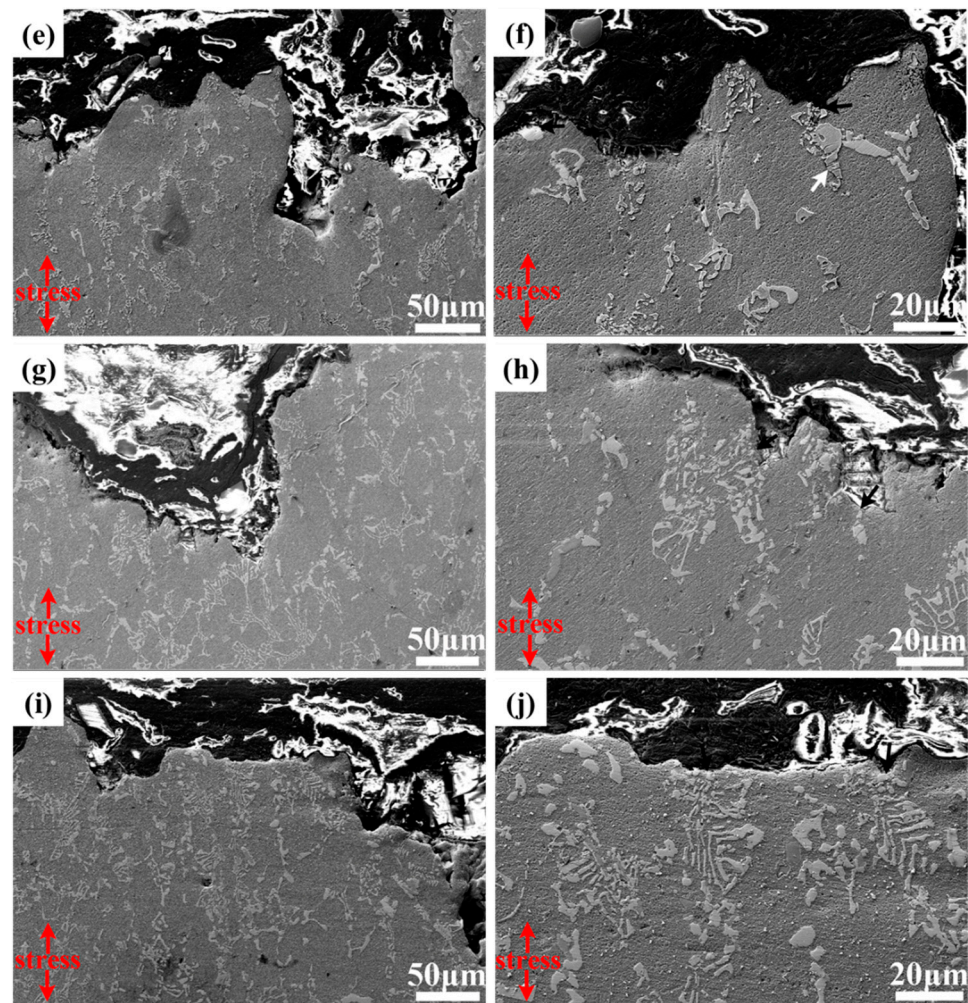


Figure 9. SEM microstructure beneath the fracture surfaces of the tensile specimens of heat-treated alloys tested at 400 °C: (a,b) 0 Ni alloy, (c,d) 0.5 Ni alloy, (e,f) 1.0 Ni alloy, (g,h) 2.0 Ni alloy, (i,j) 4.0 Ni alloy, (b,d,f,h,j) magnified pictures of (a,c,e,g,i), respectively. The white arrows indicate the torn intermetallic phases.

4. Discussion

It is well known that the characteristics of intermetallics (thermal stability, size, morphology, volume fraction, and distribution) play an important role in determining the mechanical properties of cast heat-resistant aluminum alloys. The mechanical properties are not only associated with the precipitated phases in the α -Al matrix, but also dependent on the intermetallic reinforcing phases at the grain boundaries [61]. The physical parameters of intermetallic compounds are summarized in Table 4.

Table 4. Summary of the physical parameters of phases in the alloys [38–41,62–67].

Phase	Composition (weight Percent)	Crystal Structure	Thermal Stable Temperature (°C)	Young's Modulus (GPa)
Al	100 Al	Cubic	150–200	76
Al ₂ Cu	52.5–53.9 Cu	Tetragonal	~225	108
Al ₂₀ Cu ₂ Mn ₃	12.8–19 Cu, 19.8–24 Mn	Orthorhombic	~350	–
Al ₈ CeCu ₄	19.2 Ce, 42.5 Cu	Tetragonal	350–400	–
Al ₂₄ MnCu ₈ Ce ₃	25.8 Ce, 30.9 Cu, 3.4 Mn	Cubic	350–400	–
Al ₁₆ Cu ₄ Mn ₂ Ce	15.0 Ce, 27.2 Cu, 11.7 Mn	–	350–400	–
Al ₇ Cu ₄ Ni	38.7–50.7 Cu, 11.8–22.2 Ni	Rhombohedral	350–400	163
Al ₃ CuNi	31.2 Cu, 29.9 Ni	Hexagonal	350–400	180

On the one hand, since the nano-sized precipitates in the grains can effectively impede the dislocation movement during the tensile processes, their size and number are of great significance [68–70]. The mass dispersed nanophases can effectively strengthen the alloy matrix. In the 2.0 Ni and 4.0 Ni alloy, the amount of nano-sized Al_2Cu and $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ significantly decreases, which can account for the substantial reduction in the tensile strength at the tested temperatures.

On the other hand, the strength of heat-resistant cast alloys can be given by the transfer of load from the ductile α -Al matrix to the rigid intermetallic phases at the interdendritic regions and grain boundaries. The strengthening mechanism is similar to that of micro-skeleton reinforced composites in previous research [71]. According to the previous research [72], the tensile strength of cast aluminum alloys, σ , can be described by Equation (5):

$$\sigma = (1 - f_p)\sigma_m + f_p\sigma_p \quad (5)$$

where σ_m and σ_p are the fracture strength of α -Al and reinforcing phases, respectively, and f_p is the volume fraction of the reinforcing phases. It can be inferred from the elongation and fracture morphology that the dominant fracture modes of the alloys are brittle fracture at room temperature, but a mixed form of brittle and ductile fracture at 400 °C. Therefore, the elastic stage is critical to the tensile properties of the alloys. In the elastic stage, the strain of α -Al must be consistent with the other phases; otherwise, cracks or overlaps will occur at the interface between α -Al and other phases. In addition, $\sigma = E \cdot \varepsilon$, then Equation (6) is written as:

$$\sigma = ((1 - f_p)E_m + f_pE_p) \varepsilon \quad (6)$$

where E_m and E_p are Young's moduli of the α -Al and the strengthening phases. Thus, the strengthening phases with high modulus suffer more stress than the low modulus phases do in the elastic stage. Compared with the 0 Ni alloy, the nanophases inside the grains in the 0.5 Ni alloy show no significant difference. However, the heat-resistant $\text{Al}_7\text{Cu}_4\text{Ni}$ phase in the 0.5 Ni alloy appears at the interdendritic regions and grain boundaries, and the thermal stable temperature of this phase approaches 350–400 °C. Meanwhile, the total volume fraction of intermetallic phases at the interdendritic areas and grain boundaries increases from ~12.5% to ~15.0% with the Ni content increasing from 0 to 0.5% as shown in Figure 10. In Table 4, the elastic modulus of the $\text{Al}_7\text{Cu}_4\text{Ni}$ phase (163 GPa) is much higher than that of the α -Al (76 GPa). Moreover, Figures 2 and 4 present that there are no defects at the interface between $\text{Al}_7\text{Cu}_4\text{Ni}$ and α -Al matrix. Consequently, higher stress can be transferred from the matrix to intermetallic compounds at the interdendritic regions and grain boundaries in the 0.5 Ni alloy.

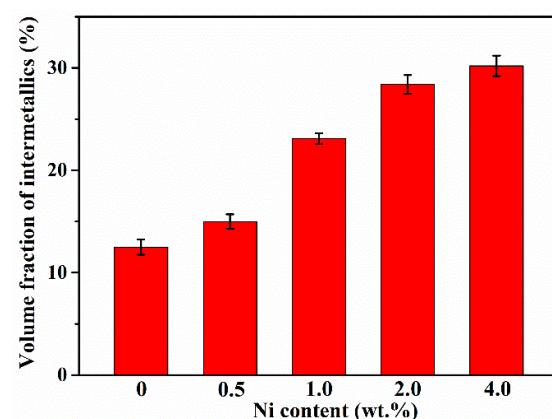


Figure 10. The volume fraction of intermetallics at the grain boundaries and interdendritic regions as a function of Ni content.

Further adding Ni (1.0%, 2.0%), plenty of Al_2Cu and $\text{Al}_7\text{Cu}_4\text{Ni}$ phases are formed as shown in Figure 4. However, the thermally stable Al_8CeCu_4 and $\text{Al}_{24}\text{MnCu}_8\text{Ce}_3$ phases,

which can effectively impose a drag on the boundary and impede the grain boundary sliding and migration at elevated temperatures, disappear in the 1.0 Ni alloy and 2.0 Ni alloy. Although the volume fraction of intermetallics at the interdendritic regions and grain boundaries obviously increases, the thermal stable temperature of Al_2Cu is only about 225 °C. As a result, the pinning effect of these intermetallics on the grain boundaries at 400 °C is reduced, and the elevated-temperature strength shows a slight downward trend. When adding 4.0% Ni, the Al_3CuNi phase is formed and substitutes for part Al_2Cu and $\text{Al}_7\text{Cu}_4\text{Ni}$ phases. The elastic modulus of Al_3CuNi (180 GPa) is higher than that of Al_2Cu (110 GPa) and $\text{Al}_7\text{Cu}_4\text{Ni}$ (163 GPa), which leads to enhanced tensile strength at 400 °C.

Table 5 shows the volume fraction of the reticular-like $\text{Al}_{24}\text{MnCu}_8\text{Ce}_3$ and Al_8CeCu_4 , blocky-like $\text{Al}_{16}\text{Cu}_4\text{Mn}_2\text{Ce}$, and skeleton-like $\text{Al}_7\text{Cu}_4\text{Ni}$ in the 0.5 Ni alloy reach 6.7%, 4.3%, 3.2%, respectively. Meanwhile, the width of the intermetallics distributed along the grain boundaries is less than 10 μm as seen in Figure 4, which is attributed to the low volume fraction of each phase and the regulation of heat treatment. In contrast to the 0 Ni alloy, these intermetallic phases become smaller and more evenly distributed, which is beneficial to reduce local stress concentration. As the external load increases, the strengthening phase breaks and the severe plastic deformation of α -Al occurs. The SDAS is refined in the 0.5 Ni alloy. As a result, the ductility is improved because of the reduced stress concentration and more uniform plastic deformation. As the increase of Ni content, the $\text{Al}_7\text{Cu}_4\text{Ni}$ and Al_2Cu at the grain boundaries and interdendritic areas are completely spheroidized and fragmentized into small spheres or transformed into a network shape with smooth surfaces after heat treatment. The refined smooth phases permit localized plastification of the matrix and help accommodate more damage, resulting in an increase of ductility in the 1.0 Ni and 2.0 Ni alloy. However, the coarse Al_3CuNi phase exhibits aggregated distribution, and thus, dissects matrix, which is responsible for the decreased ductility in the 4.0 Ni alloy.

Table 5. The volume fraction of intermetallic compounds phases at the grain boundaries and interdendritic areas in the 0.5 Ni alloy.

Phase	Volume Fraction (%)
$\text{Al}_8\text{CeCu}_4 + \text{Al}_{24}\text{MnCu}_8\text{Ce}_3$	6.7
$\text{Al}_7\text{Cu}_4\text{Ni}$	3.2
$\text{Al}_{16}\text{Cu}_4\text{Mn}_2\text{Ce}$	4.3
Al_2Cu	0.8
Total	15.0

5. Conclusions

The influence of Ni content (0.5%, 1.0%, 2.0%, 4.0%) on the microstructures and mechanical properties of heat-treated Al-8.4Cu-2.3Ce-1.0Mn-0.2Zr alloys was investigated. The following conclusions can be drawn.

After adding 0.5% Ni to the Al-8.4Cu-2.3Ce-1.0Mn-0.2Zr alloy, the spheroidized $\text{Al}_7\text{Cu}_4\text{Ni}$ phase is formed. With Ni content further increasing, the Al_8CeCu_4 and $\text{Al}_{24}\text{MnCu}_8\text{Ce}_3$ phases disappear, and the nano-sized $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ and Al_2Cu phases decrease gradually. When Ni content reaches 4.0%, the Al_3CuNi phase appears. It turns out that the addition of 0.5% Ni has significantly improved the tensile properties at 400 °C. The ultimate tensile strength, yield strength, and elongation of Al-8.4Cu-2.3Ce-1.0Mn-0.2Zr-0.5Ni alloy at 400 °C reach 103 MPa, 93 MPa, and 18.0%, respectively, which makes the alloy possible to be employed at 400 °C. The intermetallic micro-skeleton, composed of thermostable Al_8CeCu_4 , $\text{Al}_{24}\text{MnCu}_8\text{Ce}_3$, $\text{Al}_{16}\text{Cu}_4\text{Mn}_2\text{Ce}$, and $\text{Al}_7\text{Cu}_4\text{Ni}$ phases at the grain boundaries as well as nano-sized $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ and Al_2Cu precipitates in the grains, contributes to the good elevated-temperature tensile strength.

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administration, Z.Q.; funding acquisition, X.S. and Z.Q. All authors have read and agreed to the published version of the manuscript.

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