Effect of Annealing and Double Aging on the Microstructure and Mechanical Properties of Hot-Rolled $\text{Al}_{17}\text{Cr}_{10}\text{Fe}_{36}\text{Ni}_{36}\text{Mo}_{1}$ Alloy

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Abstract: AlCrFeNi multi-component alloys with excellent mechanical properties have been designed and extensively investigated in recent years. The massive fabrication of sheets will be an effective way for industrial application, where hot rolling is the inevitable process. After hot rolling, the mechanical properties could be further tailored. In this study, the effects of heat treatments on a dual-phase $\text{Al}_{17}\text{Cr}_{10}\text{Fe}_{36}\text{Ni}_{36}\text{Mo}_{1}$ hot-rolled plate were systematically investigated, including: (i) annealing (700 °C, 850 °C, 1000 °C and 1150 °C for 1 h, respectively), (ii) solution and single aging (1150 °C for 1 h and 700 °C for 4 h), (iii) solution and double aging (1150 °C for 1 h, 700 °C for 4 h and 650 °C for 1 h). The B2 precipitates with varied morphologies were observed in the FCC matrix of the hot-rolled alloy after a heat treatment range from 700 °C to 1000 °C for 1 h, and the BCC particles in the B2 matrix were dissolved when the heat treatment temperature was higher than 1000 °C. The hot-rolled alloy heat treated at 700 °C for 1 h had the highest yield strength, and the hot-rolled alloy treated at 1150 °C for 1 h showed the lowest yield strength. After a solution at 1150 °C for 1 h, aging at 700 °C for 4 h and 650 °C for 1 h, the L1₂ phase and BCC particles were precipitated in the FCC and B2 matrices, and B2 nanoprecipitates were observed around the FCC grain boundaries. The solution and double aging alloy exhibit the tensile strength of 1365.7 ± 9.5 MPa, a fracture elongation of 14.2 ± 1.5% at 20 °C, a tensile strength of 641.4 ± 6.0 MPa, and a fracture elongation of 16.9 ± 1.0% at 700 °C, showing great potential for application.

Keywords: AlCrFeNi; multi-component alloys; heat treatment; microstructures; double aging

1. Introduction

The concept of multi-component alloys (MCAs) breaks the design bottleneck of traditional alloys with one or two main elements and has been explored extensively over the past two decades [1–4]. Initially, MCAs were studied based on single FCC, BCC or HCP structures, and the designed single-phase MCAs exhibited better mechanical and physicochemical properties than traditional alloys [5–10]. However, in recent years, research focus has gradually changed to dual-phase MCAs, and the heterophase structure was intentionally beneficial for improving the mechanical properties of alloys [11–13]. The Co-free system of AlCrFeNi dual-phase MCAs has attracted much attention in the field of physical metallurgy. Soft FCC and the hard B2 dual-phase MCAs have been developed, and showed excellent comprehensive properties [14]. Recently, a new $\text{Al}_{17}\text{Cr}_{10}\text{Fe}_{36}\text{Ni}_{36}\text{Mo}_{1}$ MCAs has been developed, and its excellent properties showed great potential for industrial applications [15]. The different element compositions will have distinct effects on the microstructure and mechanical properties of the alloy. By microalloying $\text{Al}_{17}\text{Cr}_{10}\text{Fe}_{36}\text{Ni}_{36}$ with Mo and W, a dual-phase MCA with an ultimate strength and fracture elongation of

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1285 MPa and 16.0%, respectively, was obtained [16]. Liu et al. further investigated the impacts of non-metallic Si (0–4 at %) on the microstructure of the Fe_{36}Ni_{36}Cr_{10}Mo_{1}Al_{17–x}Si_{x} alloy, and found that the increase in Si led to a change in the crystallization sequence during solidification [17].

In addition to adjusting the composition and proportion of elements, the microstructure and properties of MCAs can also be regulated and improved by a series of thermomechanical processes, such as annealing after cold rolling [12,18,19], warm rolling [20–22], hot rolling [23–25] and hot forging [26–29]. Wang et al. conducted phase selective recrystallization followed by aging at 700 °C of the as-cast alloy, which transformed the dendrite structure into an equiaxed structure and achieved a synergistic improvement in strength and plasticity [19]. Tripathy et al. modulated the continuous deformation-induced nanosheets structure by annealing at 800 °C after warm rolling at 400 °C [22]. Pradhan et al. explored the evolution of the grain boundary characteristics during dynamic recrystallization by controlling the hot rolling deformation reduction [23]. Sourav et al. conducted a detailed analysis of the microstructures of Al_{x}CoCrFeNi (x = 0.3, 0.5, 0.7) alloys after hot forging and evaluated the contribution of different strengthening mechanisms [26]. In addition, Peng et al. explored the effects of simple heat treatments on the microstructures and mechanical properties of as-cast AlCoCrFeNi_{2.1} eutectic high entropy alloy. They found that the FCC phase precipitated in the B2 matrix and the tensile strength of the alloy increased from 1007 MPa to 1173 MPa after being treated at 600 °C, 700 °C, and 800 °C, while the FCC phase dissolved into the B2 matrix and the elongation of the material was slightly increased after being held above 1000 °C [30].

In recent years, great progress has been made in microstructure regulation and the strengthening–toughening of MCAs. The effects of heat treatments on the hot-rolled Al_{17}Cr_{10}Fe_{36}Ni_{36}Mo_{1} alloy remain to be further clarified. Therefore, it is essential to further explore the heat treatment process of an Al_{17}Cr_{10}Fe_{36}Ni_{36}Mo_{1} hot-rolled plate to obtain the superior properties of the alloy. In this paper, the influences of various heat treatment temperatures on an Al_{17}Cr_{10}Fe_{36}Ni_{36}Mo_{1} bulk hot-rolled plate were systematically investigated. The microstructures and mechanical properties of different heat treatment states were analyzed in detail, and the optimal heat treatment process was regulated. This work further complements the heat treatment behavior of the Al_{17}Cr_{10}Fe_{36}Ni_{36}Mo_{1} hot-rolled plate and provides important guidance for the industrial application of AlCrFeNi dual-phase MCAs.

2. Experiments

2.1. Preparation and Pretreatment of Samples

The vacuum degassed chromium, aluminum bean, nickel plate, industrial pure iron and nickel molybdenum alloy were added into the vacuum induction melting furnace in proportion to fabricate an Al_{17}Cr_{10}Fe_{36}Ni_{36}Mo_{1} ingot. Then, the vacuum induction ingot was further treated by electroslag remelting (ESR). After ESR, a diameter of 400 mm electroslag ingot was obtained by cooling for 1–3 h. Finally, the electroslag ingot was heated at 500 °C for 30 min, heated at 20 °C/min to 1170 °C for 20 min, and then hot-rolled repeatedly along the ingot axial direction up to 30 mm thickness. The width of the plate was 200 mm and the heat treatment experiments were conducted at 700 °C, 850 °C, 1000 °C, and 1150 °C for 1 h. The hot-rolled alloy was labeled as HR, and the heat-treated alloys under different heat treatment temperatures for 1 h were labeled as HR700, HR850, HR1000, and HR1150, respectively. The HR alloy was labeled as an HR-SA alloy after a solution at 1150 °C for 1 h and aging at 700 °C for 4 h and the HR-SA alloy was labeled as an HR-DA alloy after aging at 650 °C for 1 h. The hot-rolled samples were heated for heat treatments in a muffle furnace (Hefeikejing, Hefei, China, KSL-1200X) with an atmospheric environment (a temperature of 25.5 °C and a relative humidity of 60.5%). All heat treatment experiments were conducted by air cooling in order to avoid cracks.
2.2. Microstructural Characterization and Tensile Mechanical Property Tests

Cuboid samples with a size of 10 mm × 10 mm × 5 mm were taken at the quarter hot-rolled plate thickness position by the electro-spark wire-electrode cutting method; then, the samples in different heat treatment states were polished with 240-, 800-, 1500-, 2500-, and 4000-grit SiC papers to remove the oxide skin. The polished samples used in the microstructural characterization were electron-polished in a mixed solution of 90% anhydrous ethanol and 10% perchloric acid with a direct voltage of 30 V for 5 s at an ambient temperature. The TEM samples were mechanically abraded to an ~40 nm thickness, followed by electrolytic double spraying. An MTP-1A electrolytic double spray device was used to thin the sample in corrosive fluids of 90% anhydrous ethanol and 10% perchloric acid at −20 °C. The range of the voltage and current was 35–55 V and 45–75 mA, respectively. The microstructures of the hot-rolled alloy and different heat-treated alloys were characterized by an optical microscope (OM, OLYMPUS OLS4000, Tokyo, Japan) and a scanning electron microscopy (SEM, TESCAN MIRA3, Brno, Czech Republic) under a secondary electron mode. Electron back-scattered diffraction (EBSD, TESCAN MIRA3, Brno, Czech Republic) was used to further characterize the grain orientation and grain size. The nanoprecipitates were identified by transmission electron microscopy (TEM, Talos F200X, New York, NY, USA). The universal tensile tests at room temperature (20 °C) and elevated temperatures (600 °C, 700 °C, 800 °C and 900 °C) were conducted on a universal testing machine (UTM, TSMTE M6, Shenzhen, China) with a tensile strain rate of 10^{-3} s^{-1}. An extensometer (Sanjing Y12.5/5, Guangzhou, China) was used to monitor the strain for the room temperature testing and the thermostat and temperature controller were equipped to maintain a constant temperature for the high temperature testing. Tensile mechanical samples with a gauge dimension of 12.5 mm × 3.0 mm × 2.0 mm were used in this study. Samples of all the heat treatment state alloys were tested at least twice to verify the reproducibility of the tensile mechanical properties.

3. Results and Discussion

3.1. Short-Time Annealing at Different Temperatures

Figure 1 shows the alloys’ microstructure at five different states. As Figure 1(a1–a3) shows, the microstructure under a hot-rolled state indicates a typical dual-phase structure, in which the white region is the FCC phase and the gray region is the B2 phase [19]. Moreover, the B2 phase was squished along the rolling direction. After short-time annealing at 700 °C, 850 °C, 1000 °C, and 1150 °C for 1 h (Figure 1(a2–a5)), there were some differences. The most obvious change was the randomly distributed particles in the FCC matrix and small FCC phases in the B2 matrix, whose density changed after annealing at different temperatures. Nevertheless, no regularity related to the temperature was found.

In order to further analyze the microstructural changes under different temperatures, the five states of the alloys were characterized by scanning electron microscopy (SEM). The matrix phase composition of the primary HR alloy (Figure 1(a3)) was further determined to be an FCC phase and B2 phase. And spherical BCC particles were observed in the B2 matrix phase, consistent with a previous report [19]. The HR700 alloy (Figure 1(b3)) has a similar microstructure to the HR alloy overall. However, some nanoprecipitates were observed in the FCC matrix of the HR700 alloy, which may have a vital impact on the mechanical properties of the alloy. For the HR850 alloy (Figure 1(c3)), some needle-like (NL) precipitates with widths of 200–400 nm precipitated in the FCC phase. The length of the needle-like structures seemed to be different due to the varied orientation of the NL structures. With heat-treatment temperature rises to 1000 °C (Figure 1(d3)), a large amount of the B2 phase with a size below 2 µm precipitated in the FCC phase. Moreover, the nano-scale spherical BCC precipitates that were originally distributed in the B2 matrix in the HR alloy disappeared, which might be dissolved by the higher temperature compared to the HR700 alloy and HR850 alloy. Similarly, the BCC particles in the HR1150 alloy (Figure 1(e3)) were also dissolved and the FCC matrix became clean and tidy. The clean and tidy FCC phase was formed since the small B2 phases in the original FCC matrix (HR alloy)
gradually aggregated into the larger particles. These results indicate that the heat treatment temperature can change the precipitation behavior as well as the phase composition of the alloy, which means that the mechanical properties can be regulated by choosing the right heat treatment process to adjust the precipitation behaviors.

Figure 1. Microstructures of the five different states alloys: (a1–a3) images of HR alloy; (b1–b3) images of HR700 alloy; (c1–c3) images of HR850 alloy; (d1–d3) images of HR1000 alloy; (e1–e3) images of HR1150 alloy. The first two colored images were acquired by optical microscope while the black-and-white images were captured by scanning electron microscopy. On the right side of each image was the magnified image of the corresponding alloy.

Figure 2 shows the electron back scatter diffraction (EBSD) maps of the five different state alloys heat-treated at different temperatures. From the EBSD phase-index map (Figure 2(a1)) of the HR alloy, the structure of the B2 matrix (red color) was elongated along the rolling direction, and the B2 particles were dispersed in the FCC matrix (green color). According to the EBSD inverse pole figure (Figure 2(a2)), the HR alloy showed an obvious
equiaxial dual-phase structure, which indicates the occurrence of dynamic recrystallization in the rolling process. Compared with the HR alloy, more precipitates were observed in the FCC matrix of the HR700 alloy (Figure 2b), which was basically consistent with SEM results. As for the other three heat-treated alloys, numerous B2 precipitates were observed in the HR850 alloy and HR1000 alloy (Figure 2c,d), while the B2 particles in the HR1150 alloy (Figure 2(e1)) were coarsened into larger B2 islands. The average grain size of the FCC phase and B2 phase was calculated according to EBSD results (Figure 2(a3–e3)). The results show that the HR alloy, HR700 alloy, HR850 alloy, and HR1000 alloy had similar grain sizes, while the HR1150 alloy had the largest grain size.

Figure 2. Electron back-scattered diffraction (EBSD) results of five different state alloys: (a1–a3) images of HR alloy; (b1–b3) images of HR700 alloy; (c1–c3) images of HR850 alloy; (d1–d3) images of HR1000 alloy; (e1–e3) images of HR1150 alloy. From left to right, the results for each alloy were phase map, EBSD inverse pole figure, and corresponding frequency distribution of grain size, respectively. Due to the dual-phase structure of this alloy, the grain sizes of the FCC phase and the B2 phase were calculated, respectively.
The changed microstructures indicate different mechanical properties. Tensile tests of the five alloys were carried out at room temperature; the engineering strain–stress curves and related data are shown in Figure 3 and Table 1, respectively. The results show that all alloys demonstrated a good combination of strength and elongation. The HR alloy with yield and ultimate strengths of 471.2 ± 6.5 MPa and 1019.2 ± 8.5 MPa has a fracture elongation of 20.1 ± 1.0%. After heat treatment at 700 °C, the yield strength, ultimate strength, and fracture elongation of the alloy were slightly increased to 568.4 ± 5.0 MPa, 1107.2 ± 9.0 MPa and 22.2 ± 1.5%, respectively. This is mainly due to the B2 precipitates in the FCC matrix. Compared with the HR alloy, the yield and the ultimate strength of the HR850 alloy, HR1000 alloy, and HR1150 alloy were reduced, while the ductility improved to some extent. Interestingly, the strength of the HR1000 alloy is greater than that of the HR850 alloy, mainly due to there being more B2 precipitates in the FCC matrix of the HR1000 alloy. The large grain size and aggregation of B2 particles result in the lowest yield strength (360.7 ± 5.0 MPa) and ultimate strength (955.9 ± 7.5 MPa) of the HR1150 alloy.

![Figure 3. Stress–strain curves of HR alloy, HR700 alloy, HR850 alloy, HR1000 alloy and HR1150 alloy tested at room temperature.](image)

Table 1. Tensile properties of HR alloy, HR700 alloy, HR850 alloy, HR1000 alloy and HR1150 alloy, where σ_s, σ_b and ε_p are the yield strength, ultimate strength, and fracture elongation, respectively.

<table>
<thead>
<tr>
<th>Alloys</th>
<th>σ_s, MPa</th>
<th>σ_b, MPa</th>
<th>ε_p, %</th>
</tr>
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<tbody>
<tr>
<td>HR</td>
<td>471.2 ± 6.5</td>
<td>1019.2 ± 8.5</td>
<td>20.1 ± 1.0</td>
</tr>
<tr>
<td>HR700</td>
<td>568.4 ± 5.0</td>
<td>1107.2 ± 9.0</td>
<td>22.2 ± 1.5</td>
</tr>
<tr>
<td>HR850</td>
<td>400.8 ± 5.5</td>
<td>980.3 ± 7.0</td>
<td>31.6 ± 1.5</td>
</tr>
<tr>
<td>HR1000</td>
<td>414.1 ± 7.0</td>
<td>1008.1 ± 9.5</td>
<td>28.1 ± 1.0</td>
</tr>
<tr>
<td>HR1150</td>
<td>360.7 ± 5.0</td>
<td>955.9 ± 7.5</td>
<td>23.4 ± 1.0</td>
</tr>
</tbody>
</table>

3.2. Solution and Aging

According to the results in Section 3.1, the yield strength of the HR alloy will be increased at 700 °C due to the nanoprecipitates in the FCC matrix, and the yield strength of the HR1150 alloy will be reduced at 1150 °C for the dissolution of B2 precipitates in the FCC matrix. Therefore, 1150 °C was deemed a suitable temperature to remove the heterophases and 700 °C was considered a suitable temperature to trigger the precipitations. Meanwhile, the temperature of 650 °C, 50 °C below 700 °C, was also taken into account as the proportion...
of nanoprecipitates might be increased without changing their size as much as possible. Inspired by the results and reasonable inference, the solution, solution and single aging (SSA), solution and double aging (SDA) processing technology was investigated. As shown in Figure 4, the dot line curves of Vickness hardness versus time was obtained by calculating the average hardness acquired under different processing technology. The red five-pointed star indicates the appropriate treatment time under different temperatures. It was clear that the solution time, not an obvious characteristic over time, was not a major factor. Therefore, the minimum time of 1 h solution was enough. Single aging at 700 °C reached its peak value after treatment for 4 h, different from the 650 °C with an upward trend in the test time. The above results suggest that the first-stage aging at 700 °C was beneficial for rapid precipitation within 4 h, better than 650 °C. The temperature of 650 °C was therefore followed after 700 °C to increase the volume fraction or amount of the nanoprecipitates. According to the red line at the top of the curves, the heat treatment process of that solution at 1150 °C for 1 h, aging at 700 °C for 4 h, and 650 °C for 1 h were thus determined and corresponding structural characterization and mechanical testing were also conducted.

![Figure 4](image_url)

**Figure 4.** Vickness hardness curves of the HR alloy under different heat-treated processes. From down to up, it represents solution, solution and single aging, solution, and double aging process. The red five-pointed star in the curves indicate the suitable treating time.

Figure 5(a1,b1) show the microstructure of the HR-SA alloy and HR-DA alloy. Compared with the HR alloy, some precipitates were observed at the FCC grain boundaries in both alloys, which also had a similar precipitation behavior to the HR700 alloy. According to high-resolution SEM images (Figure 5(a2–a4,b2–b4)), nano-spherical and short rod-like precipitates were observed in the B2 matrix and FCC matrix of both the HR-SA alloy and HR-DA alloy. And what surprised us was that the number and size of the short rod-like precipitates in the HR-DA alloy were greater than those of the HR-SA alloy, and that the short rod-like precipitates would have a certain effect on the mechanical properties of the alloy.

According to the above results, the HR-SA alloy and HR-DA alloy had a similar precipitation behavior but a different amount and size of the short rod-like precipitates. The HR-DA alloy was further analyzed by transmission electron microscopy (TEM), as shown in Figure 6. Figure 6a shows the bright-field image of the B2 phase. Spherical nanoprecipitates with an average size of 30 nm were distributed homogeneously in the matrix phase. The corresponding selected-area electron diffraction (SAED) pattern is shown in Figure 6b, and the two-phase structures of BCC/B2 were further determined. The dark-field image (Figure 6c) was obtained by selecting (100)B2 superlattice diffraction spots to verify the phase structure of the nanoprecipitates and matrix. The precipitates showed dark and the matrix showed bright, indicating that the matrix was the B2 phase. A
similar characterization was conducted on the FCC phase. Some rod-like structures with an average width of 20 nm were precipitated in the matrix (Figure 6d). A further SAED (Figure 6e) pattern and dark-field image (Figure 6f) indicated that rod-like L12 precipitates were dispersed in the FCC matrix. The above results confirm that after solution and double aging, nano-spherical BCC and rod-like L12 precipitates were observed in the B2 and FCC matrices of the HR-DA alloy, respectively.

![Figure 5](image-url)  
**Figure 5.** Microstructures of solution and aging alloys: (a1–a4) images of HR-SA alloy; (b1–b4) images of HR-DA alloy. The first colored image was acquired by optical microscope while the black-and-white images were captured by scanning electron microscopy. The two alloys were composed of FCC matrix and B2 matrix, and SEM images at magnified scale of the FCC matrix and B2 matrix for each alloy were placed on the right of this figure.

![Figure 6](image-url)  
**Figure 6.** Transmission electron microscopy images of HR-DA alloy: (a) Bright-field image of BCC phase and B2 phase; (b) SAED pattern of BCC phase and B2 phase; (c) Dark-field image of BCC phase and B2 phase; (d) Bright-field image of FCC phase and L12 phase; (e) SAED pattern of FCC phase and L12 phase; (f) Dark-field image of FCC phase and L12 phase.

An uniaxial tensile test was performed to validate the differences in the mechanical properties for the HR-SA alloy and the HR-DA alloy, as shown in Figure 7a. The HR-SA alloy exhibits a yield strength of 713.0 ± 6.5 MPa and a tensile strength of 1263.9 ± 8.0 MPa, as well as a fracture elongation of 13.4 ± 1.6%. After further heat treatment at 650 °C, the yield strength, tensile strength, and fracture elongation of the HR-SA alloy increased to 813.2 ± 7.0 MPa, 1365.7 ± 9.5 MPa, and 14.2 ± 1.5% in the HR-DA alloy, respectively.
The enhancement of the HR-DA alloy’s strength was mainly attributed to the greater precipitation of \( \text{L1}_2 \) in the FCC matrix, which occurred at 650 °C. Comparing the mechanical properties of the HR-DA alloy with those of other MCAs at 20 °C, shown in Figure 8a, our alloy reveals a great combination of strength and plasticity. The high temperature tensile behavior of the HR-DA alloy at 600–900 °C was tested, and the mechanical property results are shown in Figure 7b and Table 2. The yield and tensile strength displayed a declining trend while the elongation showed an increasing trend. The HR-DA alloy shows an excellent yield strength of 576.8 ± 5.5 MPa and a competitive fracture elongation of 16.9 ± 1.0% at 700 °C. When comparing it with other alloys at 700 °C, our alloy still exhibits better high-temperature mechanical properties (Figure 8b). During the elevated temperature deformation, the B2 nanoprecipitates at the FCC grain boundaries hindered the dislocation movement as well as the grain boundary sliding. The above results show that our well-prepared MCAs exhibit good mechanical properties in a wide temperature range.

![Figure 8](image_url)

**Figure 7.** Tensile stress–strain curves of the alloys at different temperatures: (a) HR alloy, HR-SA alloy, and HR-DA alloy tested at room temperature; (b) HR-DA alloy tested at 600–900 °C.

![Figure 8](image_url)

**Figure 8.** Comparison of tensile properties of HR-DA alloy with other MCAs at different temperatures (a) 20 °C and (b) 700 °C, respectively adapted from Refs. [31–37].
were observed on the fracture surface. The above results show that the HR-DA alloy which is also consistent with a previous study [16]. The microstructures and fracture morphology of the corresponding alloys. The three alloys' fracture surfaces had similar fracture morphologies, as shown in Figure 9. All alloys exhibited two classical structural characteristics of cleavage planes and dimples, which were indicated by blue arrows and red arrows, respectively. The soft FCC matrix has more slip systems and its fracture mode was mainly controlled by dimples. The brittle B2 matrix’s fracture mode was mainly a cleavage fracture. During the rolling process, the B2 matrix was elongated along the rolling direction. Therefore, obvious disk-like cleavages with river patterns were observed in the fracture morphologies. The branches of the river patterns correspond to the various cleavage planes of the same crystal plane, and the flow direction of the river was consistent with the propagation direction of the crack. Large tearing edges were shown where the cleavage planes met the dimples, which is also consistent with a previous study [16]. The microstructures and fracture elongation of the HR alloy, HR-SA alloy, and HR-DA alloy were not very different, and the three alloys also had similar fracture modes.

The tensile fracture morphologies of the HR alloy, HR-SA alloy, and HR-DA alloy at room temperature were characterized in order to further analyze the fracture behavior of the alloys. The three alloys’ fracture surfaces had similar fracture morphologies, as shown in Figure 9. All alloys exhibited two classical structural characteristics of cleavage planes and dimples, which were indicated by blue arrows and red arrows, respectively. The soft FCC matrix has more slip systems and its fracture mode was mainly controlled by dimples. The brittle B2 matrix’s fracture mode was mainly a cleavage fracture. During the rolling process, the B2 matrix was elongated along the rolling direction. Therefore, obvious disk-like cleavages with river patterns were observed in the fracture morphologies. The branches of the river patterns correspond to the various cleavage planes of the same crystal plane, and the flow direction of the river was consistent with the propagation direction of the crack. Large tearing edges were shown where the cleavage planes met the dimples, which is also consistent with a previous study [16]. The microstructures and fracture elongation of the HR alloy, HR-SA alloy, and HR-DA alloy were not very different, and the three alloys also had similar fracture modes.

Table 2. Mechanical test results for HR-DA alloy at room temperature and elevated temperatures, where σ_y, σ_u, and ε_p are the yield strength, ultimate strength, and fracture elongation, respectively.

<table>
<thead>
<tr>
<th>Temperature °C</th>
<th>σ_y, MPa</th>
<th>σ_u, MPa</th>
<th>ε_p, %</th>
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<tbody>
<tr>
<td>20</td>
<td>813.2 ± 6.5</td>
<td>1365.7 ± 11.5</td>
<td>14.2 ± 1.0</td>
</tr>
<tr>
<td>600</td>
<td>702.6 ± 7.0</td>
<td>913.1 ± 8.5</td>
<td>20.1 ± 1.5</td>
</tr>
<tr>
<td>700</td>
<td>576.8 ± 5.5</td>
<td>641.4 ± 6.0</td>
<td>16.9 ± 1.0</td>
</tr>
<tr>
<td>800</td>
<td>339.1 ± 7.5</td>
<td>346.3 ± 7.0</td>
<td>43.5 ± 1.5</td>
</tr>
<tr>
<td>900</td>
<td>152.9 ± 5.0</td>
<td>163.6 ± 5.5</td>
<td>102.2 ± 1.0</td>
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</table>

Figure 9. Secondary electron micrographs of the fracture morphology for (a) HR alloy, (b) HR-SA alloy, and (c) HR-DA alloy testing at room temperature. The insets show the macroscopic fracture of the corresponding alloys.

In order to further understand the fracture behaviors of the HR-DA alloy at different temperatures, the mechanical fracture morphologies of the HR-DA alloy at 600–900 °C are shown in Figure 10. This alloy had a similar fracture mechanism at 600 °C and 700 °C (Figure 10a,b), the fracture mode of the FCC matrix was mainly dimples (red arrows), and the fracture mode of the B2 matrix was mainly a cleavage fracture (blue arrows). When the testing temperature was elevated to 800 °C (Figure 10c), the fracture surface of our alloy showed an obvious necking phenomenon. The fracture morphology was mainly dominated by dimples with various sizes and some oxidation products; no significant cleavage planes and rive patterns were observed. When the temperature rose to 900 °C (Figure 10d), the fracture morphology showed larger and deeper dimples and more oxidation products were observed on the fracture surface. The above results show that the HR-DA alloy exhibited a mixed fracture mode when deformed at 600 °C and 700 °C. With the increase in temperature, the B2 matrix was softened. And the fracture mode of the B2 matrix changed...
from a cleavage fracture to dimples, which was undesirable for the mechanical properties of our alloy.

![Secondary electron micrographs of the fracture morphology for HR-DA alloy testing at different temperatures.](image)

**Figure 10.** Secondary electron micrographs of the fracture morphology for HR-DA alloy testing at (a) 600 °C, (b) 700 °C, (c) 800 °C, and (d) 900 °C. The insets show the macroscopic fracture of the corresponding temperatures and that they exhibited a significant necking phenomenon above 800 °C.

4. Conclusions

This work focused on the hot-rolled Al$_{17}$Cr$_{10}$Fe$_{36}$Ni$_{36}$Mo$_{1}$ large plate, investigating the effects of different heat treatments on the microstructure and mechanical properties. The main conclusions are as follows.

1. The precipitation behaviors of the HR alloy can be affected by different annealing temperatures. In the FCC matrix, some B$_2$ nanoprecipitates were observed in the HR700 alloy, the needle-like B$_2$ structures were precipitated in the HR800 alloy, and the BCC particles precipitated in the B2 matrix of the HR alloy were completely dissolved above 1000 °C.

2. The HR-DA alloy showed better comprehensive mechanical properties than the HR-SA alloy. After the HR alloys were treated with SSA and SDA, short rod-like L$_{12}$ precipitates were observed in the FCC matrix of both the HR-SA and HR-DA alloys, and the number and size of the L$_{12}$ phases in the HR-DA alloy were greater than those of the HR-SA alloy. The L$_{12}$ precipitates can greatly improve the strength of the alloy by hindering the movement of dislocation. Therefore, the HR-DA alloy shows the excellent tensile strength of $1365.7 \pm 9.5$ MPa and fracture elongation of $14.2 \pm 1.5\%$ at room temperature.

3. The HR-DA alloy also showed acceptable mechanical properties at a high temperature. The L$_{12}$ phases and B$_2$ nanoprecipitates were observed in the FCC matrix and FCC grain boundaries, respectively. The high-density L$_{12}$ phases significantly contributed to the yield strength and the B$_2$ nanoprecipitates can effectively hinder the dislocation movement as well as the grain boundary sliding. These results led to the HR-DA alloy obtaining a high tensile strength of $641.4 \pm 6.0$ MPa and a fracture elongation of $16.9 \pm 1.0\%$ at 700 °C, showing great potential for elevated temperatures applications.
In the current study, we mainly focused on the influence of a heat treatment process on the microstructure and mechanical properties. The strengthening mechanisms of the HR-DA alloy will be further analyzed in a future investigation.

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