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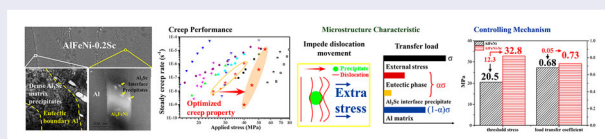
Understanding the creep property of heat-resistant Al alloy by analyzing eutectic phase/matrix interface structures

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ABSTRACT

Interface characteristics are of great importance in mechanical performance for multi-phases heat-resistant Al alloys. Herein, Al₃Sc precipitates were created at both Al/Al₉FeNi interface and Al matrix inside Al–Fe–Ni eutectic alloy by microalloyed Sc. Critically, Al₃Sc interface precipitates were found to alleviate local lattice distortion at the Al/Al₉FeNi interface to improve interface stability. Therefore, this alloy showed superior creep properties over available reports. The underlying mechanisms were quantitatively elucidated by elevated load transfer effect from interface precipitates and enhanced threshold stress from matrix precipitates. Generally, this work provided a solid understanding to optimize the creep property of eutectic Al alloys by manipulating interface structures.



IMPACT STATEMENT

This work reports that Al₃Sc precipitates at Al/Al₉FeNi interface can enhance load transfer effect of eutectic phase from microscopic/macroscale factors, illustrating fundamental mechanisms for creep improvement of eutectic Al alloy.

1. Introduction

Heat-resistant Al alloys have been widely used in aerospace and automotive fields because they are light-weight and have high strength [1–3]. Traditionally, Al–Si–(Cu) heat-resistant Al alloys are mostly used at ~200°C, subjected to the rapid diffusion rates of Si and/or Cu in the Al matrix [4–6]. This cannot meet increasing demands from practical applications. Therefore, the design of heat-resistant Al alloys with improved microstructure stability is vital to optimizing their high-temperature performances.

Introducing slow diffusion elements into the Al matrix, there are two types of newly developed heat-resistant Al alloys divided by a major strengthening

phase: (I) Eutectic structure reinforced Al alloys. Since elements (i.e. Fe, Ni, La and Ce) have slower diffusion rates in the Al matrix [7], their eutectic Al alloys (i.e. Al–Ni [1], Al–Fe–Ni [8,9], Al–La [10] and Al–Ce [11]) show good performance at 300–400°C. (II) Precipitates strengthened Al alloys. The highly stable precipitates are introduced into the Al matrix by microalloying elements (i.e. Sc, Zr or Mn) [12,13]. For example, microalloying Sc is widely applied, and Al₃Sc precipitates are created in the Al matrix to improve the thermal stability of Al alloys at 300°C [14,15]. Meanwhile, Al₃Sc precipitates can impede dislocation movement to improve the creep resistance of Al alloys [16,17]. Furthermore, Sc is used to optimize the interface structure of multi-phases in Al alloys, elevating

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their thermal stability [18]. For example, Chen and Gao [19,20] studied Al-Cu-Sc alloy and found there was a stable Sc-containing layer at the Al/ θ' interface, ensuring θ' phase stability at 300 °C and impeding dislocation movement at the interface [20].

Eutectic alloys have been widely discussed because of their excellent mechanical properties and application prospects [21]. The investigation and analysis of the abundant metal/intermetallic interface structure are helpful to understand and optimize the mechanical properties of eutectic alloys. Zhou [22] revealed the structure features of the Al/Al₂Cu interface and explained the formation mechanism of lattice dislocations. Based on this, Liu [23] prepared laser surface remelted Al-Al₂Cu eutectic alloy and elucidated that the performance of high strength and plasticity depends on the metal/intermetallic heterointerface. Furthermore, the modeling of interface strengthening in Al-Si eutectic alloys [24] and the research on laser rapid solidified Al-Si fibrous eutectics [25] demonstrated that synergistic enhancement of strength and plasticity of Al-Si can be achieved by coordinating the soft Al matrix and hard nano-scale Si eutectic phase through the heterogeneous interface. Therefore, the optimizing interface structure in eutectic alloys can improve their mechanical performance.

In combination with eutectic structures with the microalloying method, Sc is added to eutectic Al alloys to tune their creep performance [26,27]. For example, the increased Sc addition can cause a higher load transfer effect of the eutectic phase in Al-Ni-Sc alloy [26]. However, there is a lack of in-depth discussion on microstructure-mechanical property relationship for these novel heat-resistant Al alloys, including (1) Microstructure characteristics involved Sc addition to eutectic alloys; (2) Strengthening mechanisms controlled by Sc-induced microstructures to alloy creep properties.

Therefore, the creep behavior of Al-1.75wt%Fe-1.25wt%Ni-0.2wt%Sc eutectic alloy (i.e. AlFeNi-0.2Sc alloy) was studied to show the influence of induced microstructure features by microalloying Sc. In comparison with the creep behavior of the AlFeNi alloy, the controlling creep mechanisms for the AlFeNi-0.2Sc alloy were quantitatively modeled and discussed. Specifically, Al₃Sc precipitates at the Al/Al₉FeNi interface were characterized to explain the corresponding load transfer effect, which was beneficial to optimizing creep properties.

2. Experiments

The AlFeNi-0.2Sc eutectic alloy was prepared by conventional gravity cast, as detailed in our previous work [8,9].

Before microstructure characterization and mechanical test, the as-cast alloy was heat treated at 300°C@4 h as the peak-aged state. The microstructure was characterized by Scanning Electron Microscope (SEM), and the element distribution was acquired by energy dispersive spectroscopy (EDS). The precipitates inside the Al matrix or at the Al/Al₉FeNi interface were analyzed by Transition Electron Microscope (TEM) or Scanning Transition Electron Microscope (STEM). Meanwhile, the high-resolution TEM (HRTEM) image was acquired to demonstrate the lattice structure of precipitates. Based on the HRTEM image, the lattice strain nearby the interface was analyzed by Geometric Phase Analysis (GPA) [28]. Besides, the electronic creep testing machine (RDL50) was used for the creep test. The applied creep stress was in the range of 20–60 MPa.

3. Results and discussions

AlFeNi is a typical eutectic alloy with the Al₉FeNi and Al matrix as the main eutectic structure (Figure 1(a)). Comparably, AlFeNi-0.2Sc shows a similar microstructure (Figure 1(b)). Therefore, the eutectic structure can be divided into three sub-divisions according to microstructure features (Figure 1(a,b)), including primary Al (Sub-division I), eutectic boundary Al (Sub-division II) and eutectic internal Al (Sub-division III). Although microalloying 0.2 wt% Sc hardly affects the basic macro-morphology of eutectic structure (Figure 1(b)), it has promoted the formation of primary Al (Sub-division I), which is rare in eutectic AlFeNi alloy (Figure 1(a)).

Sc mainly dissolves in the Al matrix of the AlFeNi-0.2Sc alloy. Influenced by element distribution during solidification in the AlFeNi-0.2Sc alloy, Sc tends to gather at grain boundaries [29]. In detail, more Sc is pushed by the Al₉FeNi phase to the eutectic boundary Al [30], where the EDS result for Sc concentration is much higher than other sub-divisions (Figure 1(c)). Therefore, aggregated Al₃Sc precipitates inside eutectic boundary Al are revealed (Figure 1(d)), and these precipitates are called matrix precipitates. The average diameter of these matrix precipitates is ~ 5 nm [16]. However, matrix precipitates are rarely seen inside the eutectic grains (Figure 1(e)). Herein, the precipitation behavior of Sc inside the eutectic grain may be changed, and we have identified precipitates at the Al/Al₉FeNi interface by EDS and TEM analyses. For instance, a typical Al₉FeNi phase with surrounding elements distribution is shown (Figure 2(a–b)), and the Sc element is rich at the Al/Al₉FeNi interface.

Observed at another Al₉FeNi phase, several irregular patches with higher contrast are seen at the Al/Al₉FeNi interface (Figure 2(c)). Then, five featured positions are

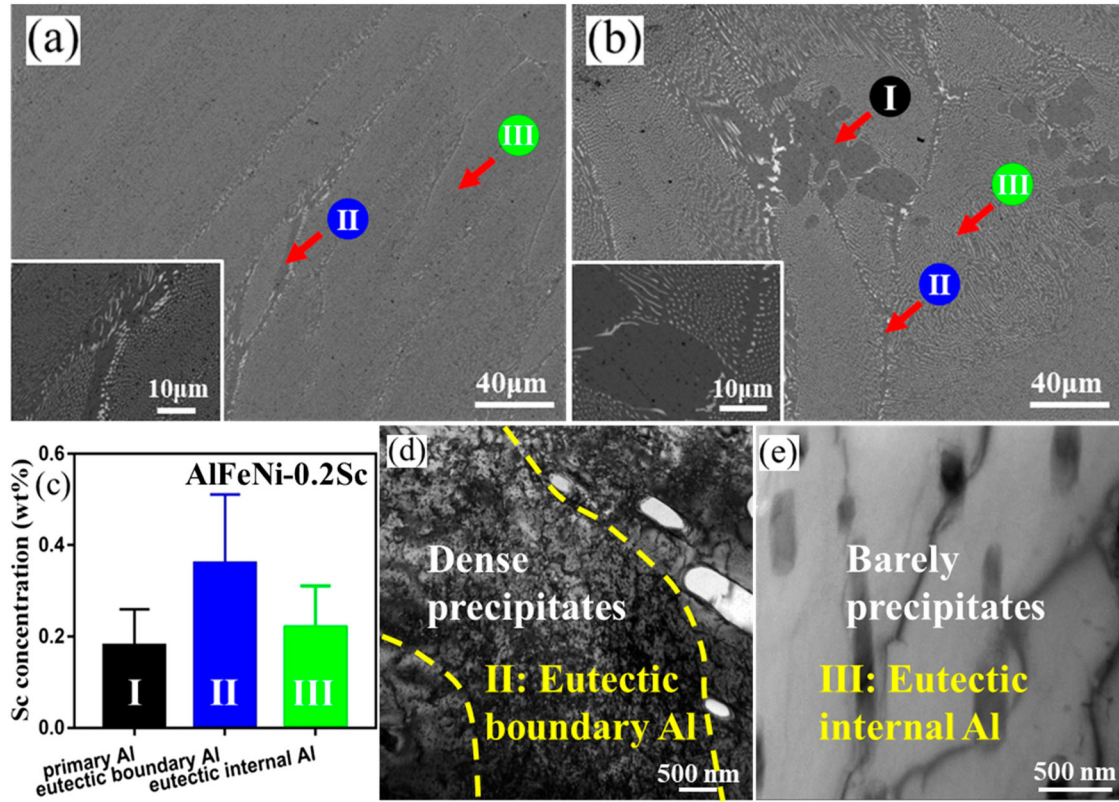


Figure 1. Microstructure characteristics: (a) AlFeNi; (b) AlFeNi-0.2Sc. (c) Sc concentration in different areas. Al_3Sc distribution in different zones of the AlFeNi-0.2Sc alloy at peak-aged state: (d) Eutectic boundary Al; (e) Eutectic internal Al.

selected to make the EDS test, and the results are shown in Table 1. Accordingly, Positions 1–3 should be precipitates containing Sc. Meanwhile, Sc is rarely detected in the Al matrix (Position 4) and eutectic phase (Position 5).

Furthermore, Position A near the precipitate (Position 3) on the Al/ Al_9FeNi interface is characterized by an HRTEM image (Figure 2(d)). Herein, such precipitate size reaches tens of nanometers, and its shape presents irregular dendritic morphology. Besides, the cross-section of this precipitate is provided in SM (Supplementary Materials)-Part 1. The thickness of this precipitate is ~ 5 nm, and the length is ~ 30 nm in the direction along the interface. Its thickness is similar to the diameter of Al_3Sc matrix precipitates, but the length is much longer than the size of matrix precipitates under a peak-aged state. The precipitate has an ordered lattice structure, and the superlattice diffraction pattern is shown in a Fourier Fast Transform image (Figure 2(e)). Therefore, the precipitate is referred to the L_{12} - Al_3Sc phase. This Al_3Sc precipitate at the Al/ Al_9FeNi interface is called interface precipitate. Also, HRTEM images of Positions B and C are provided in SM-Part 2 to confirm the wide existence of interface precipitates.

To reveal characteristic structures at Al/ Al_9FeNi , Figure 3(a) identifies a certain orientation relationship

(OR) between Al_9FeNi and the Al matrix. The zone axis for the Al matrix is determined as $[011]_{\text{Al}}$, and for the Al_9FeNi phase is $[0\bar{1}0]_{\text{Al}_9\text{FeNi}}$ (Figure 3(a)). Under these parallel zone axes, the nearly paralleling planes are $(11\bar{1})_{\text{Al}}$ and $(00\bar{4})_{\text{Al}_9\text{FeNi}}$, although there is a $\sim 4.5^\circ$ orientation difference between them. Then, the corresponding OR1 is written as

$$[011]_{\text{Al}} // [0\bar{1}0]_{\text{Al}_9\text{FeNi}} \text{ and } (11\bar{1})_{\text{Al}} // \sim 4.5^\circ (00\bar{4})_{\text{Al}_9\text{FeNi}} \quad (1)$$

According to OR1, the $(0\bar{1}0)_{\text{Al}_9\text{FeNi}}$ plane is parallel to the $(011)_{\text{Al}}$ plane, and the interface is constructed using an Edge-to-Edge manner. The $[100]_{\text{Al}_9\text{FeNi}}$ direction on the $(0\bar{1}0)_{\text{Al}_9\text{FeNi}}$ surface is nearly parallel to the $[2\bar{1}1]_{\text{Al}}$ direction on the $(011)_{\text{Al}}$ surface. Therefore, the interface perpendicular to the image section is constructed by both $(00\bar{1})_{\text{Al}_9\text{FeNi}}$ and $(11\bar{1})_{\text{Al}}$ planes. The schematic diagram of OR1 is shown in Figure 3(c).

Meanwhile, the Al_9FeNi structure viewed from $[0\bar{1}0]_{\text{Al}_9\text{FeNi}}$ is shown in Figure 3(b). The distributions of Fe and Ni atoms in the structure are consistent with the TEM observation (Inset in Figure 3(b)). Herein, the marked section is $(00\bar{1})_{\text{Al}_9\text{FeNi}}$ plane. According to the crystal structure, Al atoms on the plane can be classified into three types: A, B and C (Figure 3(b)). Type A atoms are on the plane, and type B and C atoms are on two

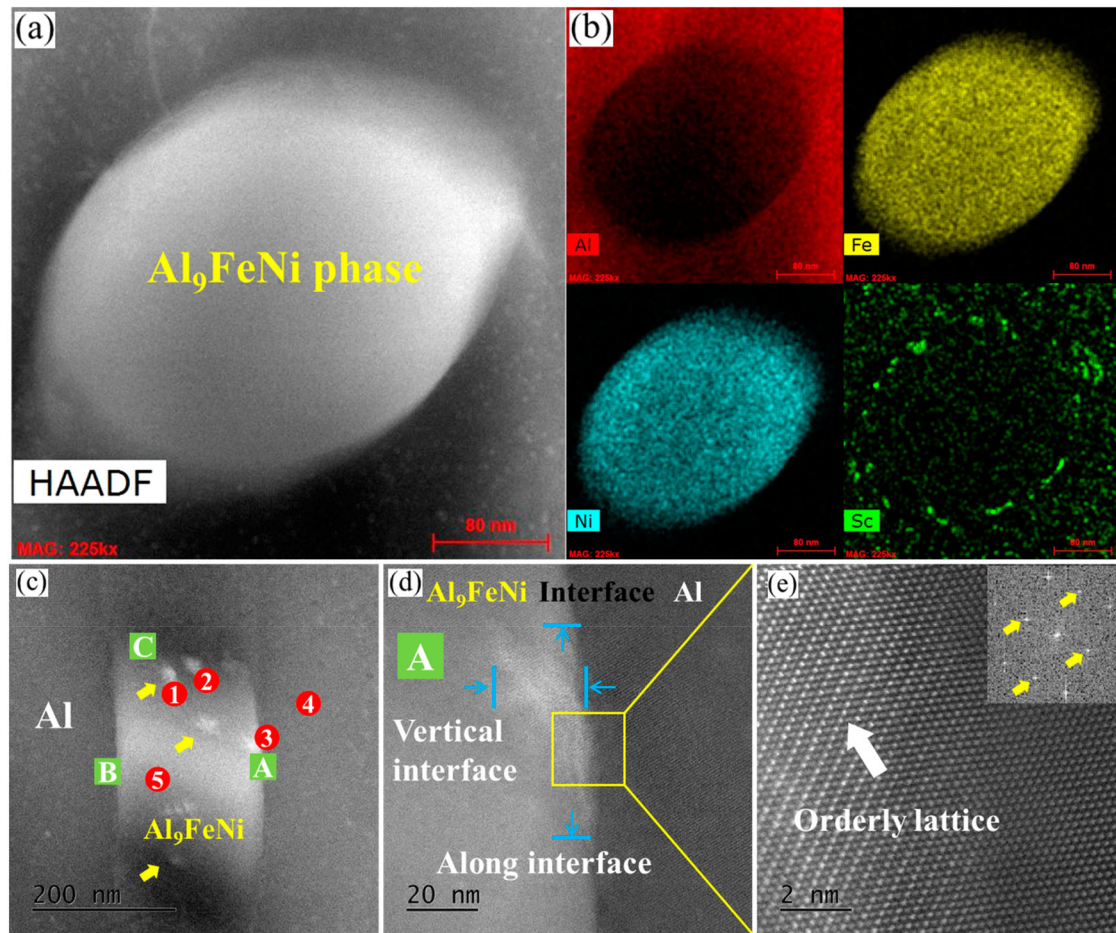


Figure 2. (a, c) Eutectic phase inside grains of the AlFeNi-0.2Sc alloy at peak-aged state; (b) Element distributions around the eutectic phase; (d) Overall morphology of the precipitate at Position A near the Al/Al₉FeNi interface; (e) HRTEM image at Position A.

sides of the plane. Therefore, the rotation angle between $(00\bar{1})_{\text{Al}_9\text{FeNi}}$ and $(11\bar{1})_{\text{Al}}$ planes may ensure more atom coplanar and the interface stability can be improved.

The top views of both $(11\bar{1})_{\text{Al}}$ and $(00\bar{1})_{\text{Al}_9\text{FeNi}}$ planes are presented in Figure 3(d,e), respectively. Thus, the vertical interface structure from $[11\bar{1}]_{\text{Al}}$ view is exhibited in Figure 3(f). Herein, the lattice structure is well matched at the local position (i.e. $[2\bar{1}0]_{\text{Al}_9\text{FeNi}} // [10\bar{1}]_{\text{Al}}$), and the local mismatch is low ($\sim 2\%$). However, there are some larger mismatches at another interface (i.e. $\sim 10\%$ between $[010]_{\text{Al}_9\text{FeNi}}$ and $[011]_{\text{Al}}$), and the relatively larger lattice distortion and higher storage distortion energy should exist at these positions. Thus, the Al lattice structure (Lower elastic modulus phase) should be stretched and affected by the Al₉FeNi lattice structure (Higher elastic modulus phase).

Accordingly, Figure 3(g) shows the interface structure after Sc precipitated at the Al/Al₉FeNi interface. Since Sc has the larger atomic size, its segregation at these locations (Indicated by Black Arrows) can alleviate local distortions. Correspondingly, this may also be the vital reason for the interface precipitation of Al₃Sc. During the

Table 1. Element contents (at%) of featured points at the eutectic phase and surrounding area of AlFeNi-0.2Sc alloy at peak-aged state.

Position	Al (at%)	Fe (at%)	Ni (at%)	Sc (at%)
1	91.21	3.67	2.95	2.18
2	95.74	0.88	1.32	2.06
3	84.71	0.55	8.51	6.23
4	100	/	/	/
5	83.07	10.07	6.87	/

heat treatment of the AlFeNi-0.2Sc alloy, Sc is attracted to precipitate at the Al/Al₉FeNi interface to reduce local distortion and improve interface stability [18,31].

Experienced 300°C creep test, both AlFeNi and AlFeNi-0.2Sc alloys exhibit optimized creep performance. The detailed creep results are provided in SM-Parts 3 and 4. In comparison with creep properties of reported heat-resistant Al alloys at peak-aged and over-aged states [16,17,20,26,27,32–35], the AlFeNi alloy shows similar creep ability with Al-Ni [32] and Al-Cu-Sc [20] alloys (Figure 4(a)). Critically, the creep resistance of AlFeNi-0.2Sc alloy at a peak-aged state is highly

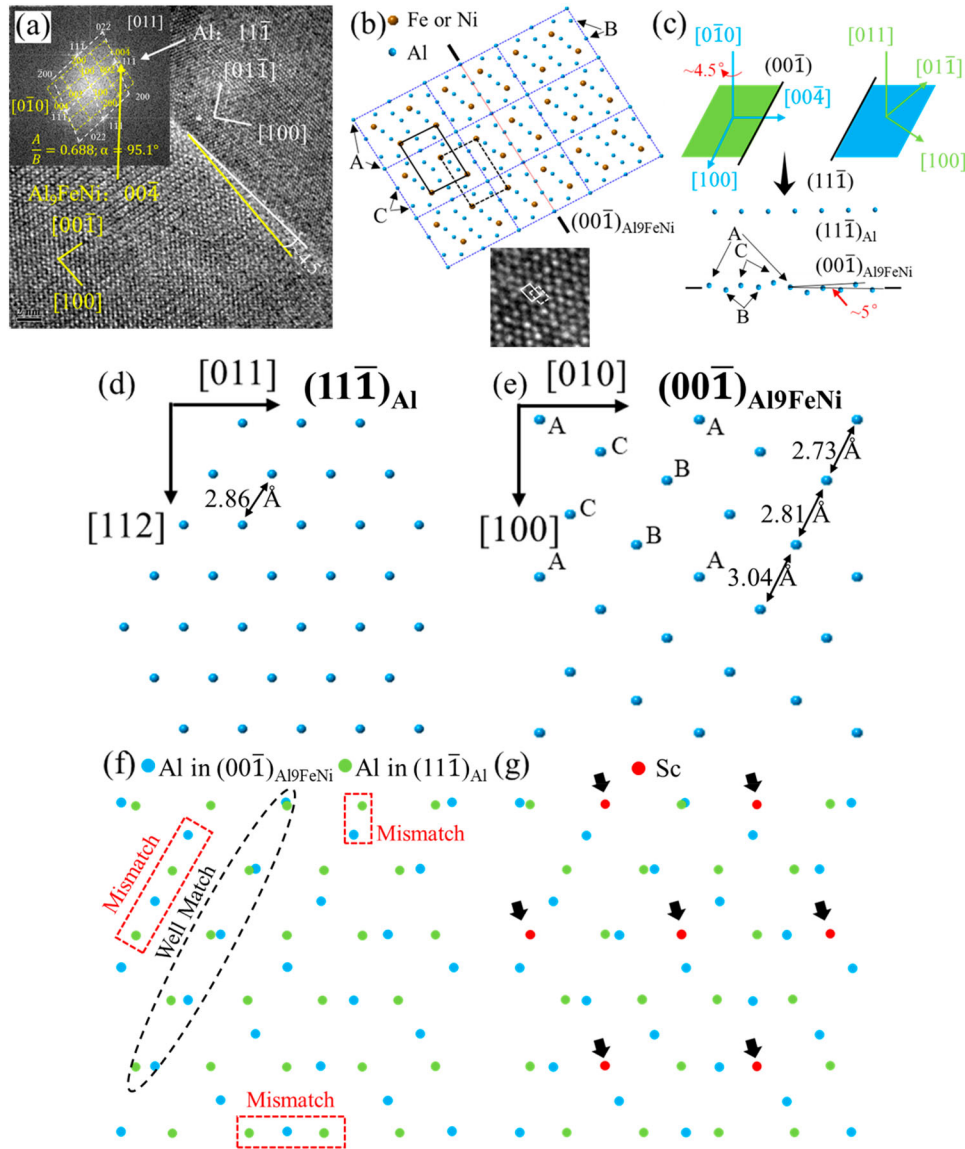


Figure 3. (a) Al/Al₉FeNi interface and crystalline orientation of each phase under this axis; (b) Lattice structure of Al₉FeNi under the [010] axis; (c) Schematic diagram of OR1; (d) Lattice structure of (111)_{Al}; (e) Lattice structure of (001)_{Al9FeNi}; (f) Interface structure from the side view; (g) Schematic diagram of Sc precipitation in the interface structure.

improved. For instance, it has a steady-state creep rate of $9.6 \times 10^{-10} \text{ s}^{-1}$ at 300°C@40 MPa, which is 1–4 orders of magnitude lower than other heat-resistant Al alloys. Although the creep property of AlFeNi-0.2Sc is inferior to that of Al-Ni-0.2Sc [21], the main reason is ascribed to the difference from the heat treatment method. This phenomenon can be explained by the research on the creep performance of Al-0.2Sc alloy treated in 300°C@6 h (Peak-aged state) or 24 h (Over-aged state) (Blue triangle in Figure 4(a)). Besides, the improved creep performance of Al-Ni-0.4Sc (Black hollow square in Figure 4(a)) over Al-Ni-0.2Sc alloy was also clear [26], which was ascribed to the higher precipitation strengthening effect to creep improvement induced by more Al₃Sc precipitates with higher Sc addition. Meanwhile, Ref. [26] also contained

a result that can support that the added Sc can cause a higher load transfer effect of the eutectic phase in the creep of the Al-Ni-Sc alloy, and this was not emphasized originally. However, this phenomenon complied well with our following in-depth analyses.

This work attempts to reveal the mechanism of Sc improving the creep properties of eutectic alloys. Therefore, the creep constitutive equation for dislocation creep of AlFeNi alloy at 300 °C (SM-Part 3) is modeled using a coupling modification [36] by introducing both threshold stress (σ_{th}) and load transfer coefficient (α):

$$\dot{\epsilon}_{300^\circ\text{C}}(\text{AlFeNi}) = A_1((1 - 0.68)(\sigma - 20.5))^4 \quad (2)$$

where $\sigma_{th-1} = 20.5 \text{ MPa}$; $\alpha_1 = 0.68$ (Figure 4(b)). Herein, the threshold stress originates from the interaction

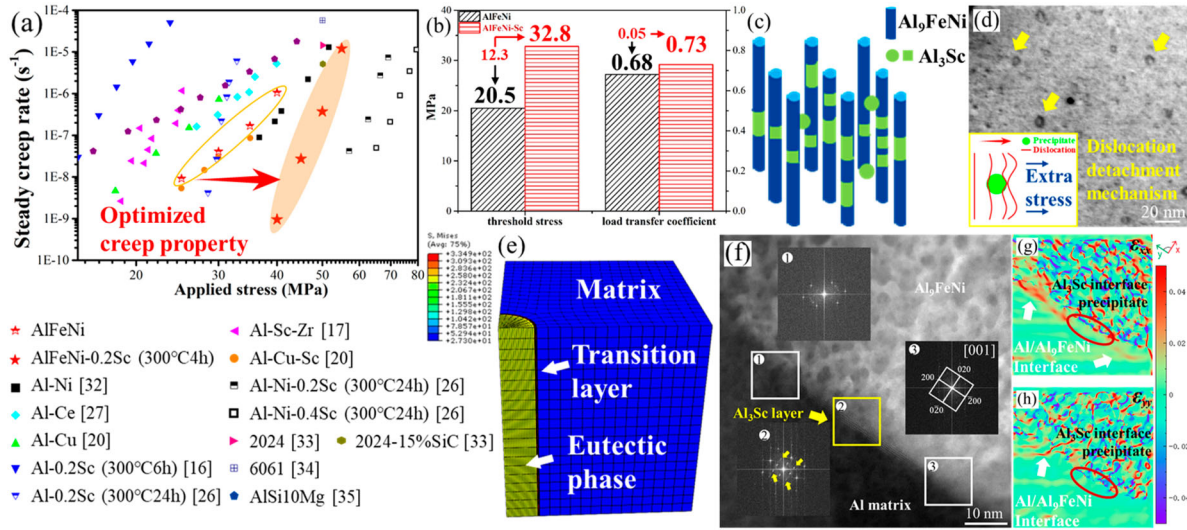


Figure 4. (a) Comparison of the creep behavior of Al alloys at 300°C; (b) Comparison of the dislocation creep behavior of AlFeNi alloy at 300°C; (c) Schematic diagram of Al₃Sc precipitates under peak aging; (d) Interaction between dislocation and matrix precipitates; (e) FEM Simulation of stress distribution after the formation of transition layer around the eutectic phase; (f) Al₃Sc precipitate layer around the eutectic phase and the lattice strain around the interface calculated by GPA; (g) Vertical interface; (h) Along interface (The white arrow points at the Al/Al₉FeNi interface; the red circle marks the Al₃Sc interface precipitate).

between Al₉FeNi and dislocations to impede creep. Besides, the load transfer effect is the main way of Al₉FeNi strengthens the matrix, due to its larger volume fraction and higher modulus [37].

Similarly, the creep constitutive equation of the AlFeNi-0.2Sc alloy at 300°C is calculated at SM-Part 4, and provided as

$$\dot{\varepsilon}_{300^{\circ}\text{C}}(\text{AlFeNi} - \text{Sc}) = A_2((1 - 0.73)(\sigma - 32.8))^4 \quad (3)$$

where $\sigma_{\text{th-2}} = 32.8$ MPa; $\alpha_2 = 0.73$ (Figure 4(b)). In addition to AlFeNi eutectic structures, Al₃Sc precipitates should offer an intensive strengthening effect on creep.

For threshold stress, AlFeNi-0.2Sc alloy has a much higher value than AlFeNi alloy. Since AlFeNi-0.2Sc alloy has a similar eutectic structure, the interaction between Al₉FeNi and dislocations for creep improvement is inherited thereby. Furthermore, the Al₃Sc precipitate also has a superior ability to hinder dislocation movement. For example, Seidman [16] found that the Al-0.2Sc alloy showed an increased creep performance than pure Al, and its threshold stress was ~ 14 MPa at 300°C. Similarly, Al₃Sc matrix precipitates (Figure 1(d)) can effectively impede dislocation movement (Inset in Figure 4(d)) in an alloy matrix. Generally, the threshold stress of AlFeNi-0.2Sc (32.8 MPa) is nearly equal to the sum of threshold stresses caused by eutectic phase (20.5 MPa ($\sigma_{\text{th-1}}$)) and Al₃Sc precipitates (14 MPa [16]).

Regarding the load transfer coefficient, the AlFeNi-0.2Sc alloy ($\alpha_2 = 0.73$) has a higher value over AlFeNi alloy ($\alpha_1 = 0.68$), indicating its higher contribution to

creep. Since both alloys have similar eutectic structures, more attention is paid to the Al₃Sc precipitate in the AlFeNi-0.2Sc alloy. For Al₃Sc matrix precipitate, there is available analysis confirming its ever contribution by the load transfer effect [16]. Therefore, the elevated load transfer coefficient should be accounted for more by Al/Al₉FeNi interface structure with interface precipitates, i.e.:

- (I) Macroscopically, Al₃Sc interface precipitates are regarded as part of the eutectic phase (Figure 4(c)), which is equivalent to an increased eutectic phase volume fraction. Meanwhile, finite element modeling (FEM) is to discuss stress distribution at the Al/Al₉FeNi interface with/without interface precipitates. In the AlFeNi-0.2Sc alloy, the Al₃Sc interface precipitates exhibit a certain length and thickness by microstructure characterization (Figure 2(c-e) and Figure S1). Although these interface precipitates do not form a continuous layer at the interface, they precipitate in large quantities hereby (Figure 2(a,b)). To discuss stress distribution around the Al₉FeNi/Al₃Sc/Al structure related to the load transfer effect of creep, the situation is very complicated. Therefore, the Al₃Sc interface precipitates are assumed to be a continuous layer for simplicity in the FEM model. Furthermore, a detailed discussion on this issue is included in SM-Part 5. The result shows that the eutectic structure with interface precipitates shares higher stress than only the eutectic structure during deformation (Figure 4(e) and Figure S5).

(II) Microscopically, Al_3Sc interface precipitates can alter the $\text{Al}/\text{Al}_9\text{FeNi}$ interface structure to affect the load transfer effect. Before the external stress applies to the alloy, there are differences in lattice parameters between Al_9FeNi and Al matrix that contributed to interface mismatch at interface structure (Figure 4(f–h)). Since both the lattice parameter and elastic modulus of the Al_9FeNi phase are larger than the Al matrix, the Al lattice is subjected to tensile stress to cause corresponding tensile strain through the interface structure. During transferring load process, the applied external stress causes the deformation of the Al matrix initially. Then, the eutectic Al_9FeNi phase sustains the load transferred by the interface structure. From coordination deformation, the eutectic phase co-shares the load from the matrix to restrain matrix deformation, and the actual matrix strain becomes smaller. Therefore, the initial tensile stress at the $\text{Al}/\text{Al}_9\text{FeNi}$ interface reduces load transfer efficiency. When Al_3Sc precipitates at the interface, Sc tends to aggregate at the position under tensile stress because an Sc atom has a slightly larger size than Al (Figure 3(g)). Furthermore, an HRTEM image of the $\text{Al}/\text{Al}_9\text{FeNi}$ interface with interface precipitates (Position 2) (Positions 1 and 3 are Al matrix) is shown (Figure 4(f)), and related lattice strains are calculated by GPA (Geometric Phase Analysis, Figure 4(g–h)). Herein, the Al matrix presents tensile stress, and it is alleviated by the Al_3Sc precipitate. Besides, the lattice strain around the Al_3Sc interface precipitate characterized by STEM and GPA analyses is made in SM-Part 6, which also supports the conclusion from Figure 4(f–h). Therefore, the Al_3Sc interface precipitate can optimize load transfer efficiency, and the load transfer coefficient is increased accordingly.

4. Conclusion

Microalloying Sc into AlFeNi eutectic can induce the formation of Al_3Sc precipitates at both the $\text{Al}/\text{Al}_9\text{FeNi}$ interface and Al matrix. Therefore, this novel AlFeNi-0.2Sc alloy shows superior creep properties over available reports. By coupling modification analyses on the creep constitutive equation, both threshold stress and load transfer coefficient are quantitatively acquired. In detail, threshold stress is increased by the Al_3Sc matrix precipitates by impeding dislocation movement, and the load transfer coefficient is improved by interface precipitates. Specifically, both microscopic and macroscopic factors for the interface structure with interface precipitates are analytically discussed to enhance the load transfer effect. Generally, this work provides a solid understanding to

improve the creep property of eutectic heat-resistant Al alloys by manipulating eutectic phase/matrix interface structure.

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Disclosure statement

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