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ORIGINAL REPORTS



Stabilizing nanoprecipitates in Al-Cu alloys for creep resistance at 300°C

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ABSTRACT

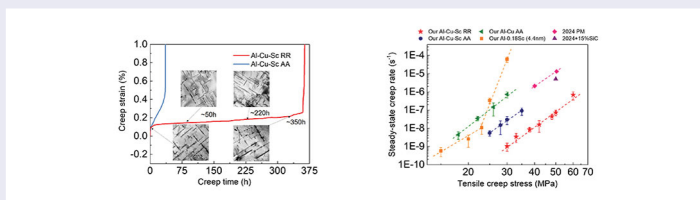
Commercial precipitation-hardened Al-Cu alloys are normally used at temperatures below its ageing temperature of $\sim 225^\circ\text{C}$, to avoid thermally induced precipitate coarsening and resultant softening. Making such popular Al alloys creep resistant at or above 300°C is thus challenging. Here we present a modified precipitation protocol, exploiting Sc-microalloying and a carefully designed three-step heat treatment to enhance Sc segregation at the matrix/ θ' -Al₂Cu precipitate interfaces. The stabilized nanoprecipitates enable an order-of-magnitude reduction in creep rate at 300°C , demonstrating the room for microstructure improvement and the potential for property elevation in traditional engineering alloys through innovative processing coupled with synergetic alloying elements.

ARTICLE HISTORY

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KEYWORDS

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IMPACT STATEMENT

A carefully designed precipitation protocol produces stable nanoprecipitates in Sc-microalloyed Al-Cu alloys, rendering an order-of-magnitude elevation of creep resistance at 300°C .

1. Introduction

Precipitation-hardened Al alloys are arguably the most popular lightweight engineering material for structural applications at room temperature [1–3]. They are also highly desirable for applications at elevated temperatures (such as 300°C) [4], for example in fuselage bulkheads and wing skins in high-performance military aircrafts, automotive engines, and heat-resistant conductors in power transmission, if the microstructure of these Al alloys can remain stable under moderately hot conditions. However, the challenge is that such a desired service temperature is considerably above the age-hardening temperature, which is $< \sim 225^\circ\text{C}$ for conventional Al

alloys to acquire strengthening precipitates [5–8], such as the θ' -Al₂Cu in the commercial 2000-series Al alloy based on Al-Cu [8–12]. This classical precipitation strengthening system is the basis for a wide range of age-hardening Al alloys used in cast conditions, and will be the base alloy of our study. At service temperatures such as 300°C , thermally induced diffusion of the alloying element (Cu, etc.) becomes so rampant that the θ' -Al₂Cu precipitates coarsen rapidly, leading to severe loss in strength [13,14].

A sensible approach is to add another minor alloying element, such as Sc, Zr and Er, that forms coarsening-resistant Al₃X (e.g. X = Sc, Er, etc.) precipitates [3,15–17].

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However, the formation of strengthening Al_3Sc requires an ageing temperature of 300–350°C, which is $\sim 100^\circ\text{C}$ higher than the θ' - Al_2Cu -precipitation temperature, thus coarsening and dissolving away the θ' - Al_2Cu precipitates. Owing to the rather limited solubility of Sc in Al, the Al_3Sc population is usually too low (volume fraction, $f_v < 1\%$) to make up for the yield strength drop due to the loss of the θ' - Al_2Cu precipitates ($f_v > 2\%$) [3,14,16–19]. Previously, several microstructural designs have been put forward in Al-Cu-based alloys, including more coarsening-resistant Ω phase [6,7,13] via Mg/Ag co-addition and synergistic coupling among multiple precipitates [8,12]. Recently, Sc segregation at the α -Al/ θ' interfaces has been shown to effectively suppress the coarsening of the θ' - Al_2Cu precipitates [18,19]. However, if the ageing temperature is kept low using the normal precipitation protocol for θ' - Al_2Cu precipitates, the precipitation time allowable before over-ageing would not be sufficiently long for the vast majority of the slowly segregating Sc atoms to reach the α -Al/ θ' interfaces to stabilize the fast-coarsening θ' - Al_2Cu precipitates (see

below). By the time the Al_3Sc form throughout the alloy upon heat treating, the fast-appearing θ' - Al_2Cu precipitates would have already formed some time earlier and coarsened.

As such, the traditional precipitation hardening routine falls short, when it comes to exploiting the majority of Sc solutes in the supersaturated Al-Cu-Sc solution, in binding them to θ' - Al_2Cu precipitates to suppress coarsening. To come up with a solution, we have been inspired by the process originally developed for the 7000-series Al alloys to achieve an optimal balance between strength and corrosion resistance, known as the Retrogression and Re-ageing (RRA) heat treatment [20,21], and multi-step/isochronal ageing in Al-Sc based alloys [3,16]. The highlight of our strategy is to apply a regression step to re-precipitate the θ' - Al_2Cu , after re-dissolving (akin to solution treatment) the θ' - Al_2Cu precipitates back into the matrix at an elevated temperature, a temperature high enough for the θ' - Al_2Cu precipitates to dissolve, but low enough such that the Al_3Sc precipitates stay put. Afterwards, the θ' - Al_2Cu precipitates are

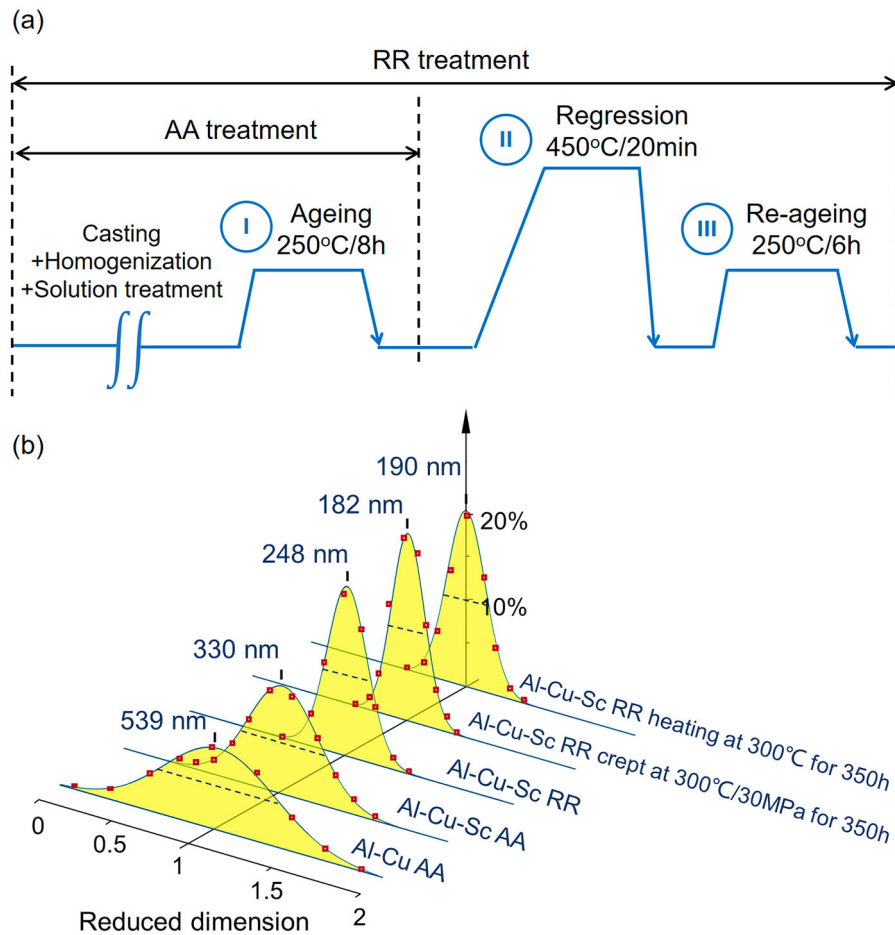


Figure 1. (a) Schematic showing the processing protocol. (b) Particle size distribution of θ' - Al_2Cu precipitates, comparing different heat treatments, as well as creep test versus heating at 300°C. The precipitate sizes (L) were measured using TEM after each treatment, with the average size L_{aver} marked for each distribution curve. The x-axis is in reduced unit, using normalized precipitate dimension L/L_{aver} .

Figure 2. Microstructural features of the Al-Cu-Sc alloy after each heat-treatment step and after creep test. Top row: schematic showing the microstructure developed in Step-I (a), Step-II (e), Step-III (i) and after creep test (m), with corresponding TEM images in (b), (f), (j) and (n), respectively. SAED pattern of Sc-enriched heterogeneities signed by red circle in (b) was given in top right corner. Corresponding 3D-APT maps showing Sc segregation at the α -Al/ θ' interface are in (c), (k) and (p), and Al_3Sc precipitates in (g) and (h), respectively. The bottom row are 3D-APT maps, showing Sc-enriched heterogeneities after Step I in (d), θ' - Al_2Cu precipitates nucleated on the Al_3Sc particles in (l) after Step III, and (p) Al_3Sc -PDZ after creep test. Note that an Al_3Sc forming on the α -Al/ θ' interface after crept was marked by arrow in (o).

3. Result and discussions

3.1. Step I (AA treatment)

The first and beginning step of our processing protocol, see Figure 1(a), after the solution treatment, was artificial ageing (AA) at 250°C, akin to the traditional T6 treatment for conventional Al alloy. This Step I forms θ' -Al₂Cu precipitates with an average size (half plate length) $L = 330$ nm, see Figure 2(a–b) and Table S2, smaller than in the Sc-free Al–Cu alloy under the same AA condition ($L = 539$ nm). Figure 1(b) compares the size distribution of θ' -Al₂Cu precipitates after different treatments (and tests), and indicates that the size distribution of θ' -Al₂Cu precipitates is the most uniform after applying our new precipitation protocol (see results later). A high density of Sc-enriched heterogeneities (precursors of Al₃Sc) were evident within the matrix (see Figure 2(d), referred to as ‘Sc clusters’ [18]), with core Sc concentration similar to Al₃Sc in previous investigations [15,17], see APT results in Figure 3(a). No coherency strain (Ashby–Brown contrast [16]) or lattice change was observed under TEM and HRTEM, nor diffraction spots indicative of Al₃Sc precipitates in the SAED pattern, see Figures 2(b) and 4(a). This is in contrast with the well-developed Al₃Sc particles that have easily identifiable crystal structure (Figure 4(b)). However, some weak diffraction patterns were observed in the FFT images, see Figure 4(a), possibly originating from some already formed Al₃Sc precipitate that are very small in size. This is in accord with the slow Al₃Sc transformation at 250°C [17]. The proxigram was displayed in Figure 3(b) to show the solute concentration across an α -Al/ θ' interface in the Al–Cu–Sc AA alloy. The solid vertical line represents the position of the coherent interface between the Al matrix and the θ' -Al₂Cu precipitate. A peak Sc concentration of about 0.8 at.% is detected at the α -Al/ θ' interface, about 10 times greater than in the matrix [18,19]. The interfacial Sc segregation layer is about 1–2 nm wide (FWHM of the concentration profile). However, the Al–Cu–Sc AA alloy still displays a rather low creep resistance (Figure 5) because Sc atoms segregated at the matrix/precipitate interfaces constitute of only about 2.6% of the total Sc atoms added in the alloy, see Table S3. Nearly 90% Sc atoms remain far away from the θ' -Al₂Cu precipitates, as in numerous locations the Sc atoms are accrued into precursors of Al₃Sc not in the close proximity of θ' -Al₂Cu precipitates. Sc diffusion is so slow such that during AA only a small number of Sc atoms manage to reach and deposit at the matrix/precipitate interfaces. As such, AA alone is insufficient for effective blocking of precipitate coarsening for satisfactory creep resistance.

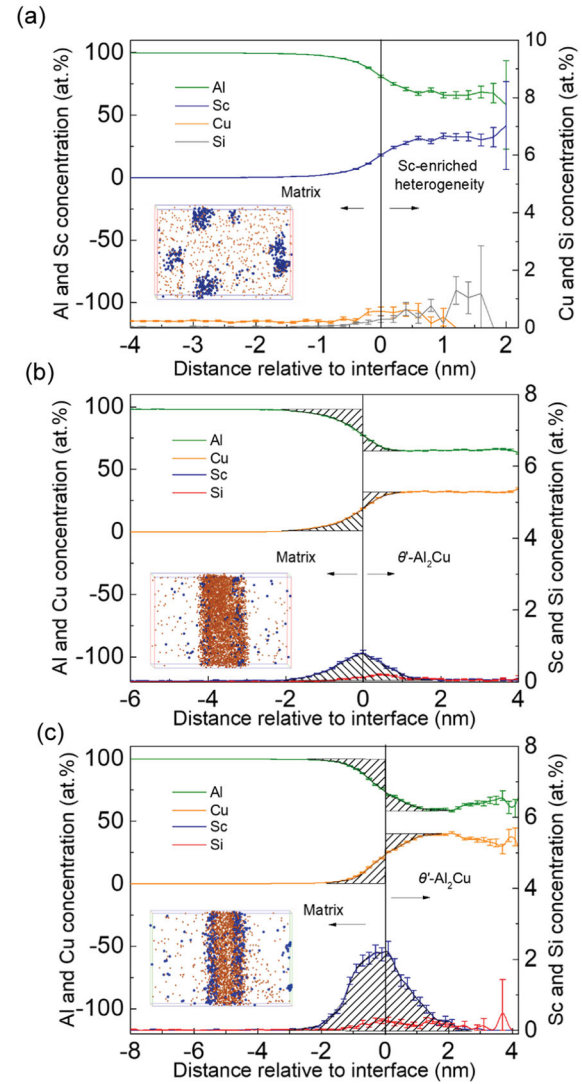


Figure 3. Representative proxigrams of Al, Cu, Sc and Si across (a) Sc-enriched heterogeneities, and α -Al/ θ' interface in the (b) Al–Cu–Sc AA alloy, and (c) Al–Cu–Sc RR alloy. The inset is the corresponding 3DAP image with dimensions of 20 × 20 × 30 nm³.

3.2. Step II (Regression)

During Step II, a regression treatment at elevated temperature of 450°C (Figure 1(a)), the θ' -Al₂Cu precipitates formed during the lower-temperature AA treatment would decompose, letting the Cu atoms dissolve back into the matrix (which was depleted to only ~ 0.2 at.% Cu after Step I), to get set to re-do their distribution in a subsequent step that takes them to the numerous Al₃Sc precipitates that form during this Step II. Now nano-sized coherent Al₃Sc precipitates are created at a high density from the Sc-enriched heterogeneities (Figure 2(d)). Figure 2(e–f) present a schematic and a TEM image, respectively, to show the Al₃Sc precipitates at a high number density. Corresponding 3D-APT images were

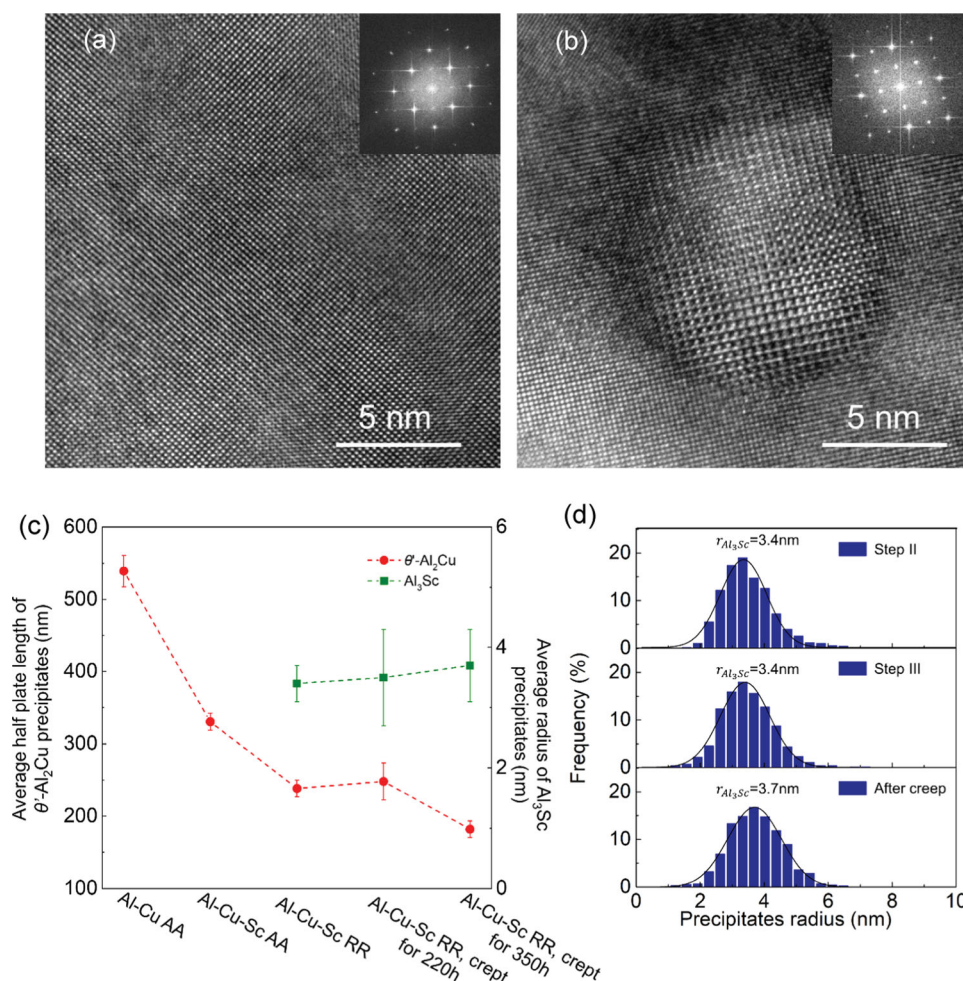


Figure 4. HRTEM image showing (a) Sc-enriched heterogeneities with no clearly identifiable crystal structure and (b) a representative coherent Al_3Sc particle. Corresponding FFT images were given in the upper right corner. (c) Statistical size evolution of Al_3Sc and θ' - Al_2Cu precipitates after Step II, Step III, and creep testing. (d) Size distribution of the Al_3Sc precipitates in the Al-Cu-Sc alloy after different treatment or creep test for ~ 350 h.

displayed in Figure 2(g and h). These Al_3Sc particles have an average radius of about 3.4 ± 0.8 nm, see Figure 4(d) and uniformly distribute in the matrix. A fraction of them will serve as potential preferred sites for nucleating copious θ' - Al_2Cu precipitates later on in Stage III, see Figure 2(l) and Figure S1.

We now point out that one cannot omit Step I and directly go to Step II. The necessity of Step I is because the lower-temperature AA pulls the Sc solutes out of its original uniform distribution in the matrix to become spread-out and populous precursors of Al_3Sc in the matrix. This happens because during AA, at the large undercooling the driving force is high for the resultant Sc-enriched heterogeneities to be small but numerous. During Step II at 450°C , the majority of the Sc-enriched heterogeneities rapidly grow to nano-sized Al_3Sc precipitates. In contrast, if one goes straightly to 450°C without Step I, the nucleation rate of Al_3Sc is low due to smaller undercooling such that the particles are lower in number density but

grow to fairly large sizes, jeopardizing the targeted copious heterogeneous θ' - Al_2Cu precipitation in Step III (see next paragraph). This is why the Step I is a prerequisite for the ensuing steps.

3.3. Step III (Re-ageing)

We are then in the position to reform the θ' - Al_2Cu precipitates in Step III, a re-ageing treatment at 250°C . As seen in the sketch and TEM image in Figure 2(i-j), the re-precipitated θ' - Al_2Cu plates have a number density more than twice that of the AA treatment, because of the high density of (Sc-)heterogeneous nucleation sites sowed in Step II, see Figure 2(l) and Figure S1. In detail, a clear orientation relationship can be detected when the θ' - Al_2Cu heterogeneously nucleate on the Al_3Sc [8,12,19]. Such nucleation assistance is efficient, whereas during AA there are only occasional clusters with neither clear crystal structure nor orientation

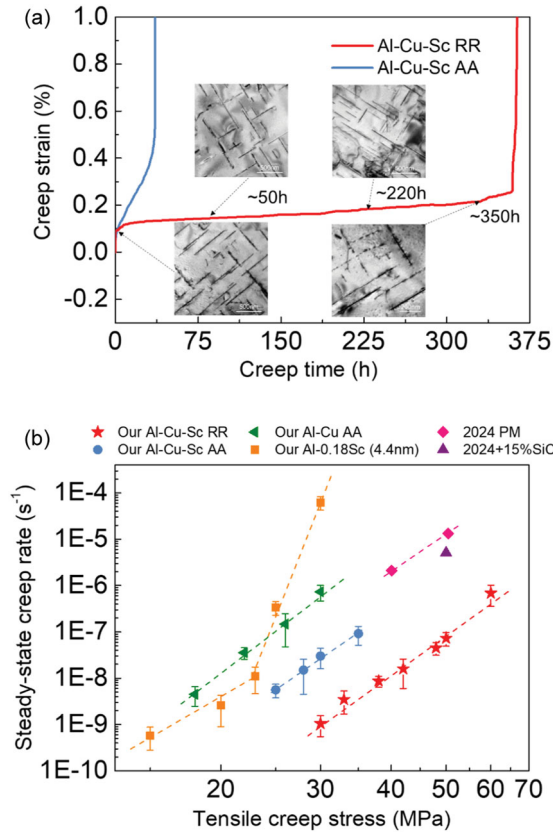


Figure 5. (a) The tensile creep curves of Al-Cu-Sc AA and Al-Cu-Sc RR alloys, all tested at 300°C/30 MPa. The θ' -precipitates remain largely stable, as seen in the TEM images (insets) after different creep time periods. (b) Stress-dependent steady-state tensile creep rates of Al-Cu-Sc RR alloy tested at 300°C are highlighted using red stars, in comparison with Al-Cu-Sc AA alloy (blue circles), Al-Cu AA (olive left triangles) and Al-Sc AA ($r_{Al_3Sc} = 4.4\text{nm}$, orange squares). Available literature data of Al-Cu-based alloys crept at 300°C under *tensile* loading are given for comparison, including 2024 Al alloy (powder metallurgy, PM) [9] or reinforced with 15 vol.%SiC [25] (pink diamonds and purple triangles, respectively).

relationship (Figure 4(a)). The distributed θ' -Al₂Cu precipitates with a high density accomplish our goal (i) to uniformly spread out abundant precipitates for strengthening.

We next demonstrate the very important realization of goal (iii), i.e. quick and ample Sc segregation to intercept the coarsening of these θ' -Al₂Cu precipitates. The Sc segregation at α -Al/ θ' interfaces is remarkably enhanced after Step III. Figure 2(k) show a representative element maps cross a α -Al/ θ' interface with an average peak Sc concentration of $2.35 \pm 0.71\text{ at.}\%$, which is about three times of that in the AA condition, see Figure 3(b and c). The interfacial energy as driving force for precipitate coarsening [22,23] is further reduced due to the greater Gibbsian interfacial excess [15,24], Γ_{Sc}^{rel} , induced by enhanced interfacial Sc segregation (see SI for more

discussions). In the meantime, kinetically this interfacial Sc aggregation is likely to slow down the Cu atom diffusion cross the interfaces, delaying the dissolution or growth of the precipitates. The overall result is a fine θ' -Al₂Cu precipitate size at $\sim 250\text{ nm}$ after Step III, see Figure 1(b), 25% smaller than after AA treatment. More importantly, counting together the interface Sc (12.8%) and Al₃Sc particles (66.0%), overall $\sim 80\%$ of the total Sc atoms in this alloy have been exploited to influence the strength and creep resistance of the alloy (see Table S3).

During Step III, what makes the Sc enrichment at the α -Al/ θ' interfaces far more pronounced and rapid than in AA is the Sc supply from our intentionally distributed Sc-enriched heterogeneities transforming into Al₃Sc particles that are now either in contact with the θ' -Al₂Cu precipitates or very close by. To begin with, the θ' -Al₂Cu precipitates initiate preferentially at some of these Sc-enriched locations, where the Sc is already available. When many θ' -Al₂Cu precipitates appear, the very small ones coarsen into a larger one, such as the examples in Figure S2. Previous reports [19] have demonstrated that an increased Cu content leads to the decreasing volume fraction of Al₃Sc dispersoids. The Sc attached to many tiny precipitates migrate to and join the larger Sc patches at the α -Al/ θ' interface. As such, Al₃Sc precipitate-depleted zones (PDZs) form both ahead and around the growing θ' -Al₂Cu precipitates, as marked in Figure 2(j). All these constitute fast supply of Sc atoms towards the matrix/precipitate interfaces, quickly enclosing the θ' -Al₂Cu precipitate and inhibiting its further coarsening. The eventual Sc segregated to the α -Al/ θ' interfaces amounts to $\sim 13\%$ of the total Sc atoms in the alloy, almost quintupling that achieved after the AA treatment. This sizable fraction is due to the more but smaller θ' -Al₂Cu precipitates compared with the AA case, and the high interfacial Sc concentration ($2.35 \pm 0.71\text{ at.}\%$), which on average is three times that of the AA case.

3.4. Stabilized nanoprecipitates contribute to much reduced creep rates

As seen in Figure 1(b) and Table S2, our three-step treatment makes the θ' -Al₂Cu precipitates far more stable than conventional AA counterpart due to improved Sc availability and segregation. Estimates using thermodynamic calculations in SI suggest a $\sim 65\%$ reduction in the interfacial energy due to interfacial Sc segregation, reducing precipitate coarsening [22,23] in the Al-Cu-Sc RR alloy, in comparison with the $\sim 24\%$ in its AA counterpart. For plate-like θ' -Al₂Cu precipitates to grow, the interface-controlled thickening [23] fed by the flux of Cu from the matrix to the migrating ledge now necessarily requires the redistribution of Sc. During the creep

test, the average size of θ' -Al₂Cu precipitates was in fact almost unchanged, remaining at about 250 nm at 300°C/30 MPa for ~ 220 h, see Figures 1(b) and 5(a). TEM examinations also reveal that the half length of θ' -Al₂Cu actually decreased to ~ 180 nm after creep test for 350 h, as a result of splitting in some of the θ' -Al₂Cu precipitates, with only a slight drop of volume fraction f_v from $2.13 \pm 0.19\%$ to $1.97 \pm 0.10\%$.

The secondary stage of the Al-Cu-Sc RR alloy now holds on for as long as ~ 350 h, a 10-fold rise in endurance time than its AA counterpart, see Figure 5(a) at 30 MPa. Figure 5(b) summarizes the creep rate data in the tensile stress range of 30–60 MPa; the present Al-Cu-Sc RR alloy sustains a creep rate several orders of magnitude slower when compared with binary Al-Cu or Al-Sc alloys, and Al-Cu based alloys and composites. Additional discussions are presented in SI on precipitate stability, creep mechanism (Fig. S4) and high-temperature tensile properties. For the latter, regular tensile test of the Al-Cu-Sc RR alloy shows a yield strength of 127 ± 12 MPa at 300°C, which is much higher than that of the Al-Cu AA (59 ± 5 MPa) and Al-Sc AA (34 ± 2 MPa) alloys, and close to some commercial alloys (*i.e.* 7075, 2219 Al). This short-term strength of the present Al-Cu-Sc RR alloy is nevertheless lower than the ~ 200 MPa yield strength at $\sim 300^\circ\text{C}$ for Al-Cu alloys with high Cu content, such as Al-6.3Cu-0.5Mg-0.5Ag-0.5Mn-0.2Zr (their long-term microstructure stability and creep rates at 300°C are not available for a direct comparison) [6,13].

We note that in addition to θ' -Al₂Cu (protected by Sc segregation), important contribution to strengthening also arises from the Al₃Sc particles (see an estimate in the SI). Our RR protocol combines these two contributing factors and exploits both types of precipitates.

4. Conclusions

In summary, for Al-Cu based alloys we have modified the precipitation protocol to boost the thermal stability of strengthening precipitates at preferred sizes for creep resistance at 300°C under relatively high tensile stresses. Specifically, our deliberate integration of three heat-treatment steps, taking advantage of the interaction and synergy between the main alloying element Cu and minor alloying element Sc, generates dispersion of θ' -Al₂Cu precipitates with a combination of high density, uniform size distribution, and efficient use of Sc sources to quickly collect Sc to the α -Al/ θ' interfaces to stabilize the θ' -Al₂Cu precipitates at desirable sizes to delay runaway coarsening. The mechanistic insight gained through this work is that one can strategically exploit two different alloying elements to deliver a coexisting and

coupled (θ' -Al₂Cu precipitates + Al₃Sc particles) nano-precipitate microstructure that performs better in terms of creep resistance. These unconventional microstructures give rise to order-of-magnitude reduction in creep rate, over previous Al-Cu based alloys. Our discovery extends the repertoire of cast Al-Cu alloys for potential use at service temperatures as high as $\sim 300^\circ\text{C}$.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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