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ORIGINAL REPORT



Ultrahigh strength and plasticity in laser rapid solidified Al–Si nanoscale eutectics

Huai-Hsun Lien ^a, Jyoti Mazumder ^{a,b}, Jian Wang ^c and Amit Misra ^{a,b}

^aMaterials Science and Engineering, University of Michigan, Ann Arbor, MI, USA; ^bMechanical Engineering, University of Michigan, Ann Arbor, MI, USA; ^cMechanical and Materials Engineering, University of Nebraska-Lincoln, Lincoln, NE, USA

ABSTRACT

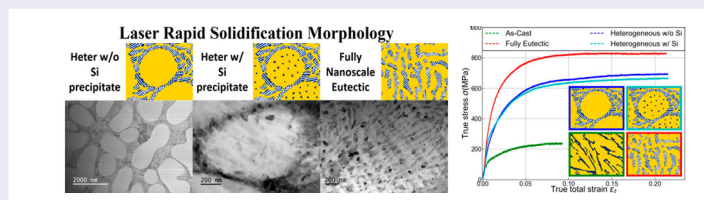
As-cast Al–20wt.% Si alloys were processed via laser rapid solidification (LRS) techniques to create eutectic microstructures with nanoscale interconnected, nanotwinned Si fibers. LRS morphologies exhibit higher flow stress, exceeding 800 MPa and uniform plastic deformation above 20% compared to as-cast alloy that fractures at strains below 8% at flow strength of approximately 200 MPa. The strengthening mechanisms of LRS morphologies are interpreted in terms of the interfacial constraints: increase in yield strength as well as strain hardening rate due to nanoscale confined slip in fibrous Al–Si eutectic, and load transfer and eventual plasticity in the nanoscale Si fibers.

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KEYWORDS

Al–Si eutectic; Laser rapid solidification; Nanotwinned Si fiber; Heterogeneous microstructure



IMPACT STATEMENT

Interconnected, nanotwinned Si fibers in hypereutectic Al–Si alloy achieved by LRS increase the flow stress to over 800 MPa while maintaining homogeneous plastic deformation to over 20% strain, due to confined slip in nanoscale eutectic that increases yield strength and strain hardening with reducing size and promotes plastic co-deformability between disparate phases.

1. Introduction

Al–Si alloys are employed widely in structural applications due to their balanced mechanical properties [1], low cost and good casting ability. However, excess coarse Si flakes produced by conventional casting adversely affect the ductility [2,3]. With the aim to increase ductility, limited success in refining Si phase had been achieved through adding rare earth element [4,5], rapid quenching such as melt spinning [6] and spray deposition [7] as refinements are limited to micrometer-scale. Recently, ultrafast-cooling laser-processed Al–Si alloys have gained attraction because of the ultrafine Si phases formed [8–10] and the ability to fine-tune the microstructure [11]. Selective laser-melted (SLM) Al–12Si showed enhanced ductility with tensile strength of ≈ 200 MPa [9]. However, the Si phase by SLM remained relatively coarse at few hundred nm, which

could be further refined for improved strength and ductility [12,13]. Enhanced plasticity in room temperature-rolled nanolamellar Al–Al₂Cu [14] was attributed to slip transmission enabled by orientation relationship. In the ternary Al–Al₂Cu–Si system, increased plasticity was attributed to bimodal morphology [15,16], whereas in commercial A356 hypoeutectic Al–Si alloy [17], the flow strength was increased to ≈ 300 MPa using aging heat treatment to produce nanoscale precipitates. This study is focused on the understanding of deformation mechanisms that enable high flow strength and uniform plasticity in nanoscale eutectic morphologies.

In this work, cylindrical micropillars with different microstructure morphologies—one as-cast eutectic and three laser rapid solidification (LRS)—were compressed via nanoindentation at room temperature. Post-mortem SEM and S/TEM analyses reveal

CONTACT Huai-Hsun Lien hhlien@umich.edu

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the deformation mechanisms unique to heterogeneous eutectic microstructures with nanoscale hard/soft phases.

2. Methods

The fabrication of specimens could be found elsewhere [11]. Cylindrical micropillars with 5 μm diameter and 15 μm height were fabricated using FEI Helios 650 Nanolab system with computerized scripted procedure. The height to diameter ratio was chosen to be < 3 to prevent plastic buckling of micropillars [18], the size effect can be ignored. Compression tests were conducted using a Hysitron TI 950 Triboindenter with a spherical probe (a spherical segment with 50 μm in radius and 45° contact angle) with the following conditions: nominal strain rate of 0.2%/s (30 nm/s constant displacement rate) and total nominal strain at 20% (maximum displacement of 3000 nm).

3. Results

3.1. Characterization of microstructures pre-compression

One major distinction between LRS and as-cast morphologies is the geometry and scale of Si phase: interconnected and nanotwinned Si fibers with diameter ranging from 45 to 65 nm are formed after LRS [11] due to ultrafast-cooling [19]. Figure 1 shows the micropillar dimension and the corresponding morphologies: (i) as-cast Al–20 wt.%Si eutectic (Figure 1(a)) with Si flake in Figure 1(e), (ii) LRS heterogeneous Al–Si (Figure 1(b)) with micro-scale Al grains embedded in nanoscale Al–Si fibrous eutectic in Figure 1(f), (iii) LRS heterogeneous Al–Si similar to (ii) but with faceted Si nanoprecipitate, with size ranging from 10 to 50 nm, in Al dendrites in Figure 1(c,g), and (iv) LRS fully eutectic nanoscale Al–Si (Figure 1(d)) with interconnected Si fibers in Figure 1(h). The nominal composition of all LRS samples is Al–16 wt.%Si, slightly less than the as-cast Al–20 wt.%Si. The internal structure of Si fibers contain stacking faults and nanotwins, that were either on a single $\{111\}$ twin boundary (TB) creating a nanolamellar nanotwinned structure within the Si fiber in Figure 1(i), or multiple intersecting $\{111\}$ TBs in Figure 1(j). These two types of nanotwinned Si fibers are frequently found juxtaposed to each other within the LRS Al–Si eutectic. Detailed morphological studies of LRS Al–Si specimen could be found elsewhere [11].

3.2. Compressive stress–strain behavior

Figure 2(a,b) show the true stress—true strain curves of the different microstructure morphologies. LRS

micropillars exhibit more than 2.5 times higher flow stress than as-cast micropillar. Furthermore, LRS micropillars exhibit uniform plastic deformation to plastic strains $> 20\%$, whereas the as-cast micropillar exhibited cracking at $\approx 8\%$ true strain.

Comparing the maximum compressive flow strength of different microstructures: fully eutectic nanoscale Al–Si exhibits the highest flow stress at 828 MPa followed by heterogeneous Al–Si at 691 MPa, heterogeneous Al–Si with Si nanoprecipitate at 668 MPa, and as-cast at 237 MPa. The as-cast micropillar show little strengthening effect from Si flakes, as the flow strength was not enhanced significantly from 220 MPa for monolithic Al micropillar (500 nm diameter and 1 μm height) compressing in $[111]_{\text{Al}}$ direction [20].

The compressive behavior observed for heterogeneous Al–Si with and without Si nano-precipitates was similar, indicating that the Si nano-precipitates had little effect on the stress–strain response. Presumably, since the Si nanoprecipitates in the Al dendrites were relatively coarse, and the density of Si nano-precipitates was too low. In addition, due to the presence of the coarse primary Al dendrites, both heterogeneous structures exhibited lower yield strength compared to the fully eutectic nanoscale Al–Si.

Strain hardening rate θ vs. true plastic strain is shown in Figure 2(c), with θ higher for LRS morphologies. The strain hardenability is higher in LRS morphologies as compared to the coarse Al–Si as-cast alloy due to nanoscale confinement of Al phase that leads to single dislocation arrays, whereas the coarse Al grains in as-cast alloys exhibit the normal strain hardening as in bulk Al limited by the easy cross-slip.

3.3. Microstructure of compressed micropillars

Figure 3 shows the micropillars after compression, one salient feature is the absence of micro-scale cracks in LRS micropillars, whereas the presence of micro-cracks in the as-cast micropillar is observed in Figure 3(a,d). Cracks indicate incompatible deformation and adversely affect the plasticity [2,3]. However, LRS micropillars show signs of plastic co-deformability between Si and Al in Figure 3(b,c).

In LRS heterogeneous Al–Si morphology, the nanoscale eutectic restricted the deformation of Al dendrites as in Figure 3(b,e). After compression, the Al dendrites on the surface protruded out of the micropillar. This protrusion is a result of different deformation degree in the lateral direction (normal to the compression axis) between the two phases. As Al dendrites are embedded within nanoscale Al–Si eutectic, the micropillar surface is the only region without nanoscale eutectic constraints.

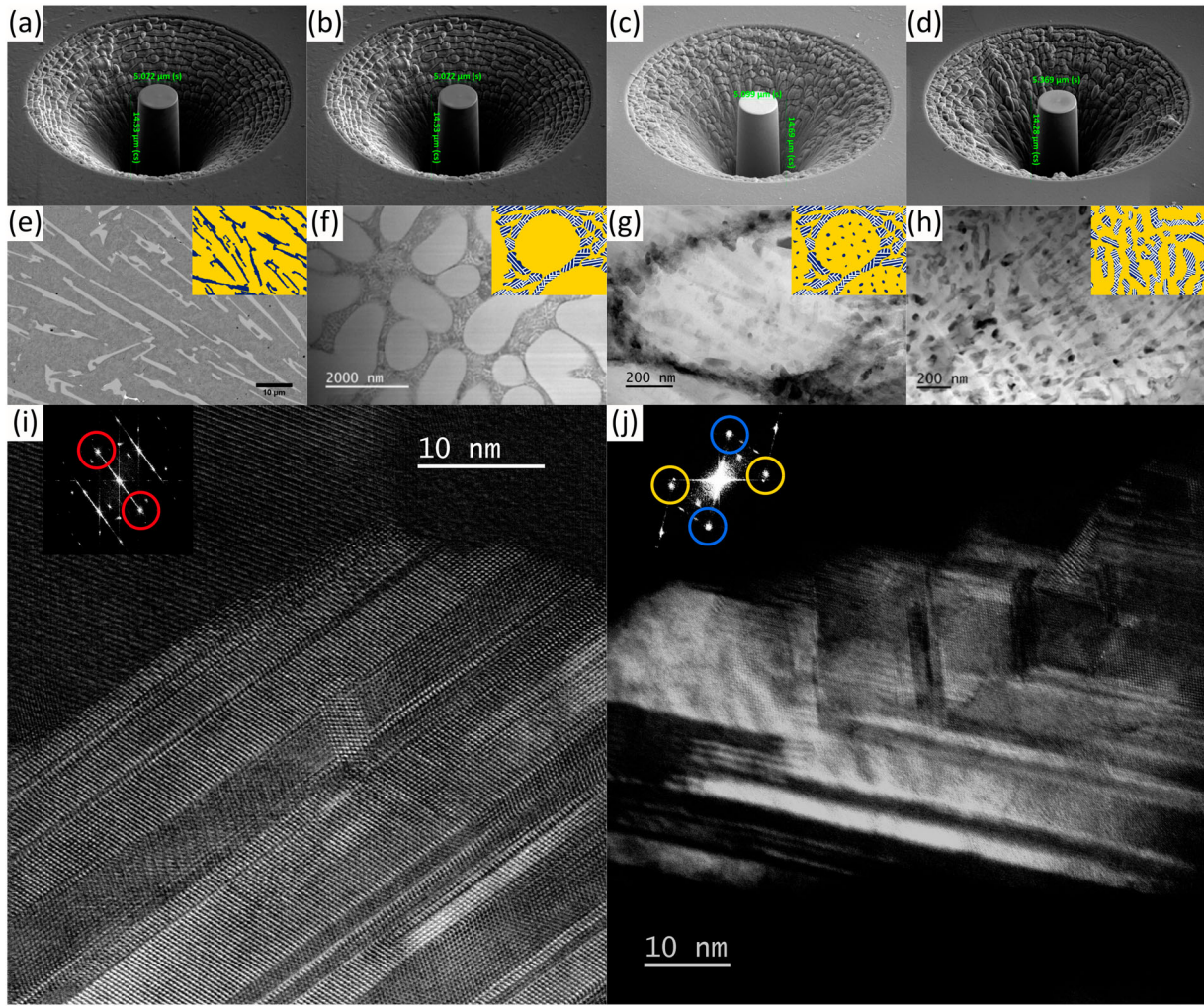


Figure 1. Pre-compression characterization of (a,e) As-cast Al–20Si. (b,f) Heterogeneous Al–Si with Al dendrites embedded in nanoscale Al–Si fibrous eutectic in LRS alloy. (c,g) same as (b,f) but contained Si nano-precipitates in Al dendrites. (d,h). Fully eutectic nanoscale Al–Si fibrous eutectic morphology in LRS alloy. The image (e) is SEM BSE while images (f–h) are STEM BF. (i) STEM HRTEM showing Si fiber with lamellar nanotwins, the inset shows the FFT with streaking primarily on the $(1\bar{1}\bar{1})_{\text{Si}}$ TB. (j) STEM HAADF of intersecting nanotwinned Si fiber, with the inset FFT showing streaking on both TBs $(111)_{\text{Si}}$ and $(1\bar{1}\bar{1})_{\text{Si}}$.

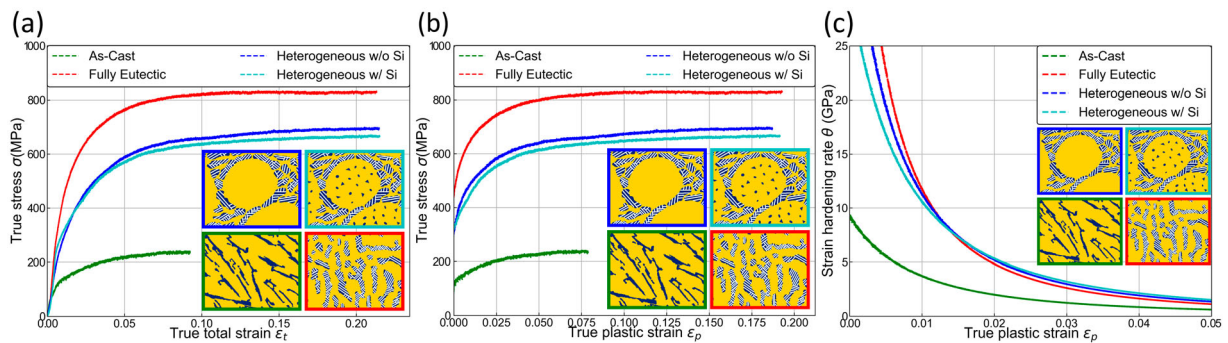


Figure 2. (a). True stress–true *total* strain curve for each morphology. For clarity, the data after the first significant load drop were excluded. (b). True stress–true *plastic* strain curve for each morphology. (c). Strain hardening rate θ vs. *plastic* strain curve. The stress–strain curves and the schematic are color coded.

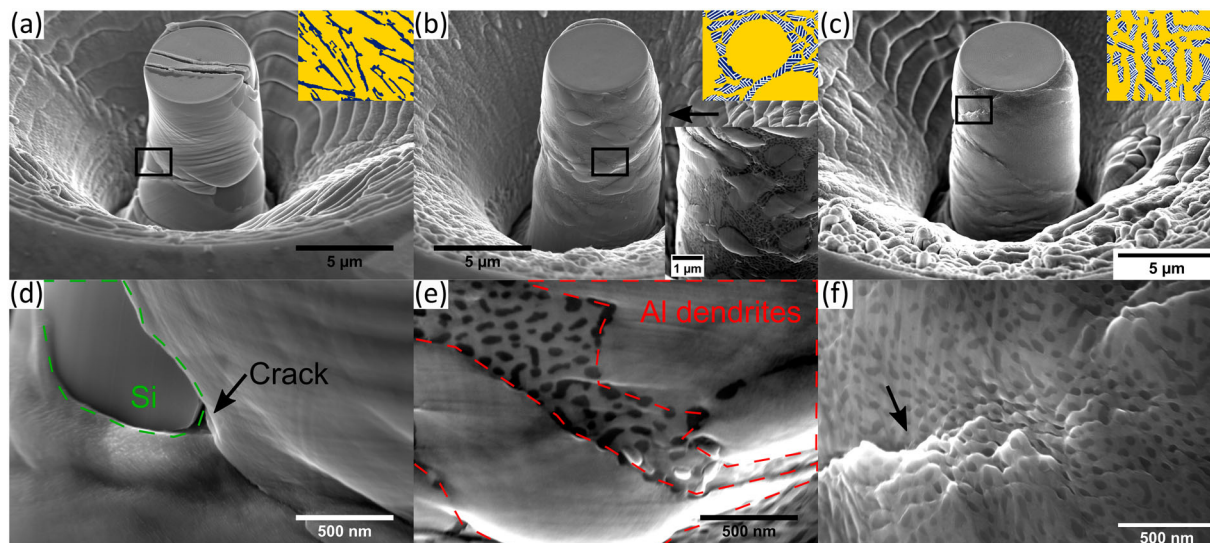


Figure 3. SEM of (a) As-cast eutectic, (b) LRS heterogeneous Al–Si w/o Si nano-precipitates with inset viewing from the arrow direction, and (c) LRS fully eutectic nanoscale Al–Si. (d). Cracks between Al phase and Si flake suggesting incompatible deformation between the phases. Protrusion of Al dendrites in (e) suggests nanoscale Al–Si eutectic are effective in constraining the deformation of the softer Al dendrites. (f). Fully eutectic nanoscale Al–Si displayed wavy surface relief absent of cracks.

For fully eutectic nanoscale Al–Si micropillar, the deformation manifested as wavy structure on the surface in Figure 3(c,f). In addition to absence of cracks, the cylindrical shape was maintained during compression, which suggests that this wavy slip promotes uniform load distribution across the micropillar by suppressing the taper caused by compression.

TEM analysis show high density of dislocations clustered near the Al–Si interface in as-cast micropillar in Figure 4(a–c). However, the deformation is confined within Al phase as no dislocations were observed in the Si flakes. As dislocations in Al phase are unable to cut through the Si flakes, the as-cast microstructures exhibit cracking at low plastic strains. Furthermore, despite slip in Al blocked by Si flakes, the relatively coarse as-cast structure (inter-flake distance around 10–20 μm) does not produce any significant strengthening. Thus, the high dislocation density at Al–Si interface is instead favorable for void formation.

In heterogeneous structure with Si nano-precipitates, dislocation sub-structure formation was observed in the coarse Al dendrites as shown in Figure 4(d,e). Moreover, the Si nano-precipitates appear to pin the glide dislocations in Figure 4(f). However, the flow stresses of the heterogeneous structures are comparable with and without Si, suggesting that the strength is dominated by the nanoscale eutectic and the hardening contribution from the coarser Si precipitates was modest.

Absence of cracking in LRS micropillars (from Figure 3) suggests plastic co-deformation for nanoscale Al–Si eutectic. HRTEM imaging and IFFT analysis reveal extra

half planes from the edge components of dislocations and has been used to demonstrate dislocation activity in hard TiN in indented Al–TiN nanoscale multilayers [21]. IFFT in Figure 4(g,h) reveal much higher density of extra $\{100\}_{\text{Si}}$ compared with pre-compression in Figure 4(i), suggesting dislocation accommodation in Si fibers is possible. This accommodation promotes uniform plastic deformation as dislocations could be evenly distributed as opposed to concentrating along localized bands, similar to observations in nanolayered Al–TiN [13] and Al–Al₂Cu [22].

4. Discussion

Post-mortem analysis of deformed pillars suggests that heterogeneous Al–Si microstructures promote the plastic co-deformation between Si fibers and Al as schematically shown in Figure 5. First, plastic deformation via dislocation pile-ups and multiplication commences in the soft-phase Al dendrites as in Figure 5(a), leading to the formation of statistically stored dislocations (SSDs) within the Al dendrite. Deformation incompatibility between Al dendrites and the hard nanoscale Al–Si eutectic contributes to geometrically necessary dislocations (GNDs) formation, which promotes back-stress strengthening and strain hardening for Al [23]. With increasing strain, dislocation density within Al dendrites saturates as the repulsive strength between dislocations approaches critical value for dislocation pile-ups [24].

Secondly, local high stress associated with dislocation pile-ups facilitates nucleation and glide of dislocations

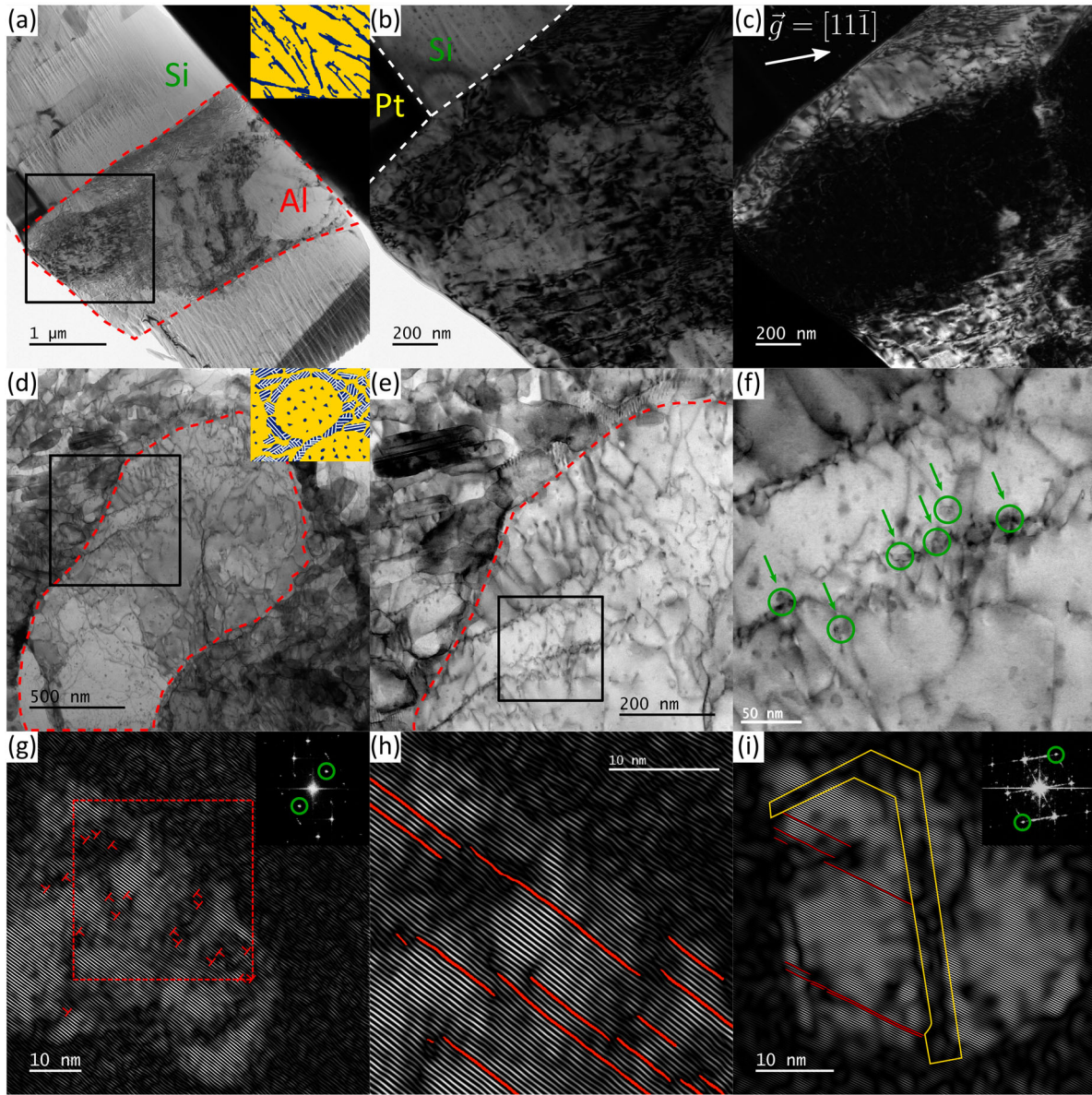


Figure 4. TEM after compression of as-cast sample in (a–c) and STEM of heterogeneous Al–Si in (d–f). (a) As-cast sample with darker contrast in the middle corresponds to Al phase. (b) Enlarged TEM BF of the interface. (c) TEM DF showing that dislocations in Al phase concentrate along the Al–Si interface. (d) Heterogeneous Al–Si with Si nanoprecipitate in Al dendrites. (e) Enlarged image of area highlighted in (d) shows high density of dislocations in Al dendrites. (f) Si nano-precipitates pinning the dislocations. (g) Extra $\{100\}_{\text{Si}}$ planes were observed and rendered in red in (h). (i) Pre-compression Si fiber show much lower density of extra $\{100\}_{\text{Si}}$ planes, with faults highlighted in yellow.

into the Al matrix of ultrafine Al–Si eutectic as in Figure 5(b). In Al matrix confined by Al–Si interfaces, dislocation pile-ups are unlikely and the glide is hypothesized to occur via single dislocation arrays on closely spaced glide planes [25]. The confined dislocations therefore have high glide stresses that scale inversely with the spacing between adjacent Al–Si interfaces [26,27].

The flow stress for moving dislocation in Al matrix between nanoscale Si fibers could be estimated through the combination of confined layer slip model [27] and the

strengthening effect from GNDs: [23,28]

$$\sigma_{\text{flow}} = M \frac{Gb}{8\pi t} \frac{(4-\nu)}{(1-\nu)} \ln \left(\alpha \frac{t'}{b} \right) + \alpha' Gb \sqrt{\frac{2\sqrt{3}\varepsilon_p}{bt}} \quad (1)$$

where Taylor factor $M = 3.1$, $G = 26.1$ GPa (converted from $E = 70.4$ GPa) [29], $b = 0.286$ nm ($a_2/2(110)_{\text{Al}}$), the core cutoff parameter $\alpha = 0.6$, $\nu = 0.34$, interfiber spacing $t = 43$ nm, projected length of slip plane $t' = t / \cos 45^\circ$ nm where 45° is the angle between $\{111\}_{\text{Al}}$ and interface normal, and $\alpha' = 0.2$. The calculated curve is

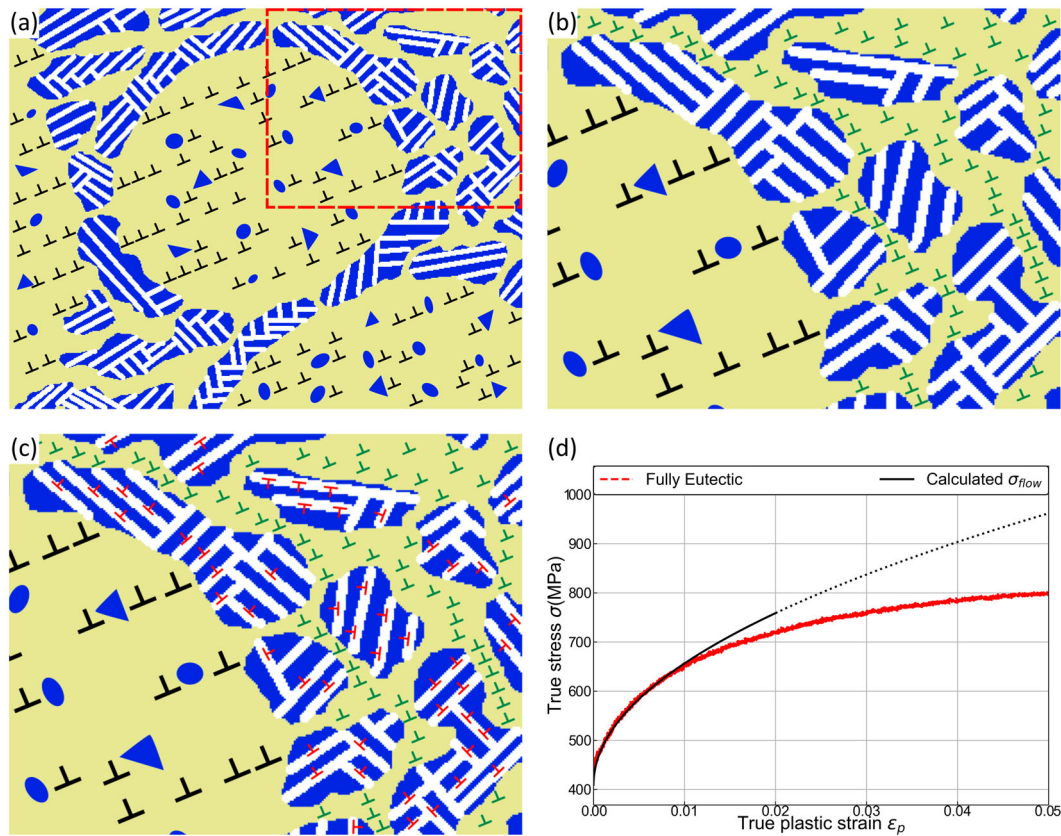


Figure 5. Schematic of the spread of dislocation plasticity in heterogeneous Al–Si with increasing strain from (a) to (c). (a) Softer Al dendrites yield first, forming pile-ups that lead to accumulation of SSDs as well as GNDs near the interfaces. (b) Enlarged image of area highlighted in (a) showing single glide dislocations (indicated by green) form in the Al matrix of nanoscale Al–Si eutectic. (c) Dislocation accommodation in nanotwinned Si nanofibers (indicated by red). The larger dislocation legend represents higher dislocation density. The dislocation evolution in the LRS nanoscale fully eutectic structure can be inferred from the matrix regions in (b,c). Yellow regions correspond to Al phase and blue to Si with the white lines representing nanotwins in eutectic Si fibers. (d) Calculated σ_{flow} from Equation (1) for fully eutectic nanoscale morphology, with deviation exceeding 5% shown as dotted line.

shown in Figure 5(d) for fully eutectic nanoscale Al–Si. Note that without pile-ups, dislocations must be accommodated by higher density of closely spaced slip planes as in Figure 5(b). With the development of plastic deformation in the Al matrix, load transfer commences in nanoscale Al–Si eutectic, consequently, high stress develops in Si fibers. Finally, this high stress facilitates slip transmission across the Al–Si interface into the rigid Si fibers as in Figure 5(c). The calculation from Equation (1) fits the experimental stress–strain at low *plastic* strains where strain hardening is highest in Al due to GNDs as rigid Si fibers deform elastically. Equation (1) overpredicts the flow strength with increasing plastic strain where Si fibers may plastically deform, thereby reducing the strain hardening.

Our results show that the fully eutectic nanoscale Al–Si (Figure 1(d)) has both higher strength and higher plasticity than the LRS heterogeneous (bimodal) dendritic/ eutectic microstructures (Figure 1(b,c)). A hierarchy of scales also exists in fully eutectic nanoscale

Al–Si: one corresponding to the nanotwin thickness in Si, and another to interfiber spacing. Appropriate constraints associate with this hierarchy approach may enhance yield stress and θ in Al matrix. Finally, the interfacial crystallography may enable slip transmission into Si from Al matrix when local stress glide exceeds loop mobility in Si, analogous to slip activity in hard TiN in Al–TiN nanolayers [13]. For the heterogeneous microstructure, the Al dendrites are presumably too coarse for the strengthening mechanisms described above. Fundamental understanding could be developed based on atomistic and meso-scale crystal elastic/plastic modeling and *in situ* straining TEM experiments, which will be addressed in future work.

5. Summary and conclusions

As-cast hypereutectic Al–20wt.% Si alloys were further processed via LRS, which results in different nanoscale morphologies with Al–16wt.% Si nominal composition.

Micropillar compression testing revealed fracture in the as-cast alloys at plastic strains of $\approx 8\%$ and flow strength of ≈ 200 MPa. However, the LRS nanoscale fully eutectic morphology with interconnected and nanotwinned Si fibers of ≈ 40 nm diameter exhibited uniform plastic deformation to strains $> 20\%$ with flow strength > 800 MPa. Likewise, LRS heterogeneous fully eutectic + primary Al dendrite morphology (which sometimes contain nanoscale Si precipitates) exhibit similar high uniform plastic deformation although at lower flow strength of ≈ 700 MPa. Post-mortem analysis revealed dislocation activity in the nanoscale Si fibers, which suggests additional dislocation accommodation mechanism involving slip transmission across nanoscale Al-Si phases, promoting uniform plastic co-deformation in LRS microstructures.

In conclusion, this study shows plastic co-deformability in nanoscale soft Al and hard Si, which results in confined layer slip and the high strain hardening effect deforming via arrays of single dislocation loops confined by interfaces of a relatively hard phase, and eventual slip transmission to the hard phase.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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Data availability

The raw/processed data required to reproduce these findings will be made available upon request.

ORCID

Huai-Hsun Lien  <http://orcid.org/0000-0003-1164-4533>

Jyoti Mazumder  <http://orcid.org/0000-0002-5295-8814>

Jian Wang  <http://orcid.org/0000-0001-5130-300X>

Amit Misra  <http://orcid.org/0000-0003-3634-6823>

References

- [1] Hirsch J. Recent development in aluminium for automotive applications. *Trans Nonferrous Met Soc China*. 2014;24:1995–2002.
- [2] García-Infanta JM, Zhilyaev AP, Cepeda-Jiménez CM, et al. Effect of the deformation path on the ductility of a hypoeutectic Al-Si casting alloy subjected to equal-channel angular pressing by routes A, BA, BC and C. *Scr Mater*. 2008;58:138–141.
- [3] Kucukomeroglu T. Effect of equal-channel angular extrusion on mechanical and wear properties of eutectic Al-12Si alloy. *Mater Des*. 2010;31(2):782–789.
- [4] Hanna MD, Lu SZ, Hellawell A. Modification in the aluminum silicon system. *Metall Trans A*. 1984;15:459–469.
- [5] Knuutinen A, Nogita K, McDonald SD, et al. Modification of Al-Si alloys with Ba, Ca, Y and Yb. *J. Light Met*. 2001;1(4):229–240.
- [6] Bendijk A, Delhez R, Katgerman L, et al. Characterization of Al-Si-alloys rapidly quenched from the melt. *J Mater Sci*. 1980;15:2803–2810.
- [7] Ferrarini CF, Bolfarini C, Kiminami CS, et al. Microstructure and mechanical properties of spray deposited hypoeutectic Al-Si alloy. *Mater Sci Eng A*. 2004;375–377:577–580.
- [8] Suryawanshi J, Prashanth KG, Scudino S, et al. Simultaneous enhancements of strength and toughness in an Al-12Si alloy synthesized using selective laser melting. *Acta Mater*. 2016;115:285–294.
- [9] Li XP, Wang XJ, Saunders M, et al. A selective laser melting and solution heat treatment refined Al-12Si alloy with a controllable ultrafine eutectic microstructure and 25% tensile ductility. *Acta Mater*. 2015;95:74–82.
- [10] Surreddi KB, Eckert J, Chaubey AK, et al. Microstructure and mechanical properties of Al-12Si produced by selective laser melting: effect of heat treatment. *Mater Sci Eng. A*. 2013;590:153–160.
- [11] Lien H, Mazumder J, Wang J, et al. Materials characterization microstructure evolution and high density of nanotwinned ultrafine Si in hypereutectic Al-Si alloy by laser surface remelting. *Mater. Charact*. 2020;161:110147.
- [12] Jia Y, Eckert J, Ma P, et al. Influence of Annealing on mechanical properties of Al-20Si processed by selective laser melting. *Metals (Basel)*. 2014;4:28–36.
- [13] Wang J, Misra A. Strain hardening in nanolayered thin films. *Curr Opin Solid State Mater Sci*. 2014;18:19–28.
- [14] Wang SJ, Liu G, Xie DY, et al. Plasticity of laser-processed nanoscale Al-Al₂Cu eutectic alloy. *Acta Mater*. 2018;156:52–63.
- [15] Kim JT, Lee SW, Hong SH, et al. Understanding the relationship between microstructure and mechanical properties of Al-Cu-Si ultrafine eutectic composites. *Mater Des*. 2016;92:1038–1045.
- [16] Park JM, Mattern N, Kühn U, et al. High-strength bulk Al-based bimodal ultrafine eutectic composite with enhanced plasticity. *J Mater Res*. 2009;24:2605–2609.
- [17] Dang B, Zhang X, Chen YZ, et al. Breaking through the strength-ductility trade-off dilemma in an Al-Si-based casting alloy. *Sci Rep*. 2016;6:1–8.
- [18] Zhang H, Schuster BE, Wei Q, et al. The design of accurate micro-compression experiments. *Scr Mater*. 2006;54:181–186.
- [19] Pierantoni M, Gremaud M, Magnin P, et al. The coupled zone of rapidly solidified Al-Si alloys in laser treatment. *Acta Metall Mater*. 1992;40:1637–1644.
- [20] Xue S, Li Q, Xie DY, et al. High strength, deformable nanotwinned Al-Co alloys. *Mater Res Lett*. 2019;7:33–39.

- [21] Li N, Wang H, Misra A, et al. In situ nanoindentation study of plastic Co-deformation in Al-TiN Nanocomposites. *Sci Rep.* **2015**;4:1–6.
- [22] Liu G, Wang S, Misra A, et al. Interface-mediated plasticity of nanoscale Al–Al₂Cu eutectics. *Acta Mater.* **2020**;186:443–453.
- [23] Ashby MF. The deformation of plastically non-homogeneous materials. *Philos. Mag.* **1970**;21:399–424.
- [24] Nieh TG, Wadsworth J. Hall-petch relation in nanocrystalline solids. *Scr. Metall. Mater.* **1991**;25:955–958.
- [25] Embury JD, Hirth JP. On dislocation storage and the mechanical response of fine scale microstructures. *Acta Metall. Mater.* **1994**;42:2051–2056.
- [26] Misra A, Hirth HK JP. Single-dislocation-based strengthening mechanisms in nanoscale metallic multilayers. *Philos. Mag.* **2002**;82:2935–2951.
- [27] Misra A, Hirth JP, Hoagland RG. Length-scale-dependent deformation mechanisms in incoherent metallic multilayered composites. *Acta Mater.* **2005**;53:4817–4824.
- [28] Ashby MF. A first report on deformation-mechanism maps. *Acta Metall.* **1972**;20:887–897.
- [29] Oliver WC, Pharr GM. An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. *J Mater Res.* **1992**;7:1564–1583.