Supplementary materials

Microstructure versus size: nano/microscale deformation of solute-strengthening Al alloys via pillar compression tests

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The ER5356 filling material is Al-Mg alloys while ER4043 is Al-Si alloys. The difference in the obtained fusion zones from these two filling materials has been discussed in Ref. [1]. For convenience of reading, here the main differences are listed:

Microstructure. Microstructure in both joints features with coarsen phases, as seen in Fig. S1 in Supplementary materials. According to EBSD testing, the FZ with ER4043 as filling materials has relatively smaller grain size (~57±51 μm) and higher dislocation density. The EDS results show that the solid solution elements for FZ with ER4043 filling material are Si and Mg, whereas only Mg can be found as solute element in FZ with ER5356 filling material.

Mechanical properties. Results from tensile testing and hardness testing show that the welded joint with ER4043 filling material has higher mechanical properties. According to the strength model proposed in Ref. [1], the higher strength in the welded joint with ER4043 filling material is due to grain size strengthening and solid solution strengthening.

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Fig. S1. SEM images of fusion zone in ER5 joint (a) and ER4 joint (b), EDS results of the phase-free areas in fusion zone of ER5 joint (c) and ER4 joint (d).
Fig. S2. (a) (b) Selected orientations for the pillar machining in the ER4 joint, (c) selected orientations for the pillar machining in the ER5 joint.

Table S1 Summary of the crystallographic data of the selected grains.

|                | Single crystal | Euler angle | Schmid factor | Initial dislocation density/ m⁻² |
|----------------|----------------|-------------|---------------|----------------------------------|
| ER4 pillars    | [101]-orientated | 253.6, 40.7, 19.9 | 0.45          | 1.32×10¹³                      |
|                | [111]-orientated | 273.3, 343.3, 39.4 | 0.3           |                                 |
|                | [312]-orientated | 268.5, 32.9, 73.1 | 0.45          |                                 |
|                | [532]-orientated | 124.4, 24.8, 23.6 | 0.46          |                                 |
| ER5 pillars    | [101]-orientated | 268.1, 44.1, 89.9 | 0.44          | 9.19×10¹²                       |
|                | [111]-orientated | 123.2, 44.2, 2.7  | 0.32          |                                 |

Fig. S3 shows the TEM results for the [111]-orientated 800nm-diameter ER4 pillar. The slip offset could be seen in Fig. S3a, and the dislocation density near the free surface seems to not change much compared to the inner area (Fig. S3b). The enlarged view (Fig. S3c) for the place indicated in Fig. S3b shows dislocations entangling with each other. For the place near the top of the pillar (Fig. S3d), high dislocation density can be seen in the form of dislocation circles and entanglement.

TEM results for the [101]-orientated ER4 pillars are illustrated in Fig. S4. Independence of the pillar’s diameter, the dislocation density near the slip offset is lower than the inner place, as suggested by Fig. S4a and c. This is possible due to the more slip events during the compression and the dislocation escaped from the free surface. For the 2 µm-diameter sample (Fig. S4d), the density and structure of the dislocation is higher and more complex than that of the 800 nm-
diameter sample (Fig. S4b). Compare to the ER4 pillars, ER5 pillars show less complex dislocation structure, as demonstrated in Fig. S5. The dislocation density for the tested pillars is calculated using the method described by Jennings et al. [2], and the results are shown in Fig. S6. At the same diameter, the dislocation density in the ER4 pillars after deformation is about two orders higher than that of the ER5 pillars. For the orientation effect, pillars with [111] orientation is higher than that of the <110>-orientated pillar. With the same microstructure, dislocation density is higher for bigger sample than that of the smaller one, as demonstrated in Fig. S6a for ER4 pillars.

Fig. S3. TEM results for the [111]-orientated 800 nm-diameter pillar taken from FZ of ER4 joint, (a) overview of the deformed pillar, (b) dislocation structure near the slip offset as
indicated in (a) with a square, (c) enlarged view of the indicated place in (b), (d) dislocation structure near the top of the pillar.

Fig. S4. TEM results for [10\bar{1}]-orientated pillars taken from FZ of ER4 joint, (a) overview for the 800 nm-diameter pillar, (b) dislocation structure near the top of the 800 nm-diameter pillar, (d) dislocation structure near the slip offset for the 2000 nm-diameter pillar, (d) enlarged view for the dislocation structure of the indicated place in (c).
**Fig. S5.** TEM results for deformed pillars taken from FZ of ER5 joint, (a) the [101]-orientated 800 nm-diameter pillar, (b) the [111]-orientated 800 nm-diameter pillar.

**Fig. S6.** (a) Dislocation density for pillars of FZ taken from ER4 joint, (b) dislocation density for pillars of FZ taken from ER5 joint.

To understand the dislocation density on the deformation behavior of the material in the FZ at nano/microscales, some bigger deformed pillars with orientation <110> were remade into smaller ones using FIB (see Fig. S6). By the remade process by FIB, the surface slip steps were removed with smooth lateral surface. The information about the original and pre-deformed pillars, such as the diameter, pre-stains, is shown in Table S2.
Fig. S6 Procedure for making pillars from deformed larger pillars, (a) deformed 5.2 μm-diameter ER5 pillar, (b) 3.2 μm-diameter pillar made from deformed 5.2 μm-diameter ER5 pillar, (c) deformed morphology of the pre-deformed 3.2 μm-diameter pillar; (d) deformed 3.2 μm-diameter ER4 pillar, (e) 2.3 μm-diameter pillar made from deformed 3.2 μm-diameter ER4 pillar, (f) deformed morphology of the pre-deformed 2.3 μm-diameter pillar

Table S2 Information of pre-deformed pillars machined from original larger pillars

| ER5 pillars | Diameter of original pillars/μm | Deformed to strain/% | Diameter of remade pillars/μm | Residual Dislocation density (m⁻²) |
|------------|--------------------------------|---------------------|-------------------------------|-----------------------------------|
| 5.2        | 7                              | 3                   | > 1.98 ± 0.8 × 10¹⁴           |
| 3.3        | 7.2                            | 2                   |                               |
| 2          | 6.8                            | 0.8                 |                               |
| ER4 pillars | 3.2                            | 9                   | ≥ 3.4 ± 0.9 × 10¹⁴           |
| 2          | 9.4                            | 0.8                 |                               |

References

[1] S. Yan, B. Xing, H. Zhou, Y. Xiao, Q.-H. Qin, H. Chen, Effect of filling materials on the microstructure and properties of hybrid laser welded Al-Mg-Si alloys joints, Materials Characterization 144 (2018) 205-218.
[2] A.T. Jennings, M.J. Burek, J.R. Greer, Microstructure versus size: mechanical properties of electroplated single crystalline Cu nanopillars, Physical review letters 104(13) (2010) 135503.