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Effects of Nb and Ta additions on the strength and coarsening resistance of precipitation-strengthened Al-Zr-Sc-Er-Si alloys

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# Abstract

A dilute Al-0.07Zr-0.02Sc-0.005Er-0.06Si (at.%) alloy was microalloyed with 0.08 at.% Nb or Ta. [Atom-probe](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/materials-science/atom-probe) [tomography](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/tomography) reveals that, upon aging, Nb and Ta partition to the coherent L12-Al3(Zr,Sc,Er) nanoprecipitates (with average concentrations of 0.2 and 0.08 at.%, respectively), with both segregating at the matrix/nanoprecipitate heterophase interface. This is consistent with the Nb- and Ta-modified alloys exhibiting, as compared to the unmodified alloy: (i) higher peak [microhardness](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/microhardness), from a higher nanoprecipitate volume fraction and/or [lattice parameter](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/lattice-parameters) mismatch; and (ii) improved aging resistance, from slower nanoprecipitate [coarsening](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/materials-science/coarsening) due to the small [diffusivities](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/diffusivity) of [niobium](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/niobium) and [tantalum](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/tantalum) in aluminum. Analogous results were previously reported for a V-modified alloy.

# Keywords

Aluminum alloys, Precipitation strengthening, Coarsening, Microhardness, Atom-probe tomography

# 1. Introduction

Within the last few decades, much research has focused on aluminum micro-alloyed with [scandium](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/scandium), which can be aged to create coherent, L12-ordered tri-aluminide nanoprecipitates (Al3Sc), which are coarsening-resistant up to 300 °C and which interact efficiently with matrix dislocations, thereby increasing the ambient strength and creep resistance of the alloys [[[1]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0005), [[2]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0010), [[3]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0015), [[4]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0020), [[5]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0025), [[6]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0030), [[7]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0035), [[8]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0040), [[9]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0045), [[10]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0050), [[11]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0055)].

Recent research has focused on identifying additional [alloying](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/alloying) elements, which can partially (or fully) replace the rather expensive scandium, while maintaining the L12 structure of the nanoprecipitates and also: (i) increasing creep resistance, by increasing the [lattice parameter](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/lattice-parameters) mismatch between the nanoprecipitates and matrix (e.g. [lanthanides](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/rare-earth-elements) [[[12]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0060), [[13]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0065), [[14]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0070), [[15]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0075), [[16]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0080), [[17]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0085), [[18]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0090), [[19]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0095), [[20]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0100), [[21]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0105)]) and thus increase the resistance by nanoprecipitates to the movement of the matrix's dislocations; and/or (ii) improving the precipitate [coarsening](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/materials-science/coarsening) resistance through their small [diffusivities](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/diffusivity) in the matrix (e.g., [transition metals](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/materials-science/transition-metals) [[[22]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0110), [[23]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0115), [[24]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0120), [[25]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0125), [[26]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0130), [[27]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0135), [[28]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0140), [[29]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0145), [[30]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0150), [[31]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0155), [[32]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0160), [[33]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0165)]), so these alloys can be used well above 300 °C. Ideally, an alloying addition would satisfy both these conditions. However, an element increasing the precipitate lattice parameter mismatch, while improving creep resistance of the alloy, may also increase the driving force for coarsening. Therefore, a compromise between creep and coarsening resistance is often necessary, and two (or more) elements can be used, one increasing lattice parameter mismatch and the other displaying small diffusivity in the aluminum matrix. To date the elements [erbium](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/erbium) and [zirconium](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/zirconium) have produced the best results as additions to Al-Sc alloys, because they very effectively: (i) increase the number density of nanoprecipitates and increase the lattice parameter mismatch and thereby the creep resistance (for Er); and (ii) significantly reduce the nanoprecipitate coarsening kinetics (for Zr), thus allowing the use of these alloys at higher temperatures [[17](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0085),[34](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0170)]. Furthermore, additions of silicon, which act as inoculants, result in Al-Sc-Zr-Er-Si alloys that have high [microhardness](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/microhardness) values upon peak-aging and are coarsening- and creep-resistant at temperatures up to 400 °C [[35](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0175)].

To further increase coarsening resistance, Zr and Sc can be partially replaced by transition metals with even smaller diffusivities in Al [[24](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0120)]. Earlier research [[23](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0115)] showed that Group 5 transition elements (G5 = V, Nb or Ta), all of which have much smaller diffusivities in aluminum than Zr and Sc, partition to the L12 Al3(Sc,G5) nanoprecipitates in small concentrations (<3 at.%). In our previous work [[36](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0180)], we studied the effects of [vanadium](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/vanadium) on coarsening- and creep-resistance of a castable Al-Er-Sc-Zr-Si alloy labeled Q2 ([Table 1](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "t0005)); V additions increased the coarsening resistance of the alloy, while the creep resistance remained unaffected. In a recent study [[37](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0185)], the three Group 5 (G5) transition metals (V, Nb and Ta) were added individually into arc-melted Al-Er-Sc-Zr alloys; these alloys were Sc-rich and Zr-poor, unlike the alloys Q2, Q4 and Q5 (with V, Nb and Ta, [Table 1](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#t0005)) studied herein, which are Zr-rich and Sc-poor. That study [[37](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0185)] revealed that vanadium decreased the coarsening kinetics during isochronal aging, whereas [niobium](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/niobium) and [tantalum](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/tantalum) had no noticeable effects. [Atom-probe](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/materials-science/atom-probe) [tomography](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/tomography) (APT) [[38](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0190),[39](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0195)] results demonstrated that only V partitioned significantly to the precipitates (~2.75 at.%).

Table 1. Average concentrations (at.%) of all [atom-probe](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/materials-science/atom-probe) tomographic (APT) nanotips analyzed for this study, compared to concentrations measured by direct current plasma-atomic emission [spectrometry](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/spectroscopy) (DCP-AES) on macroscopic volumes. Data for alloys Q1 and Q2 are from Ref. [[36](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0180)] (Nom = Nominal, ND = not detected).

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
|  | **Er** | **Sc** | **Zr** | **V** | **Nb** | **Ta** | **Si** | **L12 Formers\*** | **Technique** |
| **Q1-Nom** | 0.005 | 0.02 | 0.07 | – | – | – | 0.06 | 0.095 |  |
| **Q1** | 0.005 | 0.019 | 0.068 | – | – | – | 0.066 | 0.092 | DCP-AES |
| **Q2-Nom** | 0.005 | 0.02 | 0.07 | 0.08 | – | – | 0.06 | 0.175 |  |
| **Q2** | 0.007 | 0.013 | 0.071 | 0.074 | – | – | 0.054 | 0.165 | DCP-AES |
| **Q4-Nom** | 0.005 | 0.02 | 0.07 | – | 0.08 | – | 0.06 | 0.175 |  |
| **Q4** | 0.006 | 0.007 | 0.071 | – | 0.047 | – | 0.054 | 0.131 | DCP-AES |
| **Q4-1** | 0.004 | 0.009 | 0.242 | – | 0.157 | – | 0.057 | 0.412 | APT |
| **Q4-2** | 0.006 | 0.014 | 0.355 | – | 0.024 | – | 0.053 | 0.399 | APT |
| **Q4-3** | ND | ND | 0.205 | – | 0.176 | – | 0.064 | 0.381 | APT |
| **Q4-4** | ND | 0.018 | 0.117 | – | 0.083 | – | 0.077 | 0.218 | APT |
| **Q4-5** | 0.015 | 0.064 | 0.337 | – | 0.154 | – | 0.119 | 0.570 | APT |
| **Q4-6** | 0.001 | 0.002 | 0.100 | – | 0.020 | – | 0.075 | 0.123 | APT |
| **Q4-7** | ND | 0.002 | 0.108 | – | 0.210 | – | 0.055 | 0.320 | APT |
| **Q4-8** | 0.004 | 0.003 | 0.146 | – | 0.055 | – | 0.075 | 0.208 | APT |
| **Q4-Ave** | 0.006 ± 0.005 | 0.016 ± 0.022 | 0.201 ± 0.102 | – | 0.110 ± 0.073 | – | 0.072 ± 0.021 | 0.329 ± 0.142 |  |
| **Q5-Nom** | 0.005 | 0.02 | 0.07 | – | – | 0.08 | 0.06 | 0.175 |  |
| **Q5** | 0.006 | 0.009 | 0.074 | – | – | 0.052 | 0.082 | 0.141 | DCP-AES |
| **Q5-1** | 0.002 | 0.026 | 0.251 | – | – | 0.063 | 0.073 | 0.339 | APT |
| **Q5-2** | ND | 0.030 | 0.171 | – | – | 0.065 | 0.077 | 0.266 | APT |
| **Q5-3** | 0.002 | 0.006 | 0.149 | – | – | 0.077 | 0.080 | 0.232 | APT |
| **Q5-4** | 0.003 | 0.035 | 0.214 | – | – | 0.031 | 0.073 | 0.283 | APT |
| **Q5-5** | 0.005 | 0.040 | 0.168 | – | – | 0.083 | 0.092 | 0.296 | APT |
| **Q5-Ave** | 0.003 ± 0.001 | 0.027 ± 0.013 | 0.191 ± 0.041 | – | – | 0.064 ± 0.020 | 0.079 ± 0.008 | 0.283 ± 0.039 |  |

\*Sum of Er, Sc, Zr and Group 5 elements.

In the present study, we arc-melted two alloys with the target composition Al-0.005Er-0.02Sc-0.07Zr-0.06Si at.%, with additions of 0.08 at.% Nb (labeled Q4) or 0.08 at.% Ta (labeled Q5), matching those of Q1 (unmodified) and Q2 (with 0.08 at.% V additions) in our previous study [[36](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0180)]: all compositions are given in [Table 1](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#t0005). Due to its very high cost, the amount of Sc is minimized. A recently published study [[36](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0180)] shows that higher amounts of Sc (by a factor of 2.5 as compared to the present alloy) did not result in significant improvements in creep resistance to justify the cost. The alloys were homogenized and subjected to a double isothermal aging treatment, while measuring micro-hardness to assess peak strengthening and coarsening resistance; overaged samples were analyzed utilizing APT to assess the extent of Nb and Ta partitioning to the nanoprecipitates.

# 2. Experimental Procedure

Two 10 g buttons (Q4 with Nb additions and Q5 with Ta additions) were arc-melted (flipped and re-melted five times) in a water-cooled copper hearth furnace, using 99.99 at.% pure Al, 99.9 at.% pure Nb, 99.9 at.% pure Ta, and master alloys consisting of Al–5.9 wt% Er, Al–2 wt% Sc, Al–8 wt% Zr, and Al–12.6 wt% Si. Each button was sectioned into 2 mm thick specimens using an [abrasive](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/abrasives) blade. One 2 mm thick slice per alloy was chemically analyzed using direct current plasma-atomic emission [spectrometry](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/spectroscopy) (DCP-AES). The remaining sections were subjected to a [homogenization](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/homogenizing) treatment at 640 °C for 4 h (the same conditions as alloys Q1 and Q2 [[36](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0180)]) and a subsequent double-aging treatment at 350 °C/16 h and (400 or 450 °C)/12–720 h. The [microhardness](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/microhardness) of all mounted and polished specimens was measured as the average of at least ten [indentations](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/indentation), using a Duramin 5 microhardness tester (Struers) employing a 200 g load and a 5 s indentation time. Specimens double-aged at 350 °C/16 h + 450 °C/200 h were analyzed using a LEAP4000X Si tomograph (Cameca, Madison, WI) at a [pulse repetition rate](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/pulse-repetition-rate) of 250–500 kHz, an ultraviolet (UV) (wavelength = 355 nm) pulse energy of 50 pJ, and a sample temperature of −243 °C (30 K). The three-dimensional (3-D) tomographic data were subsequently analyzed utilizing Cameca's integrated visualization and analysis software (IVAS), version 3.6.8. APT specimens were prepared by cutting blanks with a diamond saw to ~0.5 × 0.5 × 10 mm3 and then subsequently [electropolishing](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/electropolishing) in two stages: (i) coarse electropolishing at 20–25 Vdc using a solution of 10 vol% perchloric acid in acetic acid to form a neck; and (ii) fine polishing at 15–18 Vdc using a solution of 2 vol% perchloric acid in butoxyethanol to dissolve the neck and obtain a nanotip [[40](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0200),[41](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0205)].

# 3. Results and Discussion

Scanning [electron microscope](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/electron-microscopes) (SEM) micrographs of polished cross-sections of the as-cast alloys displayed a very small number of Zr-rich particles (one example from alloy Q4 is shown in [Fig. 1](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "f0005)), about 5 μm in size, formed during [solidification](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/solidification), which are most likely primary Al3Zr precipitates. In similar alloy systems, Er- and/or Zr-rich precipitates tend to form during solidification [[34](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0170),[35](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0175)], and Er-rich precipitates dissolve during [homogenization](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/homogenizing), while Zr-rich precipitates do not. This decreases the amount of Zr in [solid-solution](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/solid-solutions), which is available for subsequent precipitation during aging (thus decreasing the peak microhardness), and/or refines the grain structure, thereby decreasing the creep resistance. In our case it is, however, safe to assume that most of the [alloying](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/alloying) elements remain in solid-solution and are available to precipitate during aging, since the number density of primary precipitates is small. Our alloys also exhibit a dendritic [microstructure](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/microstructure), as anticipated based on prior studies [[42](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0210),[43](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0215)]. All alloys are subjected to a homogenizing treatment at 640 °C for 4 h. This treatment dissolves primary Al3Er and Al3Sc (if present) precipitates. It doesn't, however, remove the interdendritic microstructure, particularly for the slow diffusers, which are Zr, Nb, and Ta. DCP-AES results display some discrepancies between the actual composition and the target compositions of both alloys. The Q4 and Q5 alloys have verified compositions listed in [Table 1](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#t0005). The Sc, Nb and Ta concentrations in alloys Q4 and Q5 are depleted by 35 to 50%, whereas the Er, Zr and Si concentrations are close to the target values.

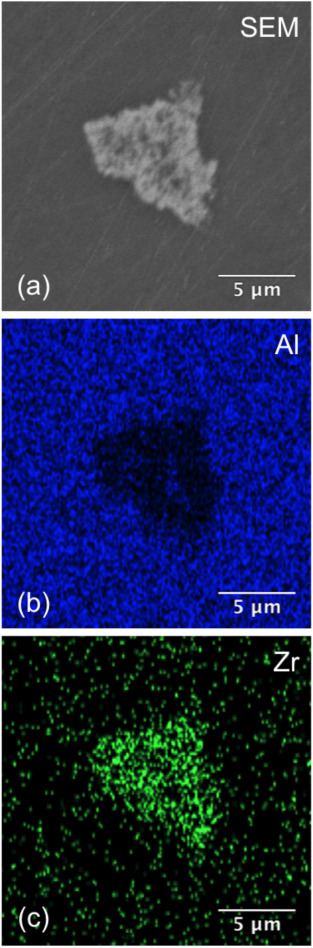


Fig. 1. (a) [Scanning electron microscopy](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/scanning-electron-microscopy) (SEM) image showing a primary Al3Zr precipitate in the as-cast alloy Q4. Energy dispersive [X-ray spectroscopy](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/x-ray-spectroscopy) (EDS) maps displaying the distributions of (b) Al and (c) Zr.

The goal of the double-aging treatment is to maximize the number density of nanoprecipitates at the lower aging temperature (350 °C) and then permit the slow-diffusing elements, Zr, Nb or Ta, to precipitate at a higher temperature (400 or 450 °C) [[36](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0180)]. Employing an isothermal aging treatment at a single high-temperature (400 or 450 °C) is not ideal as nanoprecipitates consisting of the faster diffusing elements Er and Sc, would nucleate, grow, and coarsen rapidly before the slow-diffusing elements, particularly Zr, can form a shell around these nanoprecipitates to decelerate their [coarsening](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/materials-science/coarsening) kinetics. This would result in a small number density of nanoprecipitates and lower [microhardness](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/microhardness) values and creep resistance. Alternatively, a single heat treatment at a lower temperature, while ensuring the formation of Er- and Sc-rich nanoprecipitates at a high number density, would not permit the slow-diffusing elements to precipitate from the solid-solution, at least for times relevant to industrial heat-treatments (a few days) or basic research (a few months).

[Fig. 2](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "f0010) displays the evolution of the microhardness in alloys Q4 and Q5 during the second-step of the double-aging treatment, performed at 400 or 450 °C. Also presented for comparative purposes are the microhardness values for the two alloys, Q1 (no additions) and Q2 (with V-additions), previously published [[36](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0180)]. The Q1 and Q2 alloys were solidified in graphite molds placed on ice-cooled copper platens and quenched into water after solidification [[36](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0180)]. They may therefore be less supersaturated than the present arc-melted alloys Q4 and Q5 in the as-cast state; all alloys were homogenized (640 °C/4 h) prior to aging. The difference between the dendrite sizes may have an effect on the reported microhardness values. At 400 °C ([Fig. 2](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#f0010)(a)), for 12 to 120 h of aging, alloys Q2, Q4 and Q5 display a constant microhardness of 58–60 HV (~575 MPa), derived from the initial aging treatments at 350 °C. For these aging times, all values are within error bars, but alloy Q5 displays a slightly higher average microhardness. Beyond 200 h, the microhardness of alloy Q2 decreases significantly, whereas alloys Q4 and Q5 display only a very small decrease in microhardness values up to 720 h, for the longest aging time employed. The unmodified control alloy Q1 softens rapidly upon aging at 400 or 450 °C, and it displays a microhardness value significantly lower than alloy Q5 (425 vs 540 MPa) after 720 h, which is the longest aging time at 400 °C. This result is consistent with the absence of a slow-diffusing Group 5 element in this alloy. As displayed in [Table 1](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#t0005), alloys Q4 and Q5 have smaller Sc concentrations than the V-containing alloy Q2, and smaller concentrations of L12 formers, which is the sum of Er, Sc, Zr and a Group 5 element; they, however, have a somewhat better coarsening resistance, especially at 400 °C. Increasing the Sc concentrations of alloys Q4 and Q5 to the same level as alloy Q2 alloy may further increase the strength of alloys Q4 and Q5, as Sc (unlike Zr and the Group 5 elements) precipitates strongly from a solid-solution. At 450 °C ([Fig. 2](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#f0010)(b)), all modified alloys are within a single band of data points, which decrease immediately upon aging. Based on previous results obtained from similar alloys [[[34]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0170), [[35]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0175), [[36]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0180), [[37]](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0185)], including alloy Q2 discussed in this article, it is safe to hypothesize that the continuous decrease in microhardness in all alloys is due to the coarsening of L12 ordered nano-precipitates. The unmodified Q1 alloy is mechanically weaker than the modified alloys, but the difference with the modified alloys is less than at 400 °C. At all aging times, the alloy Q5 is stronger than alloy Q1 (380 MPa vs 350 after 720 h), while alloys Q2 and Q4 are intermediate in microhardness and very close to each other. The data show that the Sc concentrations in these alloys can be decreased from 0.013 at.% (Q2) to 0.007–0.009 at.% (Q4 and Q5), a factor of almost 2, without a reduction in coarsening resistance, making these alloys significantly less expensive. Alloys Q4 and Q5 must be retested after being conventionally cast, as alloys Q1 and Q2 were, to eliminate completely the possibility that differences in aging responses may be due to differences in the dendritic cast structures or to a higher [supersaturation](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/supersaturation); the latter being unlikely, given the same homogenization conditions utilized.

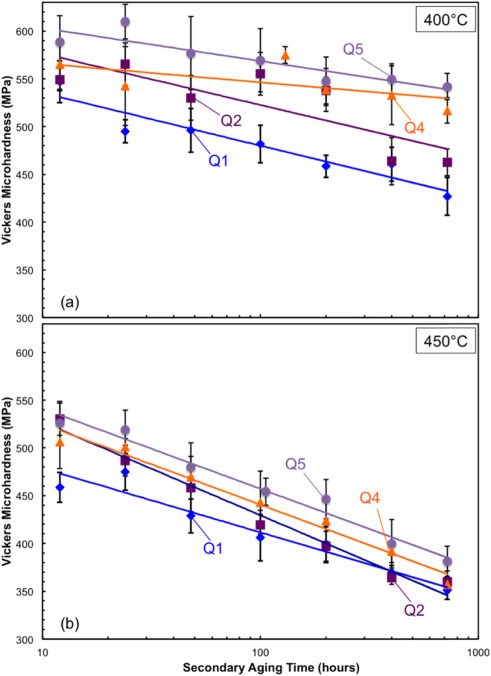


Fig. 2. Vickers [microhardness](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/microhardness) (MPa) plots versus secondary aging time (hours) displaying its evolution for alloys Q1 (unmodified), Q2 (V-modified), Q4 (Nb-modified), and Q5 (Ta-modified) during double-aging with a primary step at 350 °C for 16 h, followed by secondary steps at: (a) 400 °C; and (b) 450 °C for times between 12 and 720 h.

An APT study was performed on alloys Q4 and Q5, which were double-aged at 350 °C/16 h plus 450 °C/200 h. The long aging time, 200 h, was chosen as a compromise between excessive coarsening (observed in [Fig. 2](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#f0010) by the microhardness decrease) and insufficient diffusion of Nb and Ta. The proximity histogram [[44](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0220)] displayed in [Fig. 3](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "f0015) shows the elemental concentration distributions in the matrix and nanoprecipitates, obtained from two partial nanoprecipitates, for alloy Q4. An increased Nb concentration is detected in the outer shell of the nanoprecipitate, near the interface with the matrix, indicated by a vertical dashed line, with a peak value of ~1.2 at.% or ~8 times the matrix's Nb concentration. This suggests that Nb partitions to the tri-aluminide nanoprecipitates and is consistent with the improved coarsening resistance when compared to unmodified alloy Q1. Also visible from this proximity histogram is the uniform concentrations of Er, Sc, and Zr within the nanoprecipitates. In similar alloy systems, including alloy Q2, a core-shell nanoprecipitate microstructure is observed, where an Er- and Sc-rich core is surrounded by a Zr-rich shell. Specifically, for alloy Q4 aged for 200 h (compared to 24 h for alloy Q2) the Zr-rich shell is also enriched in Nb, whose concentration reaches locally 0.3 at.%. [Table 2](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "t0010) displays the matrix's and nanoprecipitates' average concentrations as determined by APT. It is evident that this particular nanotip is enriched in Zr and Nb, by factors of ~3.5 and ~4, respectively, as compared to the average values obtained utilizing DCP-AES. This indicates that this particular nanotip was most likely located in a dendritic core, as the peritectic-forming elements Zr and Nb are anticipated to segregate at the dendrite cores, whereas the eutectic-forming elements Er and Sc segregate in the inter-dendritic channels. The Zr concentration is higher than the average concentration measured using DCP-AES which supports the assumption that the nanotip volume sampled corresponds to the dendritic core. The compositional data also demonstrate that most of the Er, Sc, and Zr partitioned to the nanoprecipitates, as anticipated from prior studies [[36](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0180),[45](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0225)]. While there is a significant amount of Nb partitioning to the nanoprecipitates, most of the Nb remains in the matrix. The nanotip volume displayed in [Fig. 3](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#f0015) contains 0.16 at.% Nb, which is almost four times the solid-solubility limit reported for the Al-Nb system at 450 °C (0.045 at.%), which can provide solid-solution strengthening [[46](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829" \l "bb0230)].

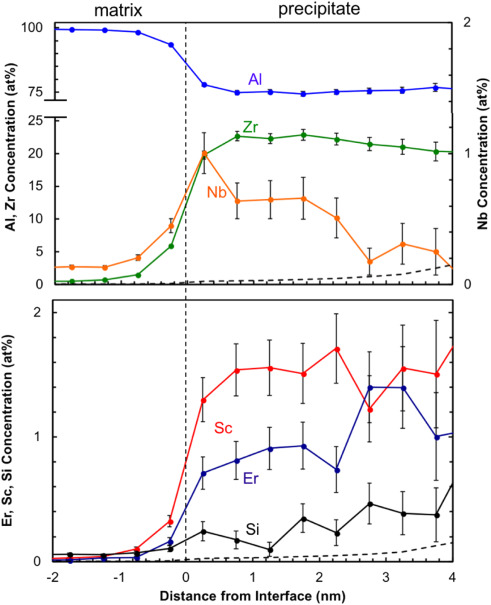


Fig. 3. Proximity histograms of two partial nanoprecipitates in alloy Q4-1 in the double-aged condition (350 °C/16 h plus 450 °C/200 h), indicating Nb partitioning to the nanoprecipitates. The vertical dashed line indicates the position of the matrix/nanoprecipitate interface. It is defined by the inflection point in the Al concentration profile. Dashed curves in both proximity histograms show the detection limit (plotted on the right ordinate), which represents a single ion detected in a bin.

Table 2. Matrix and precipitate concentrations (at.%) for alloys Q4 and Q5 nanotips analyzed utilizing [atom-probe](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/materials-science/atom-probe) [tomography](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/tomography) (APT), for nanotips listed in [Table 1](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#t0005), which contain nanoprecipitates. Data obtained from volumes with partial nanoprecipitates are less accurate than those with full nanoprecipitates (ND = not detected).

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
|  | **Volume** | **Er** | **Sc** | **Zr** | **Nb** | **Ta** | **Si** | **L12 Formers\*** | **Tip volume (nm3)** | **Number of precipitates** |
| **Q4-1** | Matrix Precipitate | ND 0.96 | 0.002 1.54 | 0.131 24.84 | 0.157 0.26 | – – | 0.118 0.17 | 0.290 27.60 | 3 × 105 | 2 partial |
| **Q4-2** | Matrix Precipitate | 0.005 0.16 | 0.005 1.43 | 0.225 21.14 | 0.024 0.01 | – – | 0.053 0.06 | 0.259 22.74 | 1 × 105 | 1 partial |
| **Q4-6** | Matrix Precipitate | ND ND | 0.002 1.12 | 0.093 23.58 | 0.019 0.3 | – – | 0.075 0.14 | 0.134 25.0 | 2 × 106 | 2 partial |
| **Q4-7** | Matrix Precipitate | ND 0.29 | 0.008 8.65 | 0.104 16.24 | 0.210 0.24 | – – | 0.139 0.12 | 0.322 25.42 | 4 × 105 | 1 partial |
| **Q5-1** | Matrix Precipitate | 0.002 0.76 | 0.016 4.84 | 0.216 18.21 | – – | 0.063 0.10 | 0.073 0.12 | 0.297 23.33 | 2 × 105 | 1 full |
| **Q5-4** | Matrix Precipitate | 0.002 0.49 | 0.024 6.19 | 0.184 16.85 | – – | 0.031 ND | 0.073 0.05 | 0.238 23.66 | 3 × 105 | 1 partial |
| **Q5-5** | Matrix Precipitate | 0.001 1.15 | 0.009 9.97 | 0.130 12.54 | – – | 0.083 0.13 | 0.091 0.36 | 0.223 23.79 | 8 × 105 | 2 partial |

[Fig. 4](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#f0020) displays a proximity histogram obtained from alloy Q5, containing one full-nanoprecipitate, which exhibits an increase in Ta concentration as compared to the matrix composition (by a factor 1.6, [Table 2](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#t0010)), without localized interfacial segregation. This result indicates that Ta, like Nb in alloy Q4 ([Fig. 3](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#f0015)) and V in alloy Q2 [[36](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0180)], partitions to the tri-aluminide nanoprecipitates, consistent with enhanced coarsening resistance, [Fig. 2](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#f0010). Unlike Nb and V, Ta ([Fig. 4](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#f0020)) is not segregated at the heterophase interface, but rather is present throughout the nanoprecipitates. Further comparisons between the APT results for these three alloys are difficult, as the analyzed volumes are depleted and enriched with alloying elements differently, due to dendritic segregation. The absolute concentrations of Group 5 elements in the nanoprecipitates may vary from nanotip-to-nanotip. Thus, comparing the matrix/nanoprecipitate partitioning coefficients, ratio of the concentration in a nanoprecipitate to that of the matrix, for the respective Group 5 elements is more informative. For alloys Q2 [[36](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#bb0180)], Q4 ([Fig. 3](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#f0015)) and Q5 ([Fig. 4](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#f0020)), the partitioning coefficients are 2 for V, 1.6 for Nb and 1.6 for Ta, respectively. This indicates that the three Group 5 elements (V, Nb, and Ta) have approximately similar partitioning behaviors; their effects on the matrix/nanoprecipitate [lattice parameter](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/lattice-parameters) mismatch, affecting creep resistance, may be different as are their [diffusivities](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/diffusivity) in the aluminum matrix, which affects the coarsening resistance of the nanoprecipitates. The results presented herein reveal that Ta additions result in stronger and more coarsening resistant alloys; additional research, especially based on TEM imaging, is required, however, to definitely establish this point.

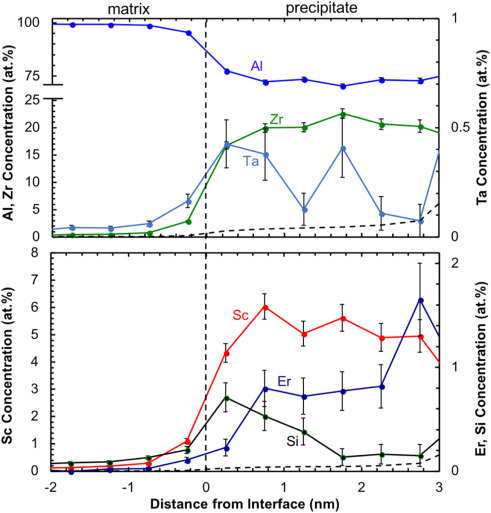


Fig. 4. Proximity histograms of one full nanoprecipitate in alloy Q5-1 in the double-aged condition (350 °C/16 h plus 450 °C/200 h), showing Ta partitioning to the nanoprecipitate. The vertical dashed-line indicates the position of the matrix/nanoprecipitate interface. It is defined by the inflection point in the Al concentration profile. Dashed curves in both proximity histograms show the detection limit (plotted on the right ordinate), which represents a single ion detected in a bin.

Due to the small number density of nanoprecipitates, there were at most two nanoprecipitates present in each of the nanotips analyzed. Although more nanotips were analyzed (listed in [Table 1](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#t0005), [Table 2](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#t0010)), only one full nanoprecipitate was detected in the small volumes (listed in [Table 2](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#t0010)), which is consistent with a significant amount of coarsening at 450 °C. Thus, statistically-relevant values for the mean nanoprecipitate radius, volume fraction and number density are not reported. Based on the nanotips analyzed, we expect the mean nanoprecipitate radius to be ~8 nm and the number density to be approximately 1021 m−3, which is close to the values obtained for alloy Q2 (6 nm and 6.8 × 10−21 m−3, respectively). [Table 1](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#t0005) also displays the compositional variations among nanotips, which is a direct effect of the dendritic microstructure and elemental macro-segregation. The Er and Sc concentrations are anticipated to be under-determined, due to the lack of full nanoprecipitates in the volumes analyzed because these elements tend to partition to the nanoprecipitates's cores. The analyzed volumes must include multiple full nanoprecipitates for accurate results. Also, it is noted that, due to dendritic macro-segregation, the composition of one nanotip to another can vary significantly (as indicated by the relatively large error values given in [Table 1](https://0-www-sciencedirect-com.libus.csd.mu.edu/science/article/pii/S1044580317335829#t0005)). Hence, it is difficult to draw further conclusions without analyzing a larger number of nanotips, which is expensive.

# 4. Conclusions

The effects of Nb and Ta on the microstructural and [microhardness](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/microhardness) evolution of arc-melted Al-Er-Sc-Zr-Si alloys are investigated. The following conclusions are reached as a result of this study:

1. As-cast alloys contain a small number density of Zr-rich primary precipitates, which were previously observed in similar alloys, but they do not have any significant negative effects on the alloys.

2. Double-aging studies are performed at a primary aging treatment at 350 °C for 16 h, which is followed by a secondary aging treatment at 400 or 450 °C for times up to 720 h. Both the Nb- and Ta-containing alloys (Q4 and Q5) displayed improved [coarsening](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/materials-science/coarsening) resistance at 400 °C compared to the base alloy Q1 and V-modified alloy Q2. This effect is not as significant at 450 °C. Alloys Q4 and Q5 are, however, still considerably stronger than alloys Q1 and Q2.

3. [Atom-probe](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/materials-science/atom-probe) [tomography](https://0-www-sciencedirect-com.libus.csd.mu.edu/topics/physics-and-astronomy/tomography) results reveal increased concentrations of Nb and Ta at the matrix/nanoprecipitate heterophase interface in over-aged (350 °C/16 h plus 450 °C/200 h) alloys Q4 and Q5, respectively. This is similar to our prior results obtained from alloy Q2, where an increased V concentration at the matrix/nanoprecipitate heterophase interface was observed.

4. Due to the small number of nanoprecipitates observed in the analyzed atom-probe tomographic volumes, no definitive conclusions could be reached regarding the number density and volume fraction of said nanoprecipitates.

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