High performance aluminum–cerium alloys for high-temperature applications

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High performance aluminum–cerium alloys for high-temperature applications

Abstract
Light-weight high-temperature alloys are important to the transportation industry where weight, cost, and operating temperature are major factors in the design of energy efficient vehicles. Aluminum alloys fill this gap economically but lack high-temperature mechanical performance. Alloying aluminum with cerium creates a highly castable alloy, compatible with traditional aluminum alloy additions, that exhibits dramatically improved high-temperature performance. These compositions display a room temperature ultimate tensile strength of 400 MPa and yield strength of 320 MPa, with 80% mechanical property retention at 240 °C. A mechanism is identified that addresses the mechanical property stability of the Al-alloys to at least 300 °C and their microstructural stability to above 500 °C which may enable applications without the need for heat treatment. Finally, neutron diffraction under load provides insight into the unusual mechanisms driving the mechanical strength.

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High Performance Aluminum-Cerium Alloys for High-Temperature Applications

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Abstract:

Light-weight high-temperature alloys are important to the transportation industry where weight, cost and operating temperature are major factors in design of energy efficient vehicles. Aluminum alloys fill this gap economically but lack high-temperature mechanical performance. Traditional alloy strengthening mechanisms, such as age hardening, are enabled by precipitates which become thermodynamically unstable at elevated temperatures. Alloying aluminum with cerium creates a highly castable alloy, compatible with traditional aluminum alloy additions, that exhibits dramatically improved high-temperature performance. These compositions display room temperature ultimate tensile strength of 400 MPa and yield strength of 320 MPa, with 80% mechanical property retention at 240°C. A mechanism is identified that addresses the mechanical property stability of the Al-alloys to at least 300°C and their microstructural stability to above 500°C. This corresponds to a homologous temperature (T/T_Melt) greater than 0.84, rivaling the stability observed in heat tolerant materials such as superalloys. Finally, neutron diffraction under load provides insight into the unusual mechanisms driving the mechanical strength.

Aluminum alloys are desirable as structural materials due to their outstanding castability, excellent mechanical properties, and low cost. They occupy the gap between inexpensive but dense iron alloys and costly high-performance titanium alloys. Within the transportation sector, the high strength to weight ratio, corrosion resistance, and high thermal conductivity of aluminum alloys have long been important for the automotive and aerospace industries and demand continues to expand due to the promise of improved performance and fuel economy. This paper describes a new family of economically competitive aluminum alloys containing 6-16 weight percent (wt. %) cerium which exhibits dramatically improved high-temperature mechanical properties, in addition to improved castability and thermal stability when compared to existing aluminum alloys. Furthermore, they may not require post-casting heat treatment, which adds significantly to the manufacturing cost in terms of energy, time, and infrastructure requirements.

Castable engineering alloys are typically strengthened through precipitation of intermetallic phases from alloying elements dissolved during the casting process or driven into solution by heat-treating[1]. These strengthening precipitates improve alloy performance by increasing stiffness and strength, while lowering thermal expansion[2, 3]; however they reside in kinetically frozen high energy architectures along chemical potential gradients that lead to
instabilities at elevated temperatures[4]. The high mobility of traditional alloying elements leads to coarsening through processes such as Ostwald ripening; thus, prolonged exposure to high temperatures leads to dramatic changes in the microstructure and a corresponding degradation of mechanical properties. The loss of mechanical performance bounds the maximum operating temperature near the alloy aging temperature during the final step of heat treatment (155-190 °C for most Al alloys). This limitation becomes particularly significant for internal combustion engines which benefit from light-weight materials compatible with higher temperatures for both the engine and nearby components.

Research developing aluminum alloys with improved high temperature performance has principally focused on systems such as Al-Sc, Al-Zr, and Al-V which form stable L1₂ precipitates[5-7]. The alloy strengthening Al₃X (X = Sc, Zr, V) precipitates are stabilized on the basis of lattice coherence with the fcc aluminum, creating interfacial strain which increases thermodynamic stability and acts as a creep-diffusion barrier[8]. This coherence breaks down above the conversion temperature, e.g. about 300 °C for Al-Sc[9], resulting in the loss of high-temperature performance.

Figure 1: a) Elongation vs. intermetallic content for Al-Ce (wt.%) alloys (blue triangles) with a line to guide the eye compared to traditional aluminum alloys (red). b) Ratio of 300°C to room temperature yield strength vs ratio of ultimate tensile strength at 300°C to room temperature, demonstrating superior thermomechanical stability for Al-Ce alloys. Ce-A206 is A206 alloy with 8 wt.% Ce. The inset shows Al-Ce based alloys at 240 °C against standard alloys at 200 °C. Al-Ce-Si-Mg alloys show increased yield at elevated temperature and Al-Ce-Mg shows no decrease relative to room temperature. c-d) Al-rich region of the Al-Ce phase diagram based on a CALPHAD assessment where the Al matrix exhibits almost no Ce solubility. e) Phase stability of major precipitate phase fraction relative to room temperature in aluminum alloys highlighting the thermal stability of Al₁₁Ce₃. Color code: 500-series (blue), 200 series (green), 300 series (red), and the new Al-10Ce (wt.%) alloy (orange).
In contrast, Al-Ce based alloys remain thermodynamically stable at high temperatures, independent of their mode of preparation (e.g. extruded, wrought, cast). For example, thermomechanical processing via extrusion of the binary alloy results in 400 MPa ultimate tensile strength (UTS) and 340 MPa yield strength, while hot isostatic pressing (HIP), equivalent to fully dense casting, leads to 280 MPa UTS and 220 MPa yield. The mechanical properties of Al-Ce alloys using an extrusion ratio of 3:1 are competitive with leading high temperature wrought alloys such as A2618 (440 MPa UTS) and A4032 (380 MPa UTS) which typically require extrusion ratios exceeding 10:1. The applications space for wrought materials is limited by the energy intensive processing associated with producing engineered metastable structures. In contrast, highly castable Al-Ce alloys form structures in thermodynamic equilibrium and remain so until near their melting point. This study focuses on alloys formed by casting rather than alternative processing methods due to the application versatility arising from their ability to adopt a greater range and complexity of structures. Characterization and correlation of the composition, microstructure, and mechanical behavior of cast Al-Ce-based alloys[10] was conducted to understand the origin and mechanisms of their highly desirable performance, with the implicit understanding that thermo-mechanical processing should further enhance the mechanical properties.

These behaviors are driven by the exceptional change in Ce solubility between the liquid and solid phases of aluminum near the eutectic point, which leads to a high stable intermetallic precipitating into a nano-scale architecture during initial solidification. The superior elongation of cast Al-Ce alloys compared to many commercial Al-alloys is illustrated in Fig. 1a. The intermetallic secondary phases are far more brittle than the surrounding aluminum matrix, and as a result, elongation to failure is driven down as intermetallic content increases[3, 11]. The retention of mechanical properties at elevated temperatures is demonstrated in Fig. 1b where the ratio of the 300°C to room temperature yield strength is compared against the ratio of the 300°C to room temperature UTS. The Ce alloys retain over 60% of their room-temperature yield and more than 40% of their UTS at 300°C. This compares favorably to the traditional aluminum alloys which at the highest retention values do no exceed 45% yield and 50% UTS retention values at 300°C. Incorporating traditional Al alloying elements such as Si and/or Mg into the Al-Ce parent alloy retains thermomechanical properties (Fig. 1b inset) at elevated temperature compared to traditional alloys.

This superior mechanical property retention in the Al-Ce alloys can be understood in terms of the behavior of their constituent phases at elevated temperatures, specifically the solubility and diffusion of Ce. Several atomic percent of common aluminum alloying additions, such as Cu, Si, and Mg can dissolve into the aluminum matrix during a heat treatment as their solubility increases with temperature (see S.I.)[11]. These are the primary additions in the A206, A356, and A535 alloys, respectively. Quenching retains the solute atoms in a supersaturated solution and enables finely dispersed precipitate formation via age hardening, which may be accelerated by soaking at 100-200°C[12]. Nonetheless, this same solubility limits the high-temperature stability of the alloy due to precipitate dissolution and the resulting changes to the microstructure. In contrast to the common Al-alloying elements, the solubility of Ce in solid aluminum is very low. Near the eutectic temperature (642°C), the upper limit of solubility is ~0.05 wt.% Ce in Al with the solubility falling below 0.02 wt.% by 600°C[13, 14] as compared...
to Cu, which has a solubility exceeding 5% by 600° C[11]. Even Sc, which leads to the most competitive high-temperature alloys has a solubility of 0.2 wt.% at 600° C, exceeding the solid solubility of Ce by an order of magnitude[14]. The Ce values are consistent with the CALPHAD thermodynamic assessment of the Al-Ce binary phase diagram (Fig. 1c-d) which predicts a similarly low solubility for Ce in the Al matrix[15], and renders the strengthening intermetallic, Al$_{11}$Ce$_3$, far more stable against dissolution in solid Al than the intermetallics of standard commercial alloys. Dissolution of strengthening phases into the aluminum matrix is illustrated in property diagrams (Fig. 1e) where the fraction of intermetallic precipitates retained decreases with increasing temperature. It is evident that the phase fraction of intermetallic, in this case Al$_{11}$Ce$_3$, retained at elevated temperatures far exceeds any of the other alloying elements. Electron microscopy data (Fig. 2) reinforce the assignment of near-zero solubility of Ce in Al and intermetallic stability of Al$_{11}$Ce$_3$ at elevated temperatures. Fig. 2c contains an Energy Dispersive X-Ray Spectroscopy (EDS) map of a 10 wt.% cerium alloy inset in the high angle annular dark-field transmission electron microscopy (HAADF TEM) image (see SI for additional images). The EDS map reveals a distinct dividing line between the Al rich phase and the Ce rich

Fig. 2: SEM micrographs of Al-12Ce (wt.%) alloy (a) as-cast, and (b) after T6 heat treatment showing mild spheroidization but no change of the larger features. (c) TEM HAADF image of Al-10Ce (wt.%) where the Al$_{11}$Ce$_3$ laths are 100-200 nm wide. The false color inset shows the Al$_{11}$Ce$_3$ regions (purple in the EDS map). (d-e) Fracture surfaces of Al-12Ce and Al-16Ce (wt.%), respectively, illustrating ductile fracture in the former and a mix of ductile and brittle fracture in the latter. Red area in (d) shows fracture along a eutectic intermetallic lath with ductile fracture surrounding. Ductile fracture can be observed in the eutectic zone surrounding the brittle primary crystals in (e). These eutectic zones lead to the elevated ductility of this alloy over alloys with similar intermetallic content. Note the large difference in scale bars between (d) and (e).
intermetallic, while detailed TEM studies show a coherent phase boundary. The lack of Ce dispersion at the phase boundary reflects the thermodynamic stability of Al11Ce3 \( (\Delta H_f = -42.7 \text{ kJ/mol at room temperature relative to Al and Ce}) \) which drives the Ce toward intermetallic formation[15].

In addition to their low solubility, the large size of the Ce atoms results in a reduced diffusion coefficient when compared to other alloying elements. As an illustration, the diffusion coefficient for Ce at 500 °C is 5.7x10^-14 cm²/s, which is about 10,000 times smaller than for Cu, 6.0x10^-10 cm²/s or Mg, 1.4x10^-9 cm²/s, at comparable temperatures[1, 16]. Strong vacancy binding to Ce atoms[17] further decreases degradation of the Al11Ce3 intermetallics as it impedes and, therefore, reduces vacancy diffusion—the dominant transport mechanism for solute atoms within the matrix[18].

Experimental evidence supporting the low solubility and diffusion of the Ce within the Al-Ce systems is provided by the scanning electron microscopy (SEM) back-scatter images of as-cast and heat-treated 12 wt.% Ce alloys respectively in Fig. 2(a-b). The as-cast alloys show a very fine interconnected eutectic microstructure (white) and the pure aluminum phase (gray). The scale of the laths, as small as 100nm, along with their uniform distribution, and interdendritic spacing all aid in improving the alloy mechanical properties[11]. Exposing the same alloy to a 20-hour soak at 520°C results in a eutectic microstructure that has undergone only minor morphological changes. Instead of thin and interconnected laths, they have rounded in many places and become less interconnected. This represents a localized minimization of the microconstituent surface energy at the eutectic through interdiffusion within the intermetallic and accompanying spheroidization, rather than bulk diffusion through the matrix. The overall scale of the intermetallic phase has not changed: laths and rods remain at widths near 100 nm, and the phase fractions are consistent across samples thereby demonstrating the conversion temperature is above 520°C. The fcc phase regions remain relatively small and well distributed throughout the sample after heat-treatment. Thus, a combination of low Ce solubility in the Al matrix and low Ce diffusion coefficients avoids coarsening mechanisms through Ostwald ripening, while the high thermodynamic stability of the Al11Ce3 intermetallic resists substantial microstructural evolution in the Al-Ce alloys and, by extension, degraded mechanical properties.

The fracture surfaces for Al-12Ce and Al-16Ce (wt.%) are shown in Fig. 2d and 2e, respectively. Significant dimpling, a characteristic of ductile fracture, dominates the fracture surface of Al-12Ce (wt.%). Primary Al11Ce3 solidification begins around 10wt.% Ce addition, just beyond the Ce-Al eutectic point (Fig. 1c). The cooling rates of the experimental castings were sufficiently fast to undercool the 12 wt.% alloy into the eutectic region, while at 16 wt.% Ce, large primary crystals of Al11Ce3 precipitate surrounded by eutectic laths. The large crystals promote brittle fracture, yet some ductility remains due to the surrounding matrix-eutectic ductile fracture mechanism. The dominant brittle fracture along the crystal faces leads to the drastic drop in ductility between the 12 wt.% and 16 wt.% Ce alloys shown in Fig 1a. Even then, ductility values remain comparable to that of many commercial aluminum alloys with equivalent intermetallic content.

The alloy mechanical properties are significantly improved with small quantities of ternary and quaternary additions. Representative castings of Al-12Ce-4Si (wt.%) and Al-12Ce-
Figure 3: a) Al-Ce-Si ternary liquidus projection based on a CALPHAD assessment (see text). b) USAXS/SAXS for Al-12Ce-4Si-0.4Mg (wt.%) illustrating heating has negligible effect on particle size (or shape: see Supplement S3-4). c-d) SEM micrograph of Al-12Ce-4Si-0.4Mg (wt.%) as-cast and after T6 heat treatment. Insets show EDS of intermetallic precipitates of same composition as white phases in SEM image (Al contribution removed for clarity) illustrating internal changes in microstructure.

4Si-0.4Mg (wt.%) illustrate these properties. Cerium reacts favorably with many traditional solutionizing elements, including Mg and Si, to form thermally stable intermetallics without microstructural coarsening in the solid state. Small additions of Si lead to the tetragonal intermetallic Ce(Si$_{1-x}$Al$_x$)$_2$, with $x = 0.1 - 0.9$, identified in Fig. 3a as the $\tau_1$ phase (I4$_1$/amd space group) which extends across the central portion of the phase diagram and exhibits a high range of temperature stability in the aluminum matrix once formed[15, 19-21], similar to that of Al$_{11}$Ce$_3$ in the binary. The low solubility of Ce in Al and Si[22] along with the tight bonding of vacancies to Ce[17] and the formation enthalpy of the $\tau_1$ phase, which reaches a minimum of -67 kJ/mol near $x$=0.5, all contribute to the stability of this phase. The solubility of Si in the Al matrix phase is 1.5 wt.% at high temperatures and it is possible to quench a supersaturated solids solution to 11 wt.%, whereas the low mobility and reactivity of Ce leads to immediate intermetallic formation. Thus, the structure of the as-cast Al-Ce-Si comprises Al$_{11}$Ce$_3$ intermetallic laths formed through an invariant reaction in an Al matrix that seed precipitation of Si from the supersaturated solution as the matrix solidifies. After a T6 heat-treatment the morphology persists, with the precipitates serving as templates for the ternary Ce(Si$_{1-x}$Al$_x$)$_2$. The associated mechanical properties improve from a yield and UTS of 83 MPa and 150 MPa, respectively to 128 MPa and 255 MPa after a T6 heat treatment. Similarly, the elongation improves from 2% to 8.5% before fracture.
The stability of these multicomponent phases at elevated temperatures was explored through a series of volume averaged ultra-small and small angle x-ray scattering (USAXS/SAXS) measurements. Analysis of the scattering data provides insight into the size, shape, number density, and size dispersion of structural inhomogeneities (i.e. intermetallics in the alloys) and, as such, these methods are ideally suited for investigating the structure and structural evolution of these alloys. A T6 heat treated Al-12Ce-4Si-0.4Mg (wt.%) specimen was heated in 100 °C increments between which the sample was cooled to room temperature, with measurements performed at both the elevated and base temperature (Fig 3b). Should any microstructural changes result, deflections would be observed in the scattering vector. That there is no deviation between individual spectra in successive measurements indicate no changes in the underlying microstructure in either size or shape to at least 400° C (see S.I. for a detailed

Figure 4: a) Neutron Spectrums showing change in scattering intensity as applied compressive strain increases. b) Strain measurements of Al-12Ce and Al-12Ce-0.4Mg (wt.%) performed under compressive load (the latter is offset by 100 MPa for visibility). Here the arrows denote onset of phases II (red), and III (black) described in the text. c) phase load-sharing for Al-12Ce under compressive load. d) phase load-sharing for Al-12Ce-0.4Mg under compressive load. Shaded region denote difference between binary and ternary alloy composition’s mechanical response.
analysis)[23, 24]. SEM images before and after heat-treatment of the Al-12Ce-4Si-0.4Mg (wt.%) alloy (Figs. 3c-d) further illustrate the high-temperature stability of the system. In the as-cast state, primary crystals of Ce-rich intermetallics are surrounded by thin laths of Al-Si and Al-Si-Mg intermetallics. After 10 hrs. at 540 °C the Mg diffuses uniformly throughout the aluminum matrix while the primary crystals transition to Ce(Si1-xAlx)2, as illustrated in the TEM false-color images (insets to Figs. 3c-d). The surrounding eutectic displays similar morphological changes to those seen in the binaries with more isolated and less interconnected intermetallic structures forming from surface energy minimization. Thermodynamic stability is important to retaining the high temperature mechanical strength, but identification of the underlying strengthening mechanisms requires further investigation.

To this end, neutron diffraction measurements enabled investigation of the mechanical behavior as a function of compressive loading. These experiments focused on the simplest alloys, whose behavior is reflective of this family of materials: the Al-12Ce (wt.%) binary and the structurally equivalent Al-12Ce-0.4Mg (wt.%) ternary. These specimens provide distinct peaks for both the Al matrix and intermetallic Al11Ce3, as shown in Fig. 4a for the binary alloy. Analysis of these diffraction peaks as a function of loading enables assignment of the lattice strain within each phase, and by extension, identification of load partitioning in the system. Emphasis is placed upon analysis of the diffraction data from the Al matrix because it has a simple cubic unit cell; since the Al-Ce alloys are two component systems, the properties of the complex anisotropic Al11Ce3 intermetallics can then be inferred from the behavior of the Al matrix. Fig. 4b details the true strain behavior of the Al matrix in both the binary and ternary alloys, revealing a three-stage behavior with anomalous lattice strains instead of the linear stress-strain behavior expected in a conventional Al alloy[25, 26]. Fig. 4c-d details how the load is partitioned or shared between the two phases present in the alloy. During the initial loading (Stage I), the Al matrix and intermetallic deform elastically under low stress (i.e. below 50 MPa). After early yielding, there is a transition to Stage II, denoted by red arrows in Fig. 4b, where additional stress leads to the Al matrix showing a decelerated lattice strain response while applied stress increases. During this stage the intermetallic phase carries an increasing share of the applied load. The increasing deformation observed at stage III is triggered (black arrows) once the dislocations reach a critical density and the intermetallic yields. Here, the Al matrix starts to take on more stress, indicated by the increase in slope, and the load partition rebalances between the two phases leading to destructive plastic deformation in the aluminum matrix. The transitive load behavior of Al-Ce and Al-Ce-Mg alloys is similar to the load partitioning characteristics of dispersion strengthened metal matrix composite alloys[27], which have been shown to exhibit high-strength and good thermal stability[28]. The reverse exchange behavior between phase II and III is attributed to elastic saturation in the fine intermetallic phase which leads to subsequent plastic flow in the ductile matrix accompanied by cracking and slip in the intermetallic. The cracking leads to isolated nanoparticles. A significant residual compressive strain exists in the hard Al11Ce3 phase while a slight tensile load resides in the soft Al matrix, which is expected behavior after unloading given the two-phase coexistence and complex load sharing. Comparing Fig 4c and 4d reveals that the strengthening mechanism of the Al-Ce alloy does not change following the addition of Mg. Instead, Mg increases the magnitude of the load which can be carried by the intermetallic phase before dislocation saturation and subsequent redistribution of load to the aluminum matrix. The difference is depicted by the shaded region in Fig. 4d.
The room temperature strength of the Al-Ce alloy family derives from the extremely fine distribution of dendritic intermetallic phases uniformly across the alloy which form during casting. The very low solubility of Ce in the solid Al matrix favors retention of this structure to very high temperatures compared to traditional casting Al alloys, and this is reflected in the superior retention of mechanical properties to above 300°C. These features lead to complex load-sharing in the binary Al-Ce alloy, where slight Mg addition markedly improves the material strength and offers a guide to further improvements in this new class of Al alloys.

Outlook

Cerium strengthened aluminum alloys exhibit highly desirable behavior for many applications: high ductility, robust room-temperature mechanical properties, exceptional high-temperature mechanical property retention, high tolerance to casting defects, and good castability across a broad range of compositions. In fact, the cast materials approach the mechanical properties of traditional wrought alloys. Given the high availability and low cost of cerium metal, these alloys are economically viable for large volume industries such as the transportation sector, where their properties make them ideally suited for vehicle light-weighting. Elimination or reduction of heat-treatment amplifies the economic and environmental benefits of light-weighting in the transportation sectors. Adoption of these alloys by industry will not only impact current technologies, but will provide the basis with which to develop the next generation of high temperature aluminum alloys. Finally, by creating demand for Ce, which is overproduced, the economics of rare earth mining improve, and reflect a step toward a stabilization of global production and diversification of the rare-earth supply chain.

Methods

CALPHAD Thermodynamics:

The phase diagrams presented herein have been thermodynamically assessed[15-17, 19] within the CALPHAD methodology, and the commercially available software Thermo-Calc has been used to calculate the equilibrium phase diagrams based on a user-defined thermodynamic database[29-31]. In this approach, the Gibbs energy of individual phases is modeled, and the model parameters are collected in a thermodynamic database. Models for the Gibbs energy are based on the crystal structures of the phases and interaction parameters are assessed to reproduce both the diagrammatic and thermodynamic data available for binary and ternary systems. For more details about the CALPHAD methodology, please refer to the classical textbooks on the subject.

Casting and sample characterization:

Alloys were cast using industrial practices. Industrial grade ~30lb aluminum ingots were brought to a molten state in a tilt pour resistive furnace. Once the metal was molten and temperature stabilized at 750 °C, alloying elements were added one at a time, with cerium being the final addition. If multiple compositions were being cast during a single trial, additional melts were prepared from the heel of the previous melt. Metal was poured into a ceramic lined permanent mold heated to 400 °C; each mold comprised two dog bone style test-bars 25cm in length. Each casting was removed from the heated mold and allowed to cool.
under ambient atmosphere. Selections of bars at each composition were heat-treated with either a T6 (10 hrs at 540 °C, warm water quenched and then artificially aged for 3 hrs at 150 °C) or T4 heat-treatment (10 hrs at 540 °C, warm water quenched). As-cast and heat-treated test-bars were mechanically tested in tension using a United Calibration and Testing Universal Testing machine.

For high-temperature measurements test-bar grips were threaded and bars were held at temperature for no less than thirty minutes. Once the bars reached an equilibrium temperature, they were strained under tension until failure.

Gauge lengths of fractured bars were sectioned into 2mm disks using a low-speed diamond saw. Disks were polished and etched using Keller’s reagent. Imaging was performed on a Hitachi S-4700 Cold field emission Scanning Electron Microscope. Phase analysis was completed via X-Ray diffraction using the Panalytical X’Pert Pro system combined with Rietveld analysis through use of the High-Score plus software suite.

TEM measurements:

For TEM sample preparation, 3mm disks were cut from fractured bars and mechanically polished to ~150 μm thick and then further electro-polished at -15° C using an 80% methanol/15% perchloric acid /5% HF electrolyte until electron transparent. TEM measurements were performed using the TitanX Scanning Transmission Electron Microscope at the National Center for Electron Microscopy at the Molecular Foundry located at Lawrence Berkeley National Laboratory. The TitanX was operated at 200 kV for the STEM EDS measurements.

SAXS /USAXS:

The USAXS data were collected on a combined Bronse-Hart/Pinhole SAXS/WAXS instrument at 9-IDC at the Advanced Photon Source located at Argonne National Laboratory[32]. All samples were prepared to varying thickness and exposed to a monochromatic X-ray beam of 24 keV for two minutes. With the sample thicknesses known, all USAXS data are calibrated and on an absolute scale[33]. Features in USAXS/SAXS arise from scattering of x-rays by inhomogeneities in electron density due to distinct phases and are sensitive to features with dimensions between ~1nm and 5μm.

Load partitioning and sharing studied through in-situ neutron diffraction under load:

The in-situ neutron diffraction experiment was conducted at the Vulcan diffractometer[34] at the Spallation Neutron Source (SNS), Oak Ridge National Laboratory. The cylindrical specimen of 10x20mm was mounted horizontally in the loadframe with the axial direction parallel to the loading direction. An extensometer was attached to the specimen for measuring the engineering strain. Compressive loads were applied at a rate of -10 MPa/min (negative denotes compression) with a 15-min dwell at -25 and -50MPa in the elastic region. To avoid stress relaxation during step wise loading during plastic deformation[25, 35, 36], the specimen was then continuously compressed at a strain rate of -0.01 h-1 until the strain reached -0.1. The specimen was subsequently unloaded at a stress rate of 0.8 MPa/min. The incident neutron beam, 45° to the loading direction, was focused at the center of the specimen, which remained stationary as a result of uniform displacement from both sides. The gauge volume was
defined to about 5×5×5 mm3 by the incident slits and radial collimators. During the mechanical test, the two detectors, located at -90° and +90° to the incident beam, continuously recorded the diffracted neutrons with the scattering vector parallel to the longitudinal and transverse directions, respectively. The diffraction data were averaged in a 15-min interval using the VDRIVE software. The lattice parameters were extracted from Rietveld refinement of entire scattering pattern using GSAS and EXPGUI software.

The lattice strain $\varepsilon$ of individual phases was calculated by $\varepsilon_x = (L_x - L_{x,0}) / L_{x,0}$ along a principal (i.e. crystallographic) axis, where the reference $L_{x,0}$ under zero stress was estimated using the result before loading[37]. The corresponding microstress ($\sigma_x$) of a phase along the particular axis was obtained by $\sigma_x = E_x \cdot \varepsilon_x$, where $E_x$ is the elastic diffraction constant. $\varepsilon_x$ was estimated from the linear unloading behaviors which is assumed as an elastic process, by using $E_x = \Delta \sigma_x / \Delta \varepsilon_x$, where $\Delta \sigma_x$ and $\Delta \varepsilon_x$ are the changes of stress and strain, respectively, during the elastic unloading along the particular direction. For the Al matrix phase, the lattice strain and microstress were measured against changes in the fcc lattice parameter “a”. For Al11Ce3 intermetallic phase, the behavior along the b-axis, one of the principal axes, was selected to represent the behavior of the phase.

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Author Contributions

DW, ORR, and ZCS produced the samples, PEAT, AP, ORR, and ZCS performed the modeling, JRIL, JAH, MB-H and TMW performed the SAXS/USAXS measurements and analysis, TTL provided TEM images and analysis, KA and YC performed the neutron measurements, ORR and SKM designed the experimental program and coordinated the project. AHK provided critical insight into the project. All authors contributed to the interpretation of results. ZCS, ORR, JRIL, AP, DW, and SKM wrote the paper.

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