



Automated high-throughput data collection and analysis of composition-process-structure relations by utilizing casting segregation in a Ni-base superalloy

Thomas Hoefler^{a,*}, Ayako Ikeda^a, Satoshi Utada^a, Makoto Osawa^a, Kyoko Kawagishi^a, Toru Hara^a, Takahito Ohmura^a, Daichi Akama^b, Masaki Taneike^b, Toshio Osada^{a,*}

^a Research Center for Structural Materials, National Institute for Materials Science, NIMS, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan

^b Mitsubishi Heavy Industries, Ltd., 2-1-1 Shinhamma, Arai-cho, Takasago, Hyogo 676-8686, Japan

ARTICLE INFO

Keywords:

High-throughput method
Image analysis
 γ/γ' Superalloy
Aging heat treatment
Casting segregation

ABSTRACT

We attempt to address the problem of generating large, reliable experimental datasets that interrelate composition, processing and microstructure in a short timeframe by combining a heterogeneously composed sample with a gradient heat treatment. Composition-Process-Structure datasets are essential for both physical and purely statistical models aiming to predict material behavior. Using the segregated microstructure of a cast single-crystal sample of gas turbine blade Ni-base superalloy MGA1400 in combination with a temperature gradient furnace, we cover a whole range of heat treatment temperatures and compositions according to elemental partitioning. Data collection uses automatic, correlative FE-SEM and EPMA analysis for microstructure images with corresponding local concentration. Image analysis combined with a precipitate shape fitting method using superellipses allows automatic evaluation of phase fractions, precipitate size/shape/aspect distributions as well as inter-precipitate distances and relative angles for investigating clustering and alignment phenomena. As a result, we were able to generate a pseudobinary phase diagram indicating the γ' solvus. The activation energy for coarsening of γ' was found to be mostly independent of as-segregated composition. Further, results showed lattice misfit between the γ and γ' phase to not only cause precipitate shape to vary with treatment temperature, but also cause precipitate aspect ratio to vary between the dendrite core and interdendrite regions, indicating the presence of dendritic stresses from solidification.

1. Introduction

The microstructure of materials is a key aspect affecting material properties. In turn, microstructure is greatly influenced by chemical composition and processing. Gaining a better understanding of these Composition-Process-Structure-Property (C-P-S-P) interrelations is essential for efficient materials development [1–5]. Superalloys, especially alloys for rotating turbine component applications, are a class of materials used under extreme conditions, requiring high-level structural reliability. They typically feature a large variety of alloying elements and require complex heat treatment processes under strictly controlled temperature conditions to achieve ideal properties and meet required reliability. Furthermore, even with the same alloy, the heat treatments must be optimized while accounting for unavoidable compositional variations

stemming from the casting process. Consequently, there are a large number of parameters needing optimization [6]. Price fluctuations and supply chain vulnerabilities also pose significant and unforeseeable challenges, often imposing additional difficulty on alloy design – optimization of alloying elements – besides optimizing for performance [7,8]. Therefore, a deep understanding of the interrelationships among composition, processing, microstructure, and properties (C-P-S-P) is invaluable not only for alloy design itself but also for determining composition and processing specifications that ensure the robustness and reliability of superalloy component productions.

While almost all commercially successful superalloy grades today are the result of laborious trial-and-error experiments building upon empirical relations, computational methods aiding the design of new alloys have been steadily gaining importance for several years [9–12].

* Corresponding authors at: Research Center for Structural Materials, National Institute for Materials Science, NIMS, 1-2-1 Sengen, Tsukuba, Ibaraki, 305-0047, Japan.

E-mail addresses: hoefler.thomas@nims.go.jp (T. Hoefler), osada.toshio@nims.go.jp (T. Osada).

<https://doi.org/10.1016/j.matdes.2026.116820>

Received 28 May 2026; Received in revised form 24 July 2026; Accepted 17 August 2026

Available online 18 August 2026

0264-1275/© 2026 The Authors. Published by Elsevier Ltd. This is an open access article under the CC BY license (<http://creativecommons.org/licenses/by/4.0/>).

Thermodynamic models such as those based on CALPHAD [4,13–19] or the Cluster Variation Method (CVM) [20,21] can predict equilibrium phase compositions for the purposes of optimizing the volume fractions of desirable phases (such as the strengthening γ' phase) and avoiding undesirable phases (e.g. TCP phases). Phase-field methods enable investigating solidification [22–25] and microstructure formation kinetics [26–31], often relying on CALPHAD data for equilibrium conditions [32–42]. Precipitate strengthening by hindered dislocation movement can be predicted from microstructures, especially the size, shape and spacing of precipitates [43,44]. Crystal plasticity models can be used to calculate stress–strain response at low strain rates [45–52] and under cyclic loading conditions [50,51,53–58] as well as predicting high-temperature creep behavior [53,54,59–63], with some works further using coupling to phase-field methods to include the effects of microstructure [59–61]. Empirical methods performing statistical analysis of large experimental datasets have also proven successful, such as the Alloy Design Program (ADP) developed at NIMS, using multivariate regression to find an appropriate alloy composition given a set of desired properties [64,65].

All of these methods still rely heavily on experimental data, with the quality of results highly depending on the data used. To this day, ab-initio methods without any reliance on empirical relations take a mostly supporting role in the creation of the datasets necessary [14,15,66,67]. On the other hand, emerging methods using artificial intelligence make statistical predictions from existing data (experimental or by simulation), with no (or only minimal) reliance on physical models for generating results [68–72], with many examples pertaining to superalloys [73–75]. The quality, consistency and acquisition speed of experimental results dictate the success of any predictive method. Even if modeling can be performed without any prior empirical data, promising results will need confirmation by experiment eventually.

High-throughput methods attempt to address this need for gathering large amounts of experimental data in an efficient manner. They typically entail correlative analysis of a graded sample (e.g. composition or processing gradient), relating the local conditions to the properties of interest. One of the earliest examples for the rapid generation of Composition-Process-Structure (C-P-S) datasets is the use of diffusion couples for exploration of phase diagrams [76], also in combination with temperature gradients for increased efficiency [77]. A single sample accounts for a whole range of compositions, replacing the need for several single-composition samples. Advances in image analysis and computer vision have further allowed the automatic evaluation of microstructures, including area fractions and sizes of grains, precipitates and particles. Superalloys are often investigated using diffusion multiples in order to capture the effects of the many alloying elements [78–83]. Furthermore, alternative approaches to introduce concentration gradients such as using the Bridgman method for zone remelting exist [84].

A common hindrance for the wide adoption of high-throughput testing methods is the difficulty of sample preparation. For example, the creation of diffusion couples is often hindered by poor bonding and porosity at the interface between the individual elements, while other methods such as additive manufacturing typically require highly specialized equipment. Another approach for the rapid generation of datasets is using data mining methods to automatically gather results from open literature, aggregating several decades worth of data [85–87]. However, data curation is a challenge, and the consistency of results produced by different researchers with different methods is questionable [88].

Samples accessible to high-throughput testing can also be generated using standard preparation methods. For most alloys, elemental segregation during casting results in local concentration gradients. While typically undesirable and therefore mitigated by homogenization heat treatment, these gradients can be used to relate local composition, microstructure and properties. Recent works have successfully investigated mechanical response in non-homogeneous cast samples using

nanindentation mapping [89,90]. While diffusion multiple methods cover much wider composition ranges for identification of desirable, undesirable or novel phases, segregation-based methods start from an already viable alloy with typically much smaller variations in concentration, making them especially suited for optimization of existing alloys.

In the present work, we combine the segregation-induced concentration gradients of a γ/γ' superalloy with a temperature-graded aging heat treatment, allowing investigation of C-P-S relations using one single crystal sample cast using standard directional solidification. The effects of local aging temperature and composition on the resulting microstructure are simultaneously analyzed by automated scanning electron microscopy (SEM) imaging and electron probe microanalysis (EPMA) spot analysis at 4500 sample locations. Automatic image analysis gives area fractions, size and shape information for each individual γ' precipitate as well as interparticle distances and precipitate alignment angles. From this, aging coarsening kinetics, clustering and alignment behavior of the γ' phase, thermodynamic equilibrium conditions for the creation of phase diagrams and local residual stresses can be deduced. We view the correlative analysis combining fast SEM and EPMA analysis to explore the composition (C) and processing (P, aging temperature) space of a complex alloy with subsequent extensive image analysis to extract the maximum amount of information from the SEM micrographs (S) in an automated manner as a significant improvement upon prior work.

2. Methods

2.1. Workflow of high-throughput automated evaluation

We expand upon the method presented in a previous work [91], using a sample with residual concentration gradients from segregation instead of a fully homogenized sample. An overview of the method is given in Fig. 1. A cast, cylindrical, single-crystal sample with a segregated microstructure is heat treated in a temperature gradient furnace, with the primary dendrite axis parallel to the temperature gradient. This results in a processing gradient over the length of the sample, while segregation causes local composition to vary mostly along the radial direction, normal to the primary dendrite growth axis. The specimen is then automatically sampled using SEM and EPMA over a regular grid spanning a few thousand points, while ensuring that the same locations are used across both instruments. Finally, compositions from EPMA spot analysis are related to processing temperature, which is inferred from the sample position in relation to the furnace temperature profile, and microstructure data gathered via image analysis from the SEM images to create a vast Composition-Process-Structure dataset from a single sample in a manner of a few days.

2.2. Temperature and composition gradient sample preparation

For the present work, a single crystal model alloy based on the industrial gas turbine blade superalloy MGA1400, designed by Mitsubishi Heavy Industry, was used, excluding the grain boundary strengthening elements C, B and Zr [92]. Nominal and actual compositions are given in Table 1. Samples were cast as single crystal round bars ($\varnothing$ 13 mm $\times$ 130 mm) to mitigate the effects of different crystal orientations. Following a solution heat treatment at 1230 °C for 1 h with subsequent quenching in unagitated room temperature water, a 5.8 mm $\times$ 80 mm long cylindrical sample was cut from the central part of one of the round bars using Electrical discharge machining (EDM). This sample then underwent a 2 h aging heat treatment in a vertical temperature gradient furnace described in previous works [91,93], covering the temperature range between 800 and 1310 °C that is relevant for homogenization, solutionization, forging and aging treatments. Next, quenching was performed by dropping the sample into an unagitated, room-temperature water bath below the furnace. The sample was then longitudinally cut into two

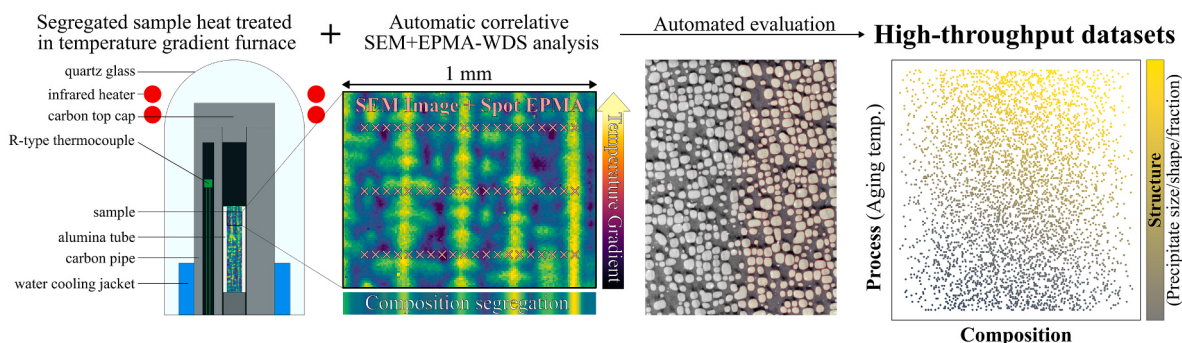


Fig. 1. Method for High-throughput generation of Composition-Process-Structure datasets, combining a gradient furnace [91] with a single-crystal sample featuring a segregated microstructure. Heating and cooling at opposing ends of the furnace generate a steady-state temperature gradient, which is mapped onto the sample as an aging temperature (processing) gradient. The sample is then analyzed using SEM and EPMA at the exact same locations in a regular grid, with each row (normal to the primary dendrite axis) corresponding to a single aging temperature and columns varying in composition due to segregation. Correlating the EPMA spot quantification with data gathered from SEM images by automatic image analysis allows relating microstructure to local aging temperature and composition, e.g. phase diagrams from local phase fractions.

Table 1

Composition by mass (wt.-%) for the model Ni superalloy based on MGA1400 used in the present study. Actual composition was determined as the mean of two ICP-OES measurements using aqua regia and HF as solvent.

	Al	Co	Cr	Mo	Ta	Ti	W	Ni
Nominal	4.0	10.0	14.0	1.5	4.7	2.7	4.3	Bal.
Actual	3.96	9.74	13.5	1.51	4.69	2.60	4.16	Bal.

halves along the (100) plane, which again were cut to 43 mm length to cover the whole length of the sample with an overlap region of 6 mm. Finally, the samples were polished up to 30 nm colloidal silica (vibratory polish).

The short, high-temperature solution heat treatment serves to dissolve the coarse eutectic γ' precipitated at inter-dendrite region during casting while keeping elemental segregation mostly intact, resulting in local concentration gradients over the span of several tens to hundreds of micrometers. This combines with the aging temperature gradient to give dual-gradient samples, as outlined in Fig. 2. Each of the two samples was analyzed using location-correlated SEM and EPMA analysis over a grid of 75×30 points, with a spacing of $574 \mu\text{m}$ and $41.2 \mu\text{m}$ for the high-temperature sample (sample 1), and $572 \mu\text{m}$ and $48.2 \mu\text{m}$ for the low-temperature sample (sample 2). Thus, a total of 4500 positions at 150 defined aging temperatures and varying compositions was investigated.

2.3. Automatic composition analysis via EPMA

EPMA-wavelength dispersive X-ray spectroscopy (WDS) (Shimadzu EPMA-8050G) was used as a high-throughput, quantitative composition analysis method to complement the microstructure data gathered via SEM. Due to the limited number and different types of analyzing crystals in the WD spectrometer, a maximum of four elements could be measured at a time. As measuring all relevant elements requires three passes and moving the crystals takes several seconds, only 60 positions evenly spaced across the samples were fully analyzed, while the rest used only a single pass for measuring Al, Cr, Ti and W. The remaining elements can be approximated by regression using the 60 fully analyzed points as standards. Supplementary figure 1 shows the correlation between X-ray intensity at the characteristic peak wavelengths of each element and its composition obtained by EPMA point analyses performed on all elements at 60 points within the range of 800–1140 °C (see appendix). It was confirmed that the composition of every element exhibits a linear relationship with X-ray intensity. For the point analysis of the four elements, the regression lines derived from this correlation were applied as

calibration curves to the measured characteristic X-ray intensities to determine the compositions of Al, Ti, Cr, and W.

2.4. Automatic microstructure analysis via FE-SEM

SEM microstructure images (1024×768 pixels resolution) were acquired using a Zeiss GeminiSEM 300 field emission (FE)-SEM at 1 kV using the In-Lens secondary electron (SE) detector at a working distance of approximately 5 mm. Under these conditions, the γ' phase appears brighter than the γ matrix phase. Magnification was scaled automatically so that the mean diameter of the as-aged, coarser γ' phase would be 25–30 pixels in each micrograph, ensuring consistent sampling even with the precipitate sizes changing significantly between the different aging temperature regions. These settings were chosen to provide a balance between the number of precipitates sampled, resolution and acquisition speed for each micrograph. Typically, precipitates below approximately 30 nm size could not be resolved well enough for automatic image analysis at the chosen imaging settings. In addition, the automatic magnification scaling also introduces a detection limit of about 1/15th the size of the mean diameter of the as-aged γ' phase, where any precipitates smaller than this would have less than two pixels diameter. To ensure that any ultra-fine precipitates (e.g. formed during the quenching step after heat treatment) unable to be resolved from a single image do not significantly affect phase area fractions, experimental results were compared with predictions using CALPHAD as well as the Alloy Design Program (ADP) developed at NIMS.

The individual precipitates were automatically identified using image analysis by slight restoration (bilateral filter for noise reduction and unsharp masking), automatic Otsu thresholding, watershed segmentation and statistical elimination of outliers based on relative brightness [94,95]. Differentiation between γ' precipitates formed mostly by Ostwald ripening during the gradient aging treatment (aging/secondary) and γ' formed during water quenching after the gradient treatment (cooling/tertiary) was performed based on their significantly different sizes by using a morphological top-hat filter, with a circular structuring element sized between the largest cooling and the smallest aging γ' based on size distribution data. The watershed segmentation using seed points gathered as local maxima from a Euclidean distance transformation ensured automatic resolution of partially coalesced precipitates. After initial segmentation, the fraction of contours shared with any directly adjacent segment labels was also used to correctly identify individual precipitates in the early stages of merging as well as ensure that irregularly shaped but non-coalescing precipitates are not erroneously split into multiple segments. A common case of the latter were slightly elongated, cuboidal precipitates where the distance transform introduced multiple seed points, resulting in multiple

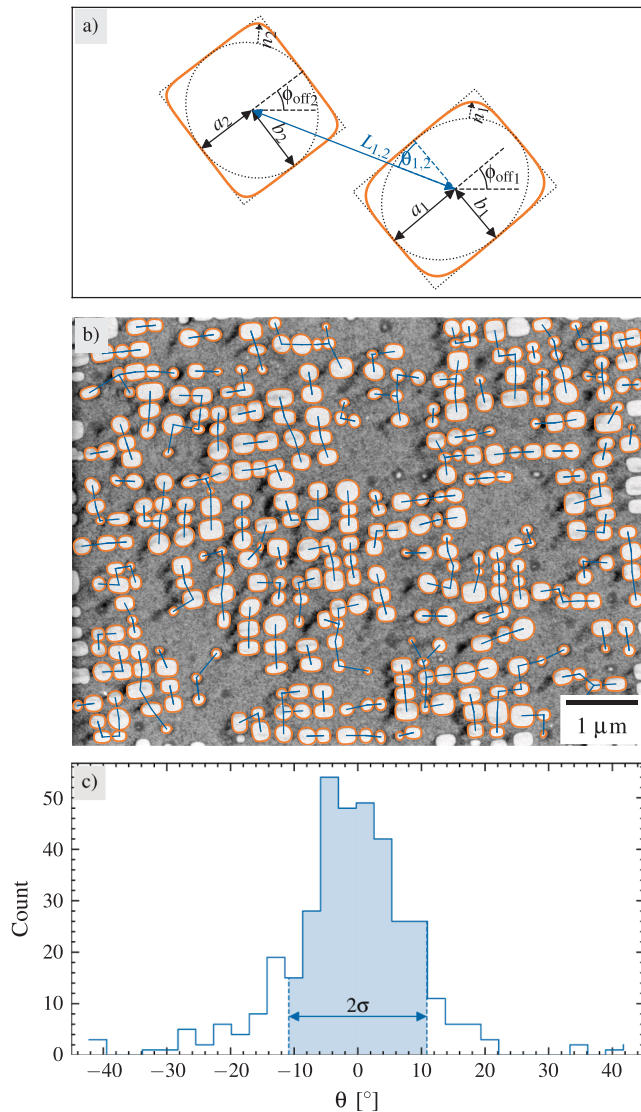


Fig. 2. Method for automatic microstructure geometry evaluation by image analysis. a) Superellipses (orange) defined by four parameters (a , b , n and ϕ_{off}), allowing a shape varying between ellipse and rectangle (shown with dotted lines for reference), are fitted to each γ' precipitate identified. Clustering and alignment are evaluated by finding the length (interparticle spacing, L) and angle to the closest principal axis (θ) of the vector (blue) connecting the centers of each particle with that of its closest neighbor. b) Exemplary evaluation result for a SEM image corresponding to an aging temperature of 1100 °C, showing alignment but no significant clustering. c) Resulting distribution of θ , where closeness to 0° implies alignment along crystal directions. The degree of alignment is evaluated as the standard deviation (σ) of the distribution. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

segments for a single precipitate without such a correction step. No significant amount of mislabeled precipitates was found after automatic segmentation, and no manual correction steps were performed.

While area fractions (f) and area-equivalent diameters (d) can be easily inferred from the area pixel counts for each precipitate, information on shape and higher-order microstructure features such as clustering or alignment along crystallographic orientations of precipitates requires a more in-depth analysis, described in Fig. 2. First, a superellipse is fitted to the contour of each precipitate, using the centroid as the origin (x_0 , y_0) (Fig. 2a). Superellipses can be seen as an extension of ellipses introducing the shape parameter n , given by Equation 1 [96]. Restricting n to the interval $[2, \infty)$, shapes varying from

ellipses ($n = 2$) to edge-rounded rectangles ($n > 2$) and approaching rectangles in the limit ($n \rightarrow \infty$) can be described. Thus, each isolated precipitate is described by its semi-principal axes (a , b), shape parameter (n) and an offset angle allowing description of the relative rotation of the precipitates (ϕ_{off}). While typical parameterizations ensure that $a \geq b$, we choose a to always be the length of the semi-principal axis closest to the direction of the temperature gradient (the width axis of each micrograph) to allow for discussion of precipitate aspect ratios in relation to the local structure and composition. This also implicitly restricts ϕ_{off} to the interval $(-45^\circ, 45^\circ]$.

$$r(\phi) = \left(\left| \frac{\cos(\phi - \phi_{off})}{a} \right|^n + \left| \frac{\sin(\phi - \phi_{off})}{b} \right|^n \right)^{-\frac{1}{n}} \quad (1)$$

Clustering and alignment are evaluated by finding the nearest neighbor for each precipitate, depicted as blue lines connecting the precipitate centers in Fig. 2a ($L_{1,2}$) and b. Interparticle distances can be directly gathered as the length of these lines, allowing relation to clustering phenomena by comparison to statistically expected mean distances. Assuming precipitates to be regularly spaced on a square lattice, the expected mean particle spacing is given by Equation 2, with the volume fraction f and the particle diameter d [97–99].

$$L = \sqrt{\frac{\pi}{6f}} d \quad (2)$$

Alignment is evaluated by calculating the angle θ of the vector connecting the precipitate centers to the closest principal axis. Values closer to zero indicate higher degree of alignment for individual pairs of precipitates, particularly for cuboidal precipitates where the faces typically align with crystallographic planes. To measure the overall alignment for all precipitates, we use the distribution of θ , an example of which can be seen in Fig. 2c. Assuming completely random arrangement of the precipitates, we would expect a uniform distribution of θ with standard deviation $\sigma = 25.98^\circ$. In contrast, the given example exhibits a high degree of alignment, resulting in a histogram resembling a normal distribution with a significantly lower standard deviation. Thus, we use the standard deviation σ of θ as a measure for alignment, with lower values indicating stronger alignment.

3. Results

3.1. Initial microstructure and composition

The water-quenched microstructure after solution treatment showed uniformly distributed γ' precipitates with a mean diameter of approximately 50 nm, with no significant dependence on local composition. Somewhat surprisingly, an area fraction of about 50 % γ' was observed despite the rapid cooling conditions, indicating quick precipitation kinetics, which is however consistent with previous literature [91,100–103].

Composition data indicating the segregation between the dendrite core and interdendrite regions is given in Table 2, using data from all 4500 EPMA-WDS spot measurements. The elements in order of increasing amount of partitioning as determined by $\frac{c_{95}}{c_5} - 1$, with c_{95} and

Table 2

Overall composition by mass (wt.-%) for the high-throughput superalloy samples as measured by EPMA-WDS spot analysis. Mean and median compositions were equal at the given precision. The 5th percentile and 95th percentile concentrations indicate the amount of segregation for each element. Shaded columns denote elements partitioning to the dendrite core region, while unshaded partition to the interdendrite region.

	Co	Cr	Mo	W	Al	Ni	Ta	Ti
5th percentile	9.5	12.8	1.5	3.2	3.9	59.1	4.0	2.2
Mean/Median	9.7	13.5	1.5	4.2	4.0	59.8	4.7	2.6
95th percentile	10.0	14.0	1.5	5.2	4.1	60.6	5.3	3.1

c_5 denoting the 95th and 5th percentile values, are Mo, Ni, Al, Co, Cr, Ta, Ti, W. For the present alloy, Co, Cr, Mo and W were found to partition to the dendrite core region, while Al, Ni, Ta and Ti featured higher concentrations in the interdendrite region.

An overview of the segregated microstructure over the whole sample length is given in Fig. 3. Local aging temperatures (Fig. 3a) are determined by cubic spline interpolation from measurements made with a trailing type R thermocouple in the unloaded furnace. The sample overlap region marked in blue serves to prevent cutting losses and mitigate the influence of potential preparation errors (e.g. edge rounding, phase relief) on microstructure analysis. A mostly qualitative visualization of the casting segregation is given in the form of SEM-EDS mappings for the relevant elements (Fig. 3b). To allow a uniform depiction of the widely varying intensities (counts) for the different spectral lines, the intensities are normalized by the total count rate (CPS: counts per second) and then converted into percentile ranks. W, Ti and Cr showed large differences in signal intensity between the dendrite core and interdendrite regions. Segregation remained consistent up to approximately 1230 °C (solvus region), while higher temperatures showed significant homogenization and thus loss of concentration gradients.

A comparison of EPMA spot measurements with a magnified view of the SEM-EDS results in the area outlined in red in Fig. 3 is given in Fig. 4. As the slowest diffusing element among alloying elements, W showed the largest mass fraction difference due to segregation and was thus chosen as reference element in the following analyses. While the EDS setup used does not readily allow for quantitative analysis, the relative intensities are consistent with EPMA results, showing higher intensities in the mapping directly corresponding to higher concentrations.

The primary dendrite arms in the (100) cross section were found to be spaced approximately 200–250 μm apart. Taking only the concentration gradients normal to the primary dendrite arms – which we expect to be much larger than in the parallel direction – into account, we can calculate the absolute gradient values given in Table 3 from the spot measurements. These values can serve as a reference for the expected amount of concentration difference within each individual SEM micrograph, with image heights (normal to the primary dendrite axis) varying between 1.07 μm and 11.3 μm according to local aging temperature (and therefore roughly scaling precipitate size). Using the 95th percentile of the W concentration gradient and the maximum image height, we can calculate a worst-case deviation of ± 0.16 wt-% from the concentration measured in the image center by EPMA spot analysis, while we would expect ± 0.06 wt-% when using the mean gradient value. Further, since

most micrographs use a much higher magnification (i.e. smaller image height), we assume the deviation in virtually all cases to be within measurement error.

3.2. SEM-EPMA correlated analysis

A selection of SEM images covering most of the range of aging temperatures and compositions observed over 4500 sample locations is given in Fig. 5. Since the images were rearranged according to W concentration as measured by EPMA, the layout here differs from the actual physical location on the samples, i.e. neighboring images are not necessarily from neighboring positions. Magnification was automatically scaled so that the apparent size of aging (secondary) γ' precipitates is similar across the whole range of aging temperatures. The cooling (tertiary) γ' phase formed during water quenching after the temperature gradient aging treatment can only be seen in the top row in the form of tiny speckles, in contrast with the much larger, clustered aging precipitates. The decrease in aging precipitate area fraction with both increasing temperature and amounts of γ -stabilizing W can be clearly seen, with the right half of the top row showing the microstructure after water quenching from the supersolvus state.

Looking at the size and shape of the aging precipitates, we can see no obvious dependence on the local composition. Coarsening seems to depend almost exclusively on aging temperature, with the bottom row (892 °C) featuring only a very slight degree of coarsening as compared to the initial microstructure (58 nm as opposed to 50 nm). The second row from the bottom (1002 °C) exhibits a high frequency of pear- and L-shaped, coalesced precipitates. The precipitates are almost perfectly spherical up to this temperature, again with no apparent effect of local composition. A transition to a cuboidal shape occurs around 1100 °C, as evidenced by the images in the third row from the bottom (1107 °C), accompanied by strong coarsening and alignment of precipitates. Finally, the top row (1216 °C) shows a departure from randomly spaced precipitates to a clustered microstructure in addition to alignment, featuring strings of aging γ' precipitates.

3.3. Microstructure image analysis

3.3.1. Phase fractions

Area fractions of γ' precipitates as determined from cross-section SEM micrographs are given in Fig. 6. Both local composition, indicated by coloration of the individual data points, as well as the aging temperature on the x-axis were found to highly affect the local phase

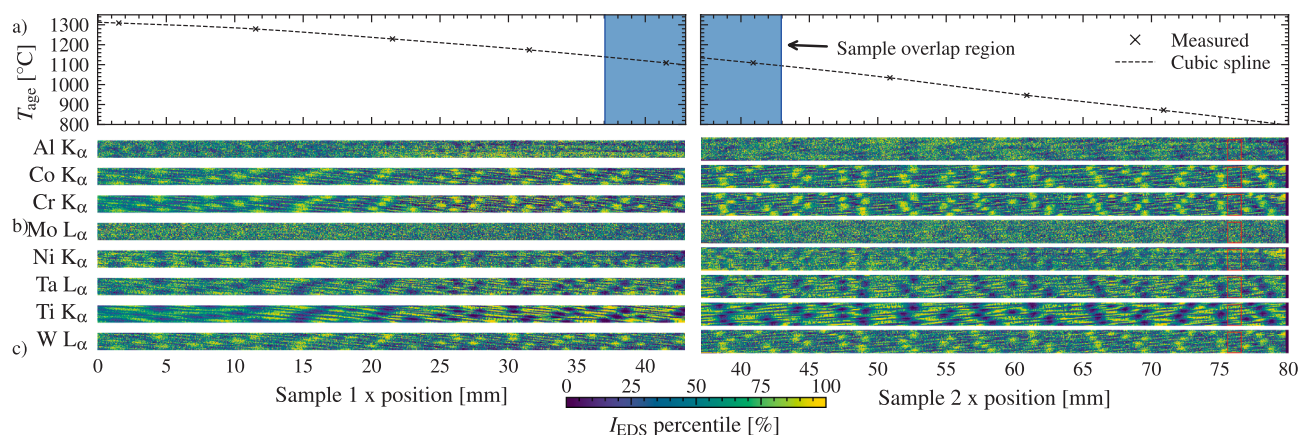


Fig. 3. Dual-gradient single crystal superalloy sample halves investigated in this work. A 2-hour aging temperature gradient imposed over the length of the sample (x-axis) by a temperature gradient furnace combines with residual elemental segregation from casting, allowing investigation of Process-Structure relations over a wide range of compositions and processing temperatures. a) Aging temperatures (T_{age}) interpolated using cubic splines from thermocouple measurements in the unloaded furnace, with the overlap region featuring equal aging temperatures between the two halves (sample 1 and 2) cut from a 5.8 mm $\times$ 80 mm long round bar is marked in blue. b) CPS-normalized intensity (I_{EDS}) percentile mappings for all alloying elements. Sections marked with red rectangles are shown magnified in Fig. 4. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

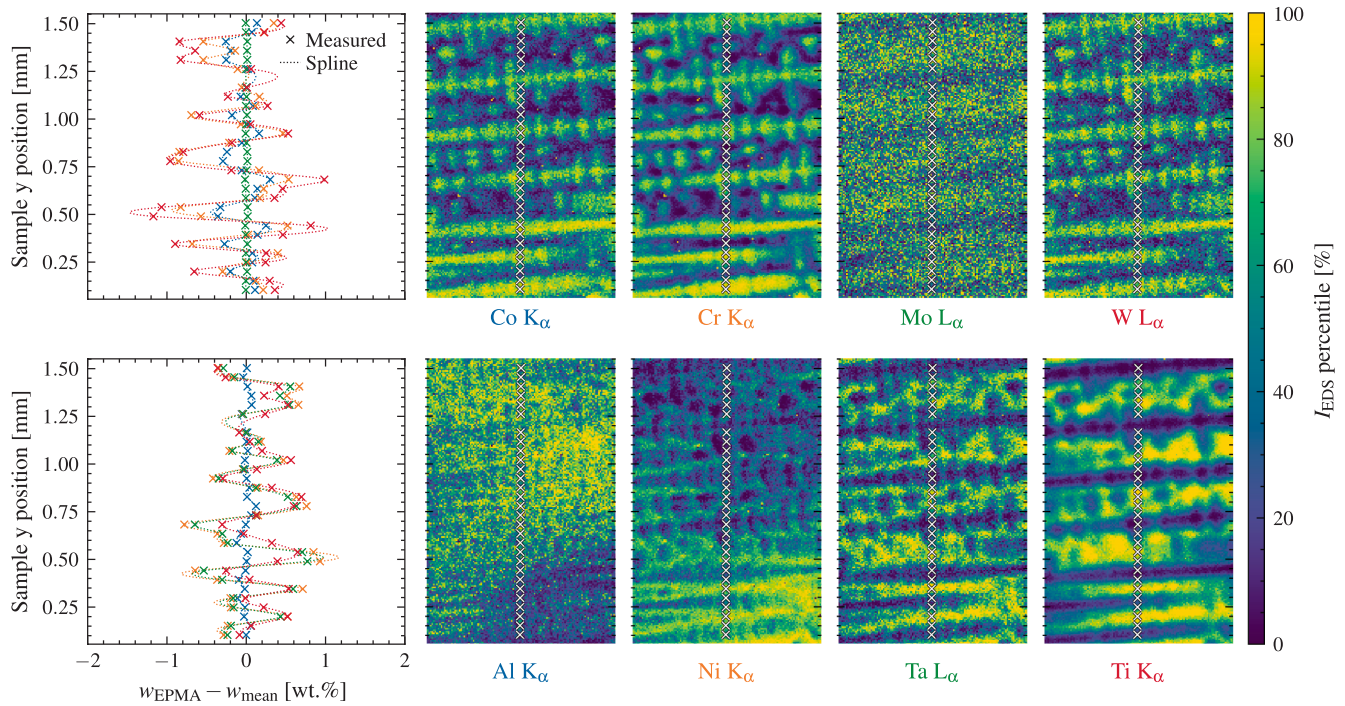


Fig. 4. Elemental segregation shown for a single scanned column (30 spots) corresponding to an aging temperature of 827 °C. Mass fraction differences between EPMA-WDS spot measurements (w_{EPMA}) and mean composition (w_{mean}) are compared to full sample SEM-EDS mappings magnified from the sections marked in red in Fig. 3. The top row shows the elements partitioning mostly to the dendrite core regions (γ' destabilizing) and the bottom row those partitioning to the interdendrite regions (γ' stabilizing), with the element label colors of the SEM-EDS mappings linking to the EPMA-WDS graphs on the left. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Table 3

Overall absolute composition gradient normal to the primary dendrite axis by mass (wt.-%/ μm) for the high-throughput superalloy samples as measured by EPMA-WDS spot analysis. Median and mean differ significantly due to the approximately half-normal distribution. The 95th percentile is intended to approximate the maximum gradient to be expected for each element.

	Co	Cr	Mo	W	Al	Ni	Ta	Ti
Median	0.0027	0.0056	0.00014	0.0092	0.001	0.0069	0.0067	0.0026
Mean	0.0034	0.0074	0.00017	0.011	0.0014	0.0084	0.0082	0.0037
95th percentile	0.0084	0.020	0.00042	0.028	0.0036	0.021	0.021	0.010

composition. The coarser aging γ' area fraction (f_{age}) is assumed to be representative of the local equilibrium state and compared with CALPHAD predictions by Thermo-Calc as well as the Alloy Design Program (ADP) developed at NIMS (Fig. 6a). Thermo-Calc with the TCNI12 database showed a rather large discrepancy compared to the experimental data. The Ni-DATA (ver. 8) database provided by Thermotech Ltd. showed similarly large deviations. While the cuboidal precipitate shape at higher temperatures above 1100 °C causing the area fractions measured from cross sections to differ from the actual phase (volume) fractions can explain part of this discrepancy, it does not address the difference of about 50 °C between solvus temperature from experiment and calculation. In contrast, the empirically based ADP purposely designed for multi-element superalloys agrees very well with observations, especially at the low and high ends of the temperature range. Again, the difference between area and volume fractions for the non-spherical precipitates at intermediate temperatures seems to cause some discrepancy. However, the amount of variation due to local composition was found to agree rather well with both predictions, with the height of the shaded regions from both Thermo-Calc and ADP similar to the degree of scattering depending on composition for the sampled aging temperatures.

Fig. 6b gives the results for cooling γ' (f_{cool}) recovered from the same microstructure images. With the image magnification set according to the coarser aging γ' precipitates, the fine cooling γ' precipitates are

represented by only a few pixels in each image, thus limiting accuracy. Nevertheless, the results are consistent: decreasing amounts of aging γ' phase at temperatures above about 1050 °C result in increasing precipitation of cooling γ' during water quenching after aging treatment. This is further apparent when looking at the total area fractions (f_{total}) in Fig. 6c, which remain mostly constant and close to the 52 % equilibrium volume fraction at 800 °C predicted by both Thermocalc and ADP. Again, results indicate that almost complete precipitation of γ' phase even during rapid cooling by water quenching.

3.3.2. Aging γ' precipitate morphology

Results for the area-equivalent diameter and superellipse fitting for the individual aging γ' precipitates identified by segmentation and labeling from SEM micrographs are given in Fig. 7. Ostwald ripening accelerating with increasing aging temperature is apparent in Fig. 7a, showing the as-aged area-equivalent mean apparent diameter from cross sections on a logarithmic scale. No obvious influence of composition (indicated by the color scale on top of the graph) is visible, but regions rich in W at intermediate aging temperatures appear to feature slightly smaller mean diameters.

Separate from the diameters determined from the area of each precipitate, fitting superellipses to the precipitates allows measuring the semi-principal axis lengths a and b , where a was chosen as the axis closer to the direction of the aging temperature gradient. From these lengths,

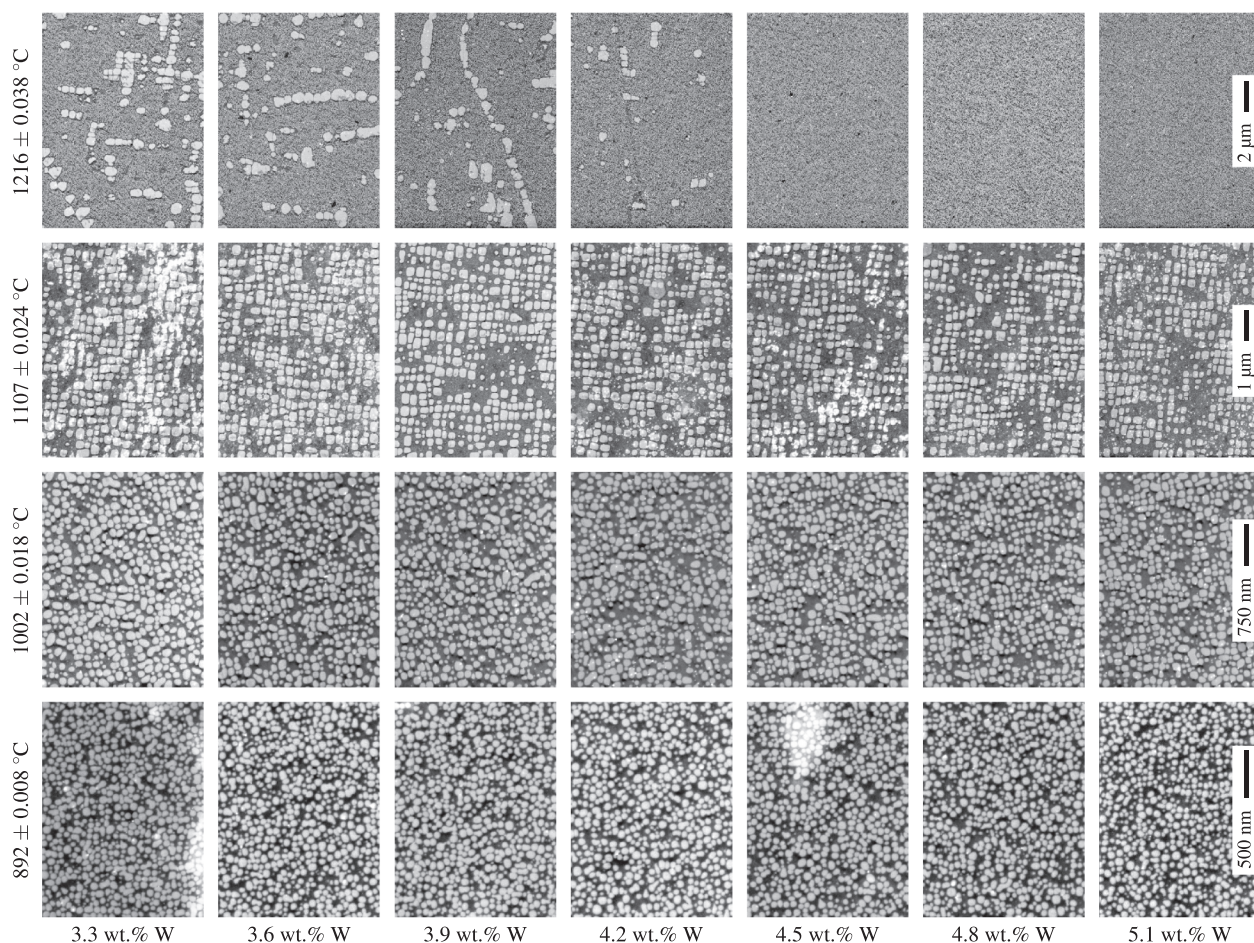


Fig. 5. Compilation of SEM microstructure images for four different aging temperatures over seven different W concentrations as determined by EPMA-WDS. Images are oriented so that the aging temperature gradient is upwards, with the ranges ($\pm$) given indicating the temperature difference between the high- and low-temperature facing sides of the scanned regions. γ' precipitates appear as a lighter gray against the darker γ matrix. The rightmost three columns (4.7 wt% W and above) of the top row (1216 °C) show the supersolvus state after water quenching. Note the differing scales between the rows (aging temperatures).

the aspect ratios given in Fig. 7b could be calculated. There appears to be a slight bias towards precipitates elongating along the temperature gradient axis ($\frac{a}{b} > 1$), with approximately 70 % of measured positions falling into this region. Moreover, there is a gap at equal aspect ratio, which grows more prominent at higher aging temperatures greater than about 1100 °C. This is also where an influence of local composition becomes apparent: Higher W contents result in γ' precipitates stretching along the temperature gradient, while lower contents cause elongation in the normal direction. This is exemplified by the inset images shown, with the image horizontal aligning with the temperature gradient.

Aging γ' precipitate shape as evaluated by the superellipse shape parameter n is given in Fig. 7c. Lower aging temperatures feature spherical precipitates, even when significant coarsening has occurred. The two leftmost inset images show the shape mostly unchanged in the region between 800 and 1050 °C. At higher temperatures, precipitates grow increasingly cuboidal up to a maximum at about 1175 °C, as shown by the third and fourth inset images from the left. At aging temperatures above this maximum, precipitates appear to become more spherical again, as exemplified by the rightmost inset image. As with the sphere-equivalent diameter, no significant influence of local composition was apparent.

3.3.3. Aging γ' inter-precipitate geometry

Increasing aging temperatures were found to correlate with an increase in both clustering and alignment phenomena, as demonstrated by Fig. 8. Inter-particle distances (center-to-center) expected for regularly

spaced, spherical precipitates were calculated using Equation 2 with the mean area fraction data given in Fig. 6 and the sphere-equivalent apparent mean diameter in Fig. 7.

An evaluation of clustering phenomena is given in Fig. 8a, showing the inter-particle distance experimentally determined by SEM image analysis compared with values calculated using Equation 2, assuming regular spacing on a square lattice. We can see good agreement up to an aging temperature of about 1100 °C, as can be seen for the regularly spaced microstructures in the left and center inset images. At higher aging temperatures, the observed spacing becomes significantly lower than the predicted values, indicating clustering above this temperature. The rightmost inset image shows an example of a clustered microstructure in this region. While the cuboidal shape reaching a maximum at around 1150 °C (see Fig. 7c) differing from the assumption of spherical precipitates in Equation 2 might contribute to this deviation, the gap between model and experiments only widens up to and above 1200 °C, where precipitates were observed to return to a more spherical shape.

Alignment effects can be discerned from Fig. 8b, showing the standard deviation σ of the inter-particle angle θ of pairs of closest neighboring aging γ' precipitates as defined in Fig. 2a over the aging temperature. At low temperatures (below 950 °C), the angle distributions are close to a fully uniform distribution with a standard deviation of about 26° (indicated by the dashed line), indicating no significant alignment effects. A gradual transition to a strongly aligned microstructure occurs between 1000 °C and 1100 °C, reducing the standard

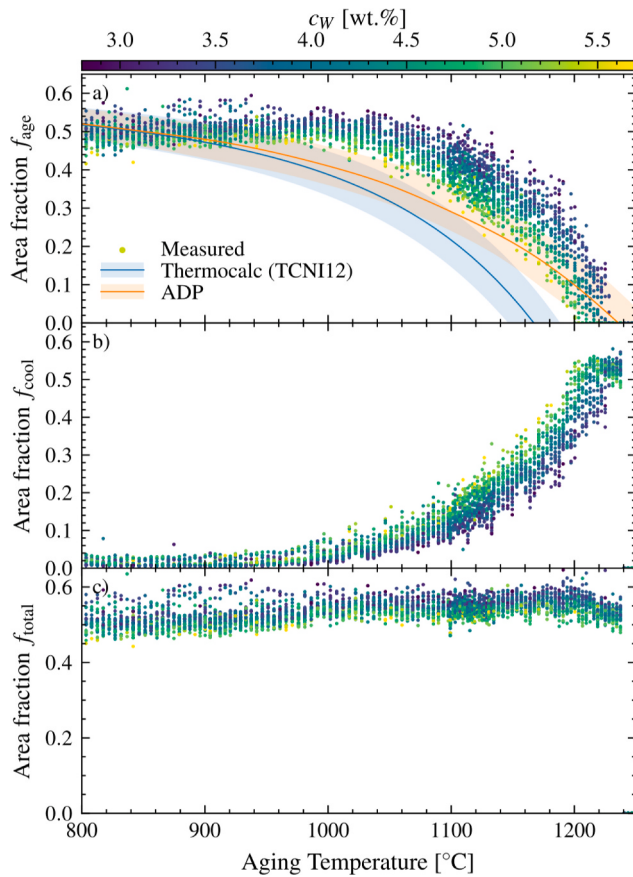


Fig. 6. γ' precipitate area fractions determined by automatic image analysis depending on local aging process temperature. a) Aging (secondary) γ' (f_{age}) including equilibrium predictions by the Thermocalc TCNI12 database (blue) and the Alloy Design Program (ADP) developed at NIMS (orange), with the center line indicating results for the mean composition and the shaded region giving the range for the 5–95 % percentile range of measured compositions, b) cooling (tertiary) γ' formed during water quenching after the aging treatment, c) total area fraction of γ' formed. Composition is visualized according to the contour bar at the top of the graph.

deviation to about half of its original value. Comparing the left and right inset images, the contrast between non-aligned and aligned microstructures can be seen.

4. Discussion

4.1. Phase diagram generation from high-throughput SEM-EPMA data

Using the local aging temperature depending on gradient sample position given in Fig. 3, the compositions as measured by EPMA spot analysis and the area fractions of aging γ' gathered by image analysis of SEM micrographs given in Fig. 6a, we can easily determine the γ' solvus temperature according to local composition. It was assumed that the 2 h

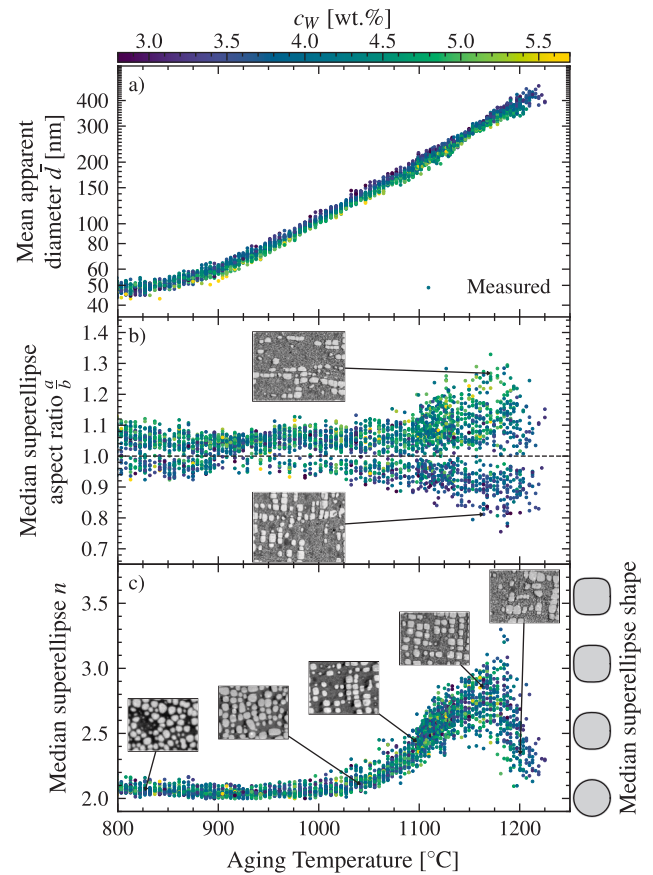


Fig. 7. Aging γ' precipitate size, aspect ratio and shape depending on aging temperature, automatically determined by superellipse fitting following image analysis. Composition as measured by EPMA spot analysis is visualized according to the colorbar at the top of the graph. a) circle-equivalent diameter calculated from label pixel counts after segmentation. b) Precipitate aspect ratio of the axis length along the temperature gradient (a) and its normal (b), with equal aspect outlined by a dashed line. Image insets show examples of vertically and horizontally stretched γ' microstructures. c) Superellipse shape factor, with $n = 2$ being an ellipse and higher values approaching cuboidal shapes. Image insets depict the transition from spherical to cuboidal shapes with increasing aging temperature.

(especially for the slowly diffusing elements such as W) and slightly affecting the phase fractions obtained. A more thorough investigation of this temperature range would require separate experiments, using significantly longer treatment times.

The resulting phase diagram section is presented in Fig. 9, showing the γ/γ' and γ stability regions with the phase boundary describing the composition-dependent solvus temperature, which was determined by fitting a quadratic surface to the non-zero aging γ' area fractions above 1150 °C (see Equations 3 and 4) and is shown as a green line. In addition, the solvus temperature calculated using Thermocalc with the TCNI12 database is shown as a blue line.

$$f_{age,sol}(c_W, T) = -3.96 \cdot 10^{-5} T^2 - 9.83 \cdot 10^{-4} c_W T + 0.115 T + 1.47 \cdot 10^{-3} c_W^2 + 1.33 c_W - 83.5 \quad (3)$$

gradient aging treatment would result in a microstructure sufficiently close to equilibrium conditions. Deviations from equilibrium are expected to be larger for the lower end of heat treatment temperatures, particularly affecting elemental partitioning among the γ/γ' phases

$$T_{sol}(c_W)[K] = 1457.41 - 12.4 c_W + 128.05 \sqrt{0.0116 c_W^2 - 0.163 c_W + 1} \quad (4)$$

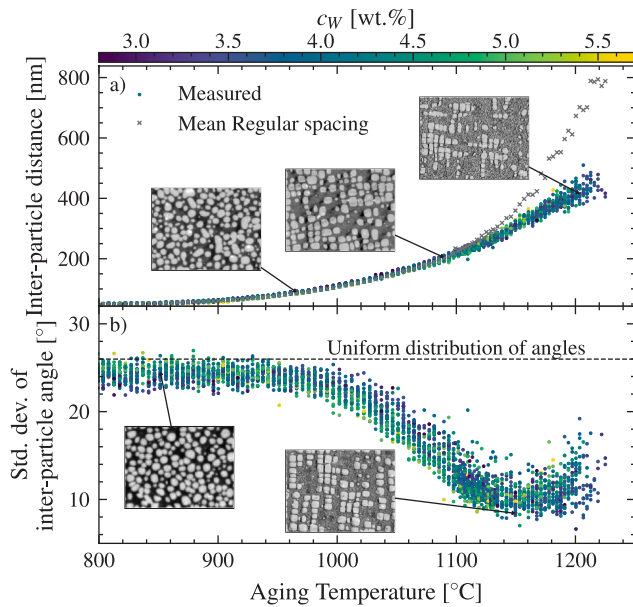


Fig. 8. Aging γ' inter-particle geometry relations describing clustering and alignment depending on aging process temperature, considering the closest neighbor of each individual particle. Composition is visualized according to the contour bar at the top of the graph. a) Mean inter-particle spacing (L) compared with regularly spaced precipitates as a measure for clustering (lower is more strongly clustered). Image insets show examples of non-clustered (left, center) and clustered (right) microstructures. b) Standard deviation of the inter-particle angle (θ) as a measure for alignment (lower is more strongly aligned). Image insets give examples of non-aligned (left) and strongly aligned (right) microstructures.

Looking at the montage of SEM microstructure images scaled to equal magnification in Fig. 9a, the solvus line can easily be visually determined as the lower boundary of the region where no large brighter aging γ' phase is present. The effect of composition is also immediately apparent, with higher concentrations of γ' -destabilizing elements leading to a decrease of the solvus temperature by a few tens of degrees. Thus, even without any fitting or even quantification of volume fractions, the solvus temperature can be easily discerned just from the SEM micrographs alone. While there is a sense of continuity between the individual images, it is important to note here that the images are rearranged according to local composition, resulting in the image positions in the montage differing from the actual physical locations on the gradient samples.

Fig. 9b shows all the data points sampled with the aging γ' area fractions color-coded, also showing the region that Fig. 9a covers in the form of images. Quantification of phase fractions by image analysis and surface fitting close to the solvus region allows a more accurate determination of the solvus line. It is notable that while there is a significant difference of about 45 °C between the predicted and experimentally determined solvus lines, their slope in relation to composition is very close. Similarly, the diameter d , superellipse shape parameters n , aspect ratio a/b , and inter-particle angle standard deviations obtained by our method can also be mapped onto a phase diagram as shown in Appendix as supplementary figures 2, 3, 4 and 5, respectively.

In order to calculate the γ' area fraction at any given temperature and reference element composition, we fit the logistic model given in Equation 5 to all measured data points. In addition to the previously determined solvus temperature in Equation 4, we used a bilinear fit for the high area fraction region at the low temperature end ($f_{age,max}$, Equation 6) and assumed a linear dependency of the parameters k and ν on the W concentration (Equations 7 and 8). The resulting fitted surface is shown in Fig. 10 together with the measured values. The coefficient of

determination was calculated as $R^2 = 95.7\%$.

$$f_{age}(c_W, T) = f_{age,max}(c_W, T) \cdot \left(1 - \left(\frac{2}{1 + e^{k(c_W) \cdot (T_{sol}(c_W) - T)}} \right)^{\frac{1}{\nu(c_W)}} \right) \quad (5)$$

$$f_{age,max}(c_W, T) = -2.94 \cdot 10^{-5} c_W T + 0.0153 c_W + 2.34 \cdot 10^{-4} T + 0.319 \quad (6)$$

$$k(c_W) = 6.89 \cdot 10^{-4} c_W - 1.31 \cdot 10^{-3} \quad (7)$$

$$\nu(c_W) = 0.0309 c_W - 0.0652 \quad (8)$$

As can be seen from Figs. 9 and 10, the compositional variation is rather small when compared with the larger ranges typically covered by diffusion couples or multiples. In addition, due to the nature of microsegregation, elemental compositions are mostly dependent on each other (e.g. regions rich in W are also rich in Cr and Mo). However, specimens can be readily prepared as single crystals, reducing complexity of the microstructural analysis by eliminating the need to account for different crystallographic orientations influencing the apparent phase area fractions.

4.2. Composition-dependent γ' coarsening kinetic analysis

Using the area-equivalent diameters determined in Fig. 7a, we can perform a coarsening kinetic analysis using the LSW model proposed by Lifshitz, Slyozov and Wagner [104,105]. Assuming a cubic coarsening kinetic, the diameter d at any given time t can be calculated from the initial diameter d_0 before aging and a coarsening rate constant K , as described in Equation 9. Various models for calculating the rate constant K exist [106–111], but it is typically assumed to be proportional to the γ'/γ interfacial energy γ_{IF} , the molar volume V_m of the γ' phase, the molar concentration c of diffusing species, the reciprocal temperature $\frac{1}{T}$ and the diffusion constant D . Neglecting the temperature and composition dependence of all parameters but the diffusion constant, we can determine K by linear regression using the Arrhenius-like model given in Equation 10. The prefactor A aggregates the temperature- and composition-invariant parameters, while the activation energy E_A stemming from the Arrhenius approximation of the diffusion constant is approximated to linearly depend on c_W .

$$d^3 - d_0^3 = 8Kt \quad (9)$$

$$\log(KT) = \log(A) - \frac{E_A(c_W)}{R} \frac{1}{T} \quad (10)$$

The initial diameters before aging were approximated by linear regression using experimental data, resulting in $d_0[\text{nm}] = 0.59c_W + 47.62$. Fitting results for the rate constant K and diameter d are visualized in Fig. 11, with $A[\text{m}^3\text{s}^{-1}\text{K}] = 1.44 \times 10^{-9}$ and $E_A[\text{kJ mol}^{-1}] = 1.73c_W + 303.2$ gathered from the regression shown in Fig. 11a. There appears to be a slight positive deviation of the rate constant from the fitted model at higher temperatures above 1140 °C, likely influenced by increasing misfit stresses, as also evidenced by the increasingly cuboidal morphology, alignment and clustering of the γ' phase (see also Fig. 7c and Fig. 8) [112]. Despite the γ' volume fraction varying greatly over the different aging temperatures and compositions, no effect on the coarsening rate constant could be inferred from the data. It is likely that the decreasing effect of lower volume fractions (due to precipitate coalescence occurring less frequently) and the increasing effect of the greater elastic strain energy at higher temperatures cancel each other out to some degree.

Using the fitted coarsening rate constant to predict the as-aged diameters after aging and comparing them to the experimentally obtained values shows good agreement, as can be seen in Fig. 11b. It is surprising that local composition has such little effect on the coarsening kinetics, with the activation energies between the interdendrite and dendrite core regions varying by less than 5 kJ mol^{-1} . However, the activation energy increasing with increasing W concentration is consistent with W

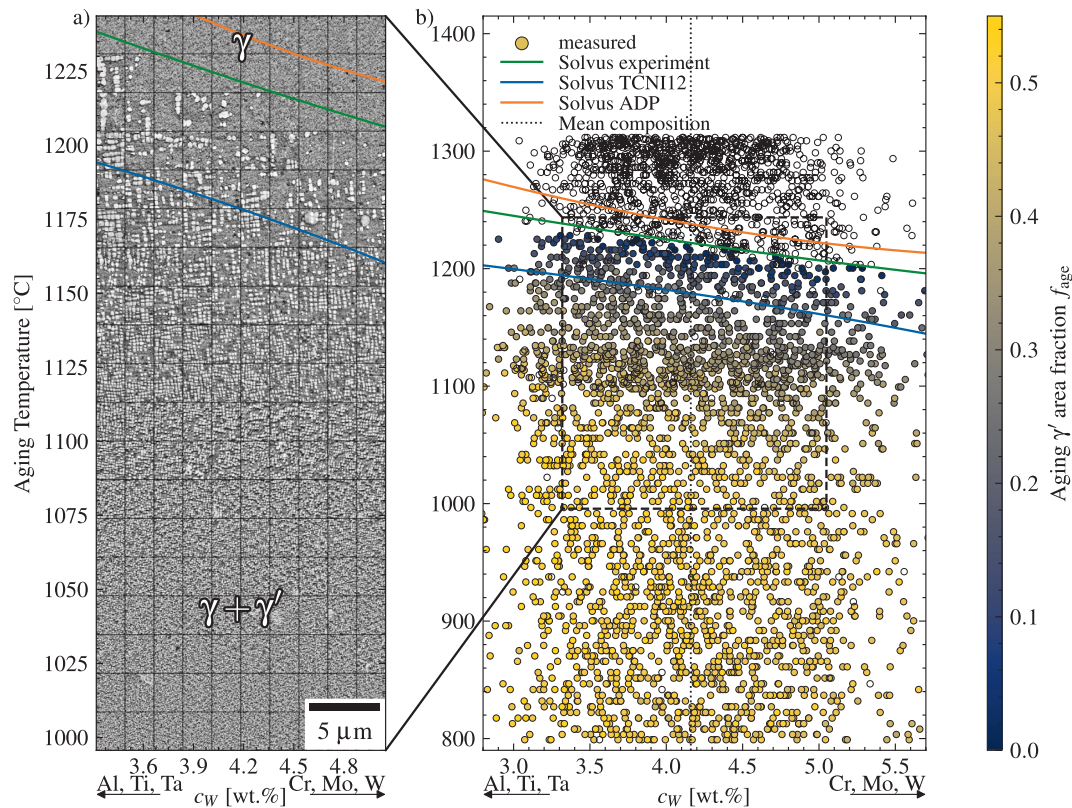


Fig. 9. Phase diagram section for the model alloy based on MGA1400, using W concentration as a reference to distinguish between interdendrite regions rich in γ' -stabilizing elements (Al, Ti, Ta) and dendrite core regions rich in γ' -destabilizing elements (W, Cr, Mo) on the lower and higher sides of the x-axis, respectively. a) Montage of SEM InLens-SE images rearranged according to local aging temperature and composition as measured by EPMA spot analysis. The displayed region is limited to the region where significant aging has occurred up to the solvus temperature. b) Area fractions determined from the SEM images for 4500 sample positions, where filled circles are colored according to the color scale on the right to represent the detected fraction, while empty circles are positions where no aging γ' was identified. The region which a) covers to is outlined with dashed lines.

featuring the highest activation energy of diffusion among all the alloying elements [113].

4.3. Effects of γ/γ' lattice misfit and dendritic stresses on γ' morphology

Calculations of the lattice misfit between the γ and γ' phases using the JMatPro material properties simulation software give 0.03 % misfit at room temperature, steadily decreasing to -0.05 % at 800 °C, -0.14 % at 1000 °C and -0.37 % at 1140 °C [16,17]. In our sample, cuboidal γ' appeared above approximately 1000 °C (see Fig. 7c). Coherency stresses caused by the increasingly negative misfit play a significant role in the coarsening kinetics [6,112].

The aspect ratios of γ' precipitates differing according to location inside the dendrite structure (see Fig. 7b) can be explained by residual dendritic stresses. Segregation of the alloying elements leads to local differences between phase fractions and lattice misfit of the γ and γ' phases, resulting in inhomogeneous coherency stresses across the dendrite structure, ultimately affecting macroscopic dendritic stresses. With [001] as the direction of the primary dendrite arms (and the aging temperature gradient), the microstructures observed could be explained by tensile stresses along the secondary dendrite arm axes ([100] and [010]) in the W-rich primary and secondary dendrite core regions combined with compressive stresses towards the primary dendrite arms (mainly along the [110] direction) in the W-poor interdendrite regions. This would result in directional coarsening of γ' in the (100) and (010) planes perpendicular to the tensile stress axis in the dendrite cores as opposed to directional coarsening in the (001) plane parallel to the compressive stress axis in the interdendrite region. Since samples were sectioned in the (100) plane in this work, elongation along the [001]

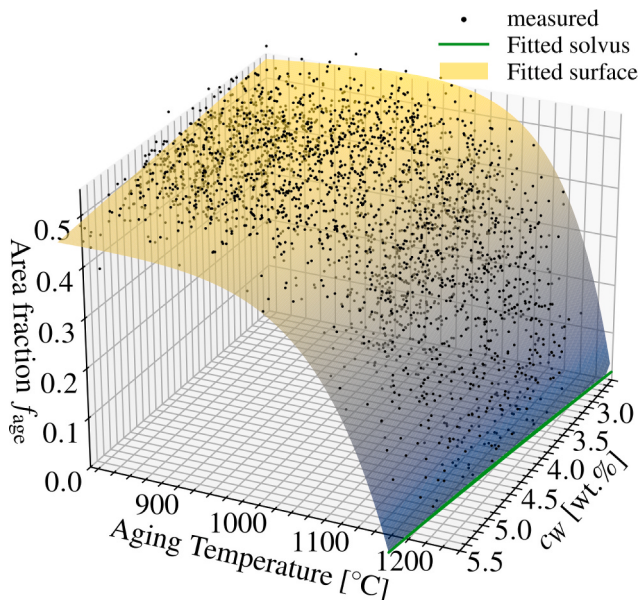


Fig. 10. Logistic surface fit to the area fractions of aging γ' according to Equation 5, using the solvus temperature (shown as a green line) determined in Equation 4. Area fractions determined by SEM image analysis are shown as black dots. Coloring of the surface is consistent with the scale used in Fig. 9b. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

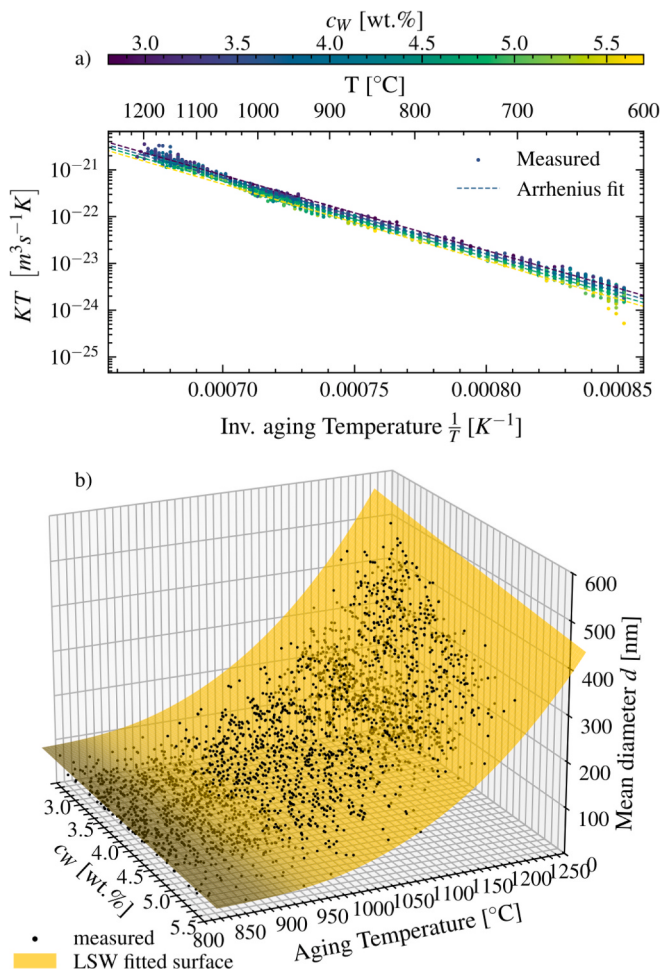


Fig. 11. Coarsening kinetic fitting results using the area-equivalent mean apparent aging γ' precipitate diameter determined in Fig. 7a, assuming a cubic LSW coarsening kinetic. K was calculated according to Equation 9. a) Measured values and linear regression for five different concentrations equally spaced on the color scale. b) Measured diameter compared to the surface calculated from the fitted coarsening rate constant using Equation 9.

axis (aspect ratio $\frac{a}{b} > 1$) was observed for the W-rich dendrite core regions while the W-poor interdendrite regions showed elongation in the $[010]$ orientation (aspect ratio $\frac{a}{b} < 1$). This is consistent with a recent work by Kou et al. using synchrotron x-ray diffraction and FEA to quantify dendritic stresses [114], while other works report completely different stress distributions [115,116]. It is likely that the differing processing and/or alloy compositions have a significant influence. However, further detailed analysis is required to identify the mechanism by which the position within the dendrite structure influences the aspect ratio of γ' precipitates. As a future work, we intend to evaluate residual stress using nanoindentation and EBSD analysis integrated into the developed high-throughput system [91].

5. Conclusion

A comprehensive Composition-Process-Structure (C-P-S) dataset was generated using just one single-crystal Ni-base superalloy sample. This high-throughput approach involved mapping a whole range of compositions and processing conditions onto the sample by utilizing the composition gradients caused by dendritic segregation and applying a temperature gradient aging treatment. Using automatic image analysis and correlative SEM/EPMA analysis, we were able to gather information on γ' phase fractions, coarsening kinetics, γ/γ' coherency stresses and

residual macroscopic dendritic stresses. Each point will be addressed in detail below:

1. Phase diagram data can be directly visualized by reordering SEM microstructure images according to composition as measured by EPMA-WDS. Image analysis further allowed quantification of phase fractions and precise determination of the γ' solvus temperature.
2. Coarsening kinetics were found to be mostly unaffected by composition differences induced by dendritic segregation. The activation energy for coarsening was found to only slightly increase with W concentration. Standard LSW kinetics fit experimental observations well, with no immediately apparent effect of volume fractions.
3. Coherency stress at the γ/γ' interfaces resulted in cuboidal γ' morphologies near 1150 °C, consistent with temperature dependence of the negative γ/γ' lattice misfit. Coinciding with significant alignment and clustering effects, a clear departure from regularly spaced precipitates was visible, invalidating key assumptions for many precipitate strengthening models.
4. Macroscopic dendritic stresses caused by local variations of coherency stress were found to visibly affect the aspect ratio of γ' precipitates, with implied tensile stresses along secondary dendrite arm axes and compressive stresses in the interdendrite region towards the primary dendrite arm axes.

Exploiting the naturally occurring dendritic segregation shows promise for the fine-tuning of complex alloy compositions. As a much simpler alternative to the typically used diffusion couple technique, the combination with a temperature-graded aging treatment allows for especially efficient creation of C-P-S datasets. These highly resolved datasets could serve as a valuable source of observational data for data assimilation methods to enhance computational models. In particular, they are a potential aid for parameter selection in phase-field simulations of complex alloys, such as elemental mobilities, phase lattice misfit and equilibrium phase fractions.

CRediT authorship contribution statement

Thomas Hoefler: Writing – original draft, Visualization, Software, Methodology, Investigation, Formal analysis. **Ayako Ikeda:** Validation, Methodology, Investigation, Data curation. **Satoshi Utada:** Writing – review & editing, Formal analysis. **Makoto Osawa:** Writing – review & editing, Formal analysis. **Kyoko Kawagishi:** Resources. **Toru Hara:** Resources, Methodology, Conceptualization. **Takahito Ohmura:** Writing – review & editing, Supervision, Project administration, Funding acquisition, Conceptualization. **Daichi Akama:** Resources, Investigation. **Masaki Taneike:** Resources, Investigation. **Toshio Osada:** Writing – review & editing, Supervision, Project administration, Methodology, Conceptualization.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Financial support was provided by Acquisition Technology and Logistics Agency and JSPS Kakenhi. The authors have a patent pending to NIMS.

Acknowledgements

This work was supported by the Innovative Science and Technology Initiative for Security, ATLA, Japan (Grant No. JPJ004596) and JSPS KAKENHI (Grant Number JP24K08022).

The authors would like to extend their thanks to the following people at NIMS: Yuji Takata for casting and evaluation of the single crystal samples, Takuma Kohata for providing guidance on SEM observations, Eri Nakagawa for offering counsel on nanoindentation testing, Koji Nakazato and Yuki Hemmi (both Materials Forming Unit, MFU) for

advice regarding metallographic preparation, Masahiko Kawasaki (MFU) for quartz encapsulation, Yu Fujii and Katsuyuki Okada (both Surface and Bulk Analysis Unit), and Kayoko Nakakita, Megumi Noro, Chieko Toku and Nana Fujibayashi for assisting sample preparation.

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.matdes.2026.116820>.

Data availability

Data will be made available on request.

References

- [1] M. Cohen, Unknownables in the essence of materials science and engineering, *Mater. Sci. Eng.* 25 (1976) 3–4, [https://doi.org/10.1016/0025-5416\(76\)90043-4](https://doi.org/10.1016/0025-5416(76)90043-4).
- [2] G.B. Olson, Computational Design of Hierarchically Structured Materials, *Science* 277 (1997) 1237–1242, <https://doi.org/10.1126/science.277.5330.1237>.
- [3] W. Xiong, G.B. Olson, Cybmaterials: materials by design and accelerated insertion of materials, *npj Comput. Mater.* 2 (2016) 15009, <https://doi.org/10.1038/npjcompumats.2015.9>.
- [4] G.B. Olson, C.J. Kuehmann, Materials genomics: from CALPHAD to flight, *Scr. Mater.* 70 (2014) 25–30, <https://doi.org/10.1016/j.scriptamat.2013.08.032>.
- [5] G.B. Olson, Z.K. Liu, Genomic materials design: CALculation of PHase Dynamics, *Calphad* 82 (2023) 102590, <https://doi.org/10.1016/j.calphad.2023.102590>.
- [6] R.C. Reed, *The superalloys: fundamentals and applications*, Cambridge University Press, 2008.
- [7] A.B. Botelho Junior, J. Kim, Recycling of superalloys: circular strategies for critical mineral supply chain resilience, *Resour. Conserv. Recycl.* (2026) 108859, <https://doi.org/10.1016/j.resconrec.2026.108859>.
- [8] M.C. Hardy, M. Detrois, E.T. McDevitt, et al., Solving recent challenges for Wrought Ni-Base Superalloys, *Metall. Mater. Trans. A* 51 (2020) 2626–2650, <https://doi.org/10.1007/s11661-020-05773-6>.
- [9] R.C. Reed, T. Tao, N. Warnken, Alloys-By-Design: Application to nickel-based single crystal superalloys, *Acta Mater.* 57 (2009) 5898–5913, <https://doi.org/10.1016/j.actamat.2009.08.018>.
- [10] Y.T. Tang, C. Panwisawas, J.N. Ghoussoub, Y. Gong, J.W.G. Clark, A.A. N. Németh, D.G. McCartney, R.C. Reed, Alloys-by-design: Application to new superalloys for additive manufacturing, *Acta Mater.* 202 (2021) 417–436, <https://doi.org/10.1016/j.actamat.2020.09.023>.
- [11] H. Harada, H. Murakami, Design of Ni-Base Superalloys, in: T. Saito (Ed.), *Computational Materials Design*, Springer, Berlin Heidelberg, Berlin, Heidelberg, 1999, pp. 39–70, https://doi.org/10.1007/978-3-662-03923-6_2.
- [12] J.J. de Pablo, N.E. Jackson, M.A. Webb, L.-Q. Chen, J.E. Moore, D. Morgan, R. Jacobs, T. Pollock, D.G. Schlom, E.S. Toberer, J. Analytis, I. Dabo, D. M. DeLongchamp, G.A. Fiete, G.M. Grason, G. Hautier, Y. Mo, K. Rajan, E.J. Reed, E. Rodriguez, V. Stevanovic, J. Suntivich, K. Thornton, J.-C. Zhao, New frontiers for the materials genome initiative, *npj Comput. Mater.* 5 (2019) 41, <https://doi.org/10.1038/s41524-019-0173-4>.
- [13] H. Lukas, S.G. Fries, B. Sundman, *Computational Thermodynamics: The Calphad Method*, 1st ed., Cambridge University Press, USA, 2007.
- [14] C.E. Campbell, U.R. Kattner, Z.-K. Liu, File and data repositories for Next Generation CALPHAD, *Scr. Mater.* 70 (2014) 7–11, <https://doi.org/10.1016/j.scriptamat.2013.06.013>.
- [15] Z.-K. Liu, First-Principles Calculations and CALPHAD Modeling of Thermodynamics, *J. Phase Equilibria Diffus.* 30 (2009) 517–534, <https://doi.org/10.1007/s11669-009-9570-6>.
- [16] N. Saunders, Z. Guo, X. Li, A.P. Miodownik, J.P. Schillé, Using JMatPro to model materials properties and behavior, *JOM* 55 (2003) 60–65, <https://doi.org/10.1007/S11837-003-0013-2>.
- [17] Z. Guo, N. Saunders, J.P. Schillé, A.P. Miodownik, Material properties for process simulation, *Mater. Sci. Eng. A* 499 (2009) 7–13, <https://doi.org/10.1016/J.MSEA.2007.09.097>.
- [18] N. Saunders, Phase diagram calculations for Ni-based superalloys, in: R.D. Kissinger et al (Ed.), *Superalloys 1996*, The Minerals, Metals & Materials Society, 1996.
- [19] N. Saunders, M. Fahrman, C.J. Small, The Application of Calphad Calculations to Ni-based Superalloys, in: *Superalloys 2000* (ninth International Symposium), 2000, pp. 803–811, https://doi.org/10.7449/2000/SUPERALLOYS_2000_803_811.
- [20] M. Enomoto, H. Harada, M. Yamazaki, Calculation of γ'/γ equilibrium phase compositions in nickel-base superalloys by cluster variation method, *Calphad* 15 (1991) 143–158, [https://doi.org/10.1016/0364-5916\(91\)90013-A](https://doi.org/10.1016/0364-5916(91)90013-A).
- [21] R. Kikuchi, A Theory of Cooperative Phenomena, *Phys. Rev.* 81 (1951) 988, <https://doi.org/10.1103/PhysRev.81.988>.
- [22] W.J. Boettinger, J.A. Warren, C. Beckermann, A. Karma, Phase-field simulation of solidification, *Annu. Rev. Mater. Sci.* 32 (2002) 163–194, <https://doi.org/10.1146/ANNUREV.MATSCL.32.101901.155803>.
- [23] W. Wang, P.D. Lee, M. McLean, A model of solidification microstructures in nickel-based superalloys: predicting primary dendrite spacing selection, *Acta Mater.* 51 (2003) 2971–2987, [https://doi.org/10.1016/S1359-6454\(03\)00110-1](https://doi.org/10.1016/S1359-6454(03)00110-1).
- [24] P. Carter, D.C. Cox, C.A. Gandin, R.C. Reed, Process modelling of grain selection during the solidification of single crystal superalloy castings, *Mater. Sci. Eng. A* 280 (2000) 233–246, [https://doi.org/10.1016/S0921-5093\(99\)00701-7](https://doi.org/10.1016/S0921-5093(99)00701-7).
- [25] C. Yang, Q. Xu, B. Liu, GPU-accelerated three-dimensional phase-field simulation of dendrite growth in a nickel-based superalloy, *Comput. Mater. Sci.* 136 (2017) 133–143, <https://doi.org/10.1016/J.COMMATSCI.2017.04.031>.
- [26] S.Y. Hu, L.Q. Chen, A phase-field model for evolving microstructures with strong elastic inhomogeneity, *Acta Mater.* 49 (2001) 1879–1890, [https://doi.org/10.1016/S1359-6454\(01\)00118-5](https://doi.org/10.1016/S1359-6454(01)00118-5).
- [27] J. Chen, M. Guo, M. Yang, H. Su, L. Liu, J. Zhang, Phase-field simulation of γ' coarsening behavior in cobalt-based superalloy, *Comput. Mater. Sci.* 191 (2021) 110358, <https://doi.org/10.1016/J.COMMATSCI.2021.110358>.
- [28] C. Wang, M. Umair, Y. Jiang, D.K. Nerella, M.A. Ali, I. Steinbach, Morphological evolution of γ' and γ'' precipitation in a model superalloy: Insights from 3D phase-field simulations, *Comput. Mater. Sci.* 256 (2025) 113972, <https://doi.org/10.1016/J.COMMATSCI.2025.113972>.
- [29] Y. Yang, Q. Ma, C. Pei, H. Yao, J. Bai, H. Li, Q. Gao, Microscopic phase-field simulation of ternary cobalt-based superalloys based on lattice inversion, *J. Alloys Compd.* 1031 (2025) 180958, <https://doi.org/10.1016/J.JALLCOM.2025.180958>.
- [30] S. Berbenni, R. Dingreville, T. Schenk, Simulation and analysis of small angle scattering (SAS) patterns of Ni-based superalloy microstructures generated by a phase-field model, *Comput. Mater. Sci.* 258 (2025) 114086, <https://doi.org/10.1016/J.COMMATSCI.2025.114086>.
- [31] J. Chen, M. Guo, M. Yang, J. Zhang, Temperature dependence of kinetics pathway of γ' precipitation in Co-Al-W superalloys: a phase-field study, *J. Alloys Compd.* 922 (2022) 166319, <https://doi.org/10.1016/J.JALLCOM.2022.166319>.
- [32] T. Kitashima, H. Harada, A new phase-field method for simulating γ' precipitation in multicomponent nickel-base superalloys, *Acta Mater.* 57 (2009) 2020–2028, <https://doi.org/10.1016/J.ACTAMAT.2009.01.006>.
- [33] T. Keller, G. Lindwall, S. Ghosh, L. Ma, B.M. Lane, F. Zhang, U.R. Kattner, E. A. Lass, J.C. Heigel, Y. Idell, M.E. Williams, A.J. Allen, J.E. Guyer, L.E. Levine, Application of finite element, phase-field, and CALPHAD-based methods to additive manufacturing of Ni-based superalloys, *Acta Mater.* 139 (2017) 244–253, <https://doi.org/10.1016/J.ACTAMAT.2017.05.003>.
- [34] J. Kundin, L. Mushongera, T. Goehler, H. Emmerich, Phase-field modeling of the γ' -coarsening behavior in Ni-based superalloys, *Acta Mater.* 60 (2012) 3758–3772, <https://doi.org/10.1016/J.ACTAMAT.2012.03.023>.
- [35] R. Laskowski, K. Wang, R. Ahluwalia, K. Bai, G. Vastola, Y.W. Zhang, Phase field model for multiphase alloys under arbitrary thermal history: an application to IN718 super-alloy, *J. Alloys Compd.* 861 (2021) 158630, <https://doi.org/10.1016/J.JALLCOM.2021.158630>.
- [36] Z. Wang, C. Liang, X. Ding, D. Wang, New insights into the partitioning behavior of Mo in nickel-based superalloys and its effect on microstructure using CALPHAD-assisted phase field modeling, *Acta Mater.* 282 (2025) 120510, <https://doi.org/10.1016/J.ACTAMAT.2024.120510>.
- [37] X. Wu, T. Cheng, J. Zhong, S. Yang, S. Ma, N. Ta, L. Zhang, An efficient microstructure simulation framework by integrating phase-field model, general coupling schema and parallelism: Demo in Ni-based superalloys, *Calphad* 88 (2025) 102798, <https://doi.org/10.1016/J.CALPHAD.2025.102798>.
- [38] W. Wu, U.R. Kattner, C.E. Campbell, J.E. Guyer, P.W. Voorhees, J.A. Warren, O. G. Heinonen, Co-based superalloy morphology evolution: a phase field study based on experimental thermodynamic and kinetic data, *Acta Mater.* 233 (2022) 117978, <https://doi.org/10.1016/J.ACTAMAT.2022.117978>.
- [39] A. Riyahi khorasgani, M. Younan, I. Steinbach, J. Kundin, Phase-Field Modeling of Kinetics of Diffusive Phase Transformation in Compositionally-Graded Ni-Based Superalloys, *J. Phase Equilibria Diffus.* 45 (2024) 1055–1067, <https://doi.org/10.1007/s11669-024-01140-9>.
- [40] Z. Wang, C. Liang, D. Wang, X. Ding, Uncovering the effect of Mo addition on the precipitation kinetics of the γ' phase in Ni-Al-Mo model superalloys using CALPHAD-assisted phase-field simulations, *J. Mater. Sci.* 59 (2024) 7893–7912, <https://doi.org/10.1007/s10853-024-09626-0>.
- [41] J.C. Wang, M. Osawa, T. Yokokawa, H. Harada, M. Enomoto, Modeling the microstructural evolution of Ni-base superalloys by phase field method combined with CALPHAD and CVM, *Comput. Mater. Sci.* 39 (2007) 871–879, <https://doi.org/10.1016/J.COMMATSCI.2006.10.014>.
- [42] T. Osada, T. Koyama, D.S. Bulgarevich, S. Minamoto, M. Osawa, M. Watanabe, K. Kawagishi, M. Demura, Virtual heat treatment for $\gamma-\gamma'$ two-phase Ni-Al alloy on the materials Integration system, *Mint. Mater. Des.* 226 (2023) 111631, <https://doi.org/10.1016/j.matdes.2023.111631>.
- [43] L. Wu, T. Osada, T. Yokokawa, Y. Chang, K. Kawagishi, The temperature dependence of strengthening mechanisms in Ni-based superalloys: a newly re-defined cuboidal model and its implications for strength design, *J. Alloys Compd.* 931 (2023) 167508, <https://doi.org/10.1016/j.jallcom.2022.167508>.
- [44] Y. Li, B. Holmedal, H. Li, L. Zhuang, J. Zhang, Q. Du, Precipitation and strengthening modeling for disk-shaped particles in aluminum alloys: size distribution considered, *Materialia* (oxf). 4 (2018) 431–443, <https://doi.org/10.1016/J.MTLA.2018.11.001>.
- [45] S. Keshavarz, S. Ghosh, Multi-scale crystal plasticity finite element model approach to modeling nickel-based superalloys, *Acta Mater.* 61 (2013) 6549–6561, <https://doi.org/10.1016/J.ACTAMAT.2013.07.038>.

- [46] S. Keshavarz, S. Ghosh, A.C.E. Reid, S.A. Langer, A non-Schmid crystal plasticity finite element approach to multi-scale modeling of nickel-based superalloys, *Acta Mater.* 114 (2016) 106–115, <https://doi.org/10.1016/j.actamat.2016.05.016>.
- [47] M. Knezevic, J.S. Carpenter, M.L. Lovato, R.J. McCabe, Deformation behavior of the cobalt-based superalloy Haynes 25: Experimental characterization and crystal plasticity modeling, *Acta Mater.* 63 (2014) 162–168, <https://doi.org/10.1016/j.actamat.2013.10.021>.
- [48] Y.Z. Liu, Z.L. Shi, Y.B. Zhang, M. Qin, S.P. Hu, X.G. Song, W. Fu, B.J. Lee, Effect of temperature on the mechanical properties of Ni-based superalloys via molecular dynamics and crystal plasticity, *J. Mater. Sci. Technol.* 203 (2024) 126–142, <https://doi.org/10.1016/j.jmst.2024.02.085>.
- [49] K.Y. Zhu, S. Dai, S.H. Zou, Y.J. Yu, Z.C. Deng, Experimental study and crystal plasticity modeling of additive manufacturing IN718 superalloy considering negative strain rate sensitivity behavior, *Eur. J. Mech. A. Solids* 106 (2024) 105304, <https://doi.org/10.1016/j.euromechsol.2024.105304>.
- [50] M. Toursangsarak, D. Du, H. Wang, A. Dong, Crystal plasticity quantification of anisotropic tensile and fatigue properties in laser powder bed fused Inconel 718 superalloy, *Addit. Manuf.* 89 (2024) 104300, <https://doi.org/10.1016/j.addma.2024.104300>.
- [51] C. Luo, X. Yang, H. Weng, H. Yuan, Degradation mechanism of nickel-base single crystal superalloy after long-term aging: Roles of nanomechanical properties and morphology of γ/γ' phases, *J. Mater. Sci. Technol.* 248 (2026) 247–265, <https://doi.org/10.1016/j.jmst.2025.04.086>.
- [52] J.Y. Wang, Y.C. Zhu, W. Bin Chen, R.D. Liu, X.K. Li, Y.W. Wang, Q.C. Zhu, J. P. Liang, C.W. Li, L. Jiang, Z.J. Li, Realizing strength-ductility combination of Ni-12Mo-7Cr-2Nb based superalloy via nano-sized MC carbides at stacking faults and grain boundaries, *Mater. Sci. Eng. A* 916 (2024) 147303, <https://doi.org/10.1016/j.msea.2024.147303>.
- [53] K.S. Li, R.Z. Wang, G.J. Yuan, S.P. Zhu, X.C. Zhang, S.T. Tu, H. Miura, A crystal plasticity-based approach for creep-fatigue life prediction and damage evaluation in a nickel-based superalloy, *Int. J. Fatigue* 143 (2021) 106031, <https://doi.org/10.1016/j.ijfatigue.2020.106031>.
- [54] G.J. Yuan, X.C. Zhang, B. Chen, S.T. Tu, C.C. Zhang, Low-cycle fatigue life prediction of a polycrystalline nickel-base superalloy using crystal plasticity modelling approach, *J. Mater. Sci. Technol.* 38 (2020) 28–38, <https://doi.org/10.1016/j.jmst.2019.05.072>.
- [55] B. Lin, L.G. Zhao, J. Tong, A crystal plasticity study of cyclic constitutive behaviour, crack-tip deformation and crack-growth path for a polycrystalline nickel-based superalloy, *Eng. Fract. Mech.* 78 (2011) 2174–2192, <https://doi.org/10.1016/j.engfractmech.2011.04.006>.
- [56] R. Bandyopadhyay, M.D. Sangid, Crystal plasticity assessment of inclusion- and matrix-driven competing failure modes in a nickel-base superalloy, *Acta Mater.* 177 (2019) 20–34, <https://doi.org/10.1016/j.actamat.2019.07.024>.
- [57] J.C. He, S.P. Zhu, J.W. Gao, R. Liu, W. Li, Q. Liu, Y. He, Q. Wang, Microstructural size effect on the notch fatigue behavior of a Ni-based superalloy using crystal plasticity modelling approach, *Int. J. Plast.* 172 (2024) 103857, <https://doi.org/10.1016/j.jiplas.2023.103857>.
- [58] S. Chaudhary, B. Sudhakar, N. Pai, M. Palit, Z. Alam, R. Sankarasubramanian, I. Samajdar, A. Patra, A crystal plasticity-based micromechanical model for precipitate shearing: Application to cyclic softening of polycrystalline Ni-based superalloys, *Int. J. Fatigue* 190 (2025) 108582, <https://doi.org/10.1016/j.jfatigue.2024.108582>.
- [59] D. Wang, Y. Li, S. Shi, X. Tong, Z. Yan, Phase-field simulation of γ' precipitates rafting and creep property of Co-base superalloys, *Mater. Des.* 196 (2020), <https://doi.org/10.1016/j.matdes.2020.109077>.
- [60] H. Men, D. Wang, Y. Li, S. Shi, S. Chen, Z. Liu, Crystal plasticity phase-field simulation of rafting and creep properties with a continuous change of the mismatch strain in a Co-Al-W superalloy, *Mater. Lett.* 306 (2022) 130868, <https://doi.org/10.1016/j.matlet.2021.130868>.
- [61] S. Shi, K. Niu, S. Wang, Z. Zhang, P. Sang, Y. Li, Creep rafting fracture and strain properties of overheating Co-based superalloy: Crystal plasticity phase-field simulation, *Comput. Mater. Sci.* 246 (2025) 113453, <https://doi.org/10.1016/j.commatsci.2024.113453>.
- [62] P.E. McHugh, R. Mohrmann, Modelling of creep in a Ni base superalloy using a single crystal plasticity model, *Comput. Mater. Sci.* 9 (1997) 134–140, [https://doi.org/10.1016/S0927-0256\(97\)00067-0](https://doi.org/10.1016/S0927-0256(97)00067-0).
- [63] P. Wang, Z. Wen, M. Li, G. Lu, H. Cheng, P. He, Z. Yue, Modified crystal plasticity constitutive model considering tensorial properties of microstructural evolution and creep life prediction model for Ni-based single crystal superalloy with film cooling hole, *Int. J. Plast.* 183 (2024) 104150, <https://doi.org/10.1016/j.jiplas.2024.104150>.
- [64] H. Harada, K. Ohno, T. Yamagata, T. Yokokawa, M. Yamazaki, Phase Calculation and its use In Alloy Design Program for Nickel-base Superalloys, *Superalloys 1988* (1988) 733–742.
- [65] T. Yokokawa, H. Harada, K. Kawagishi, T. Kobayashi, M. Yuyama, Y. Takata, Advanced Alloy Design Program and Improvement of Sixth-Generation Ni-Base Single Crystal Superalloy TMS-238, in: S. Tin, M. Hardy, J. Clews, J. Cormier, Q. Feng, J. Marcin, C. O'Brien, A. Suzuki (Eds.), *Superalloys 2020*, Springer International Publishing, Cham, 2020, pp. 122–130.
- [66] C.E. Campbell, U.R. Kattner, Z.-K. Liu, The development of phase-based property data using the CALPHAD method and infrastructure needs, *Integr. Mater. Manuf. Innov.* 3 (2014) 158–180, <https://doi.org/10.1186/2193-9772-3-12>.
- [67] Z.-K. Liu, Thermodynamics and its prediction and CALPHAD modeling: Review, state of the art, and perspectives, *Calphad* 82 (2023) 102580, <https://doi.org/10.1016/j.calphad.2023.102580>.
- [68] K. Choudhary, B. DeCost, C. Chen, A. Jain, F. Tavazza, R. Cohn, C.W. Park, A. Choudhary, A. Agrawal, S.J.L. Billinge, E. Holm, S.P. Ong, C. Wolverton, Recent advances and applications of deep learning methods in materials science, *NPJ Comput. Mater.* 2022 8:1 8 (2022) 1–26, <https://doi.org/10.1038/s41524-022-00734-6>.
- [69] R.K. Vasudevan, K. Choudhary, A. Mehta, R. Smith, G. Kusne, F. Tavazza, L. Vlcek, M. Ziatdinov, S.V. Kalinin, J. Hattrick-Simpers, Materials science in the artificial intelligence age: high-throughput library generation, machine learning, and a pathway from correlations to the underpinning physics, *MRS Commun.* 9 (2019) 821–838, <https://doi.org/10.1557/MRC.2019.95>.
- [70] W. Sha, Y. Guo, Q. Yuan, S. Tang, X. Zhang, S. Lu, X. Guo, Y.-C. Cao, S. Cheng, Artificial Intelligence to Power the Future of Materials Science and Engineering, *Adv. Intell. Syst.* 2 (2020) 1900143, <https://doi.org/10.1002/AISY.201900143>.
- [71] Y. Liu, Z. Yang, Z. Yu, Z. Liu, D. Liu, H. Lin, M. Li, S. Ma, M. Avdeev, S. Shi, Generative artificial intelligence and its applications in materials science: current situation and future perspectives, *J. Materomics* 9 (2023) 798–816, <https://doi.org/10.1016/j.jmat.2023.05.001>.
- [72] J. Schmidt, T.F.T. Cerqueira, A.H. Romero, A. Loew, F. Jäger, H.C. Wang, S. Botti, M.A.L. Marques, Improving machine-learning models in materials science through large datasets, *Mater. Today Phys.* 48 (2024) 101560, <https://doi.org/10.1016/j.mtphys.2024.101560>.
- [73] Y. Li, J. Pang, Z. Li, Q. Wang, Z. Wang, J. Li, H. Zhang, Z. Jiao, C. Dong, P.K. Liaw, Developing novel low-density high-entropy superalloys with high strength and superior creep resistance guided by automated machine learning, *Acta Mater.* 285 (2025) 120656, <https://doi.org/10.1016/j.actamat.2024.120656>.
- [74] W. Wang, X. Jiang, W. Li, C. Zhang, P. Liu, S. Tian, T. Lookman, Y. Su, Design of superalloys with multiple properties via multi-task learning, *Acta Mater.* 294 (2025) 121161, <https://doi.org/10.1016/j.actamat.2025.121161>.
- [75] W. Liao, R. Yuan, X. Xue, J. Wang, J. Li, T. Lookman, Unsupervised learning-aided extrapolation for accelerated design of superalloys, *NPJ Comput. Mater.* 2024 10: 1 10 (2024) 1–8, <https://doi.org/10.1038/s41524-024-01358-8>.
- [76] A.A. Kodentsov, G.F. Bastin, F.J.J. van Loo, The diffusion couple technique in phase diagram determination, *J. Alloys Compd.* 320 (2001) 207–217, [https://doi.org/10.1016/S0925-8388\(00\)01487-0](https://doi.org/10.1016/S0925-8388(00)01487-0).
- [77] W. Lengauer, The temperature gradient diffusion couple technique: an application of solid-solid phase reactions for phase diagram imaging, *J. Solid State Chem.* 91 (1991) 279–285, [https://doi.org/10.1016/0022-4596\(91\)90082-S](https://doi.org/10.1016/0022-4596(91)90082-S).
- [78] Z. Qin, Z. Wang, Y. Wang, L. Zhang, W. Li, J. Liu, Z. Wang, Z. Li, J. Pan, L. Zhao, F. Liu, L. Tan, J. Wang, H. Han, L. Jiang, Y. Liu, Phase prediction of Ni-base superalloys via high-throughput experiments and machine learning, *Mater. Res. Lett.* 9 (2021) 32–40, <https://doi.org/10.1080/21663831.2020.1815093>.
- [79] Z. Wang, L. Zhang, W. Li, Z. Qin, Z. Wang, Z. Li, L. Tan, L. Zhu, F. Liu, H. Han, L. Jiang, A high-throughput approach to explore the multi-component alloy space: a case study of nickel-based superalloys, *J. Alloys Compd.* 858 (2021) 158100, <https://doi.org/10.1016/j.jallcom.2020.158100>.
- [80] W. Li, L. Li, S. Antonov, C. Wei, J.-C. Zhao, Q. Feng, High-throughput exploration of alloying effects on the microstructural stability and properties of multi-component CoNi-base superalloys, *J. Alloys Compd.* 881 (2021) 160618, <https://doi.org/10.1016/j.jallcom.2021.160618>.
- [81] W. Li, J.M. Wheeler, H. Han, L. Li, Q. Feng, High-throughput investigation of the alloying effects of W, Ti, and Ta on the microstructural stability and mechanical properties of CoNi-base superalloys, *Mater. Sci. Eng. A* 925 (2025) 147857, <https://doi.org/10.1016/j.msea.2025.147857>.
- [82] K. Goto, A. Ikeda, T. Osada, I. Watanabe, K. Kawagishi, T. Ohmura, High-throughput evaluation of stress-strain relationships in Ni-Co-Cr ternary systems via indentation testing of diffusion couples, *J. Alloys Compd.* 910 (2022) 164868, <https://doi.org/10.1016/j.jallcom.2022.164868>.
- [83] M. Asano, T. Osada, A. Ikeda, T. Abe, T. Hoefler, E. Nakagawa, T. Ohmura, High-throughput evaluation of hardness dependence on composition and temperature in Ni-Co binary alloy systems, *J. Alloys Compd.* 1042 (2025) 183868, <https://doi.org/10.1016/j.jallcom.2025.183868>.
- [84] A. Ikeda, K. Goto, T. Osada, I. Watanabe, K. Kawagishi, High-throughput mapping method for mechanical properties, oxidation resistance, and phase stability in Ni-based superalloys using composition-graded unidirectional solidified alloys, *Scr. Mater.* 193 (2021) 91–96, <https://doi.org/10.1016/j.scriptamat.2020.10.043>.
- [85] L. Montanelli, V. Venugopal, E.A. Olivetti, M.I. Latypov, High-Throughput Extraction of Phase-Property Relationships from Literature using Natural Language Processing and Large Language Models, *Integr. Mater. Manuf. Innov.* 13 (2024) 396–405, <https://doi.org/10.1007/s40192-024-00344-8>.
- [86] M.J. Buehler, MechGPT, a Language-based strategy for Mechanics and Materials Modeling that Connects Knowledge across Scales, Disciplines, and Modalities, *Appl. Mech. Rev.* 76 (2024) 21001, <https://doi.org/10.1115/1.4063843>.
- [87] A.R. Durmaz, A. Thomas, L. Mishra, R.N. Murthy, T. Straub, An ontology-based text mining dataset for extraction of process-structure-property entities, *Sci. Data* 11 (2024) 1112, <https://doi.org/10.1038/s41597-024-03926-5>.
- [88] M. Hart, K. Idanwekhai, V.M. Alves, A.J.M. Miller, J.L. Dempsey, J.F. Cahoon, C. H. Chen, D.A. Winkler, E.N. Muratov, A. Tropsha, Trust not Verify? the critical need for Data Curation Standards in Materials Informatics, *Chem. Mater.* 36 (2024) 9046–9055, https://doi.org/10.1021/ACS.CHEMMATER.4C00981/ASSET/IMAGES/LARGE/CM4C00981_0002.JPEG.
- [89] A. Orozco-Caballero, C. Gutierrez, B. Gan, J.M. Molina-Aldareguia, High-throughput nanoindentation mapping of cast IN718 nickel-based superalloys: influence of the Nb concentration, *J. Mater. Res.* 36 (2021) 2213–2222, <https://doi.org/10.1557/s43578-021-00133-5>.

- [90] D.G. Kalali, H. Seekala, P.S. Phani, K. Bhanu Sankara Rao, K. V. Rajulapati, High speed nanoindentation aided correlative study between local mechanical properties and chemical segregation in equiatomic MoNb and MoNbTi alloys, *J. Mater. Res.* 38 (2023) 2919–2929, <https://doi.org/10.1557/s43578-023-01007-8>.
- [91] T. Hoefler, A. Ikeda, T. Osada, T. Hara, K. Kawagishi, T. Ohmura, Automated system for high-throughput process-structure-property dataset generation of structural materials: a γ/γ' superalloy case study, *Mater. Des.* 256 (2025) 114279, <https://doi.org/10.1016/J.MATDES.2025.114279>.
- [92] I. Okada, T. Torigoe, K. Takahashi, D. Izutsu, DEVELOPMENT OF Ni BASE SUPERALLOY FOR INDUSTRIAL GAS TURBINE, in: K.A. Green, T.M. Pollock, H. Harada, T.E. Howson, R.C. Reed, J.J. Schirra, S. Walston (Eds.), *Superalloys 2004*, The Minerals, Metals & Materials Society, n.d.
- [93] R. Yurishima, Y. Takagiwa, A. Ikeda, T. Ikeda, Microstructure Optimization of Thermoelectric τ_1 -Al₂Fe₃Si₃ via Graded Temperature Heat Treatments, *Materials* 17 (2024), <https://doi.org/10.3390/ma17235899>.
- [94] G. Bradski, The OpenCV Library, Dr. Dobb's Journal of Software Tools (2000).
- [95] S. van der Walt, J.L. Schönberger, J. Nunez-Iglesias, F. Boulogne, J.D. Warner, N. Yager, E. Gouillart, T. Yu, the scikit-image contributors, scikit-image: image processing in Python, *PeerJ* 2 (2014) e453.
- [96] E.W. Weisstein, Superellipse, (2024). <https://mathworld.wolfram.com/Superellipse.html>.
- [97] A.J. Ardell, Precipitation hardening, *Metall. Trans. A* 16 (1985) 2131–2165, <https://doi.org/10.1007/BF02670416>.
- [98] E.I. Galindo-Nava, L.D. Connor, C.M.F. Rae, On the prediction of the yield stress of unimodal and multimodal γ' Nickel-base superalloys, *Acta Mater.* 98 (2015) 377–390, <https://doi.org/10.1016/J.ACTAMAT.2015.07.048>.
- [99] R.W. Kozar, A. Suzuki, W.W. Milligan, J.J. Schirra, M.F. Savage, T.M. Pollock, Strengthening Mechanisms in Polycrystalline Multimodal Nickel-Base Superalloys, *Metall. Mater. Trans. A* 40 (2009) 1588–1603, <https://doi.org/10.1007/s11661-009-9858-5>.
- [100] S.S. Babu, M.K. Miller, J.M. Vitek, S.A. David, Characterization of the microstructure evolution in a nickel base superalloy during continuous cooling conditions, *Acta Mater.* 49 (2001) 4149–4160, [https://doi.org/10.1016/S1359-6454\(01\)00314-7](https://doi.org/10.1016/S1359-6454(01)00314-7).
- [101] P. Li, L. Chen, H. Bu, Y. Zeng, S. Li, C. Wang, Effect of cooling rate on the Morphological changes in the secondary γ' precipitation in FGH97 nickel-based PM superalloy, *Intermetallics (barking)*. 171 (2024) 108344, <https://doi.org/10.1016/j.intermet.2024.108344>.
- [102] W. Yang, H. Zong, C. Yang, P. Qu, H. Su, Y. Dong, J. Zhang, L. Liu, The effect of Fe addition on γ' precipitates during thermal exposure in Ni-Co-based single crystal superalloys, *J. Mater. Res. Technol.* 20 (2022) 894–904, <https://doi.org/10.1016/j.jmrt.2022.07.135>.
- [103] R. Radis, M. Schaffer, M. Albu, G. Kothleitner, P. Pöhl, E. Kozeschnik, Multimodal size distributions of γ' precipitates during continuous cooling of UDIMET 720 Li, *Acta Mater.* 57 (2009) 5739–5747, <https://doi.org/10.1016/j.actamat.2009.08.002>.
- [104] I.M. Lifshitz, V.V. Slyozov, The kinetics of precipitation from supersaturated solid solutions, *J. Phys. Chem. Solid* 19 (1961) 35–50, [https://doi.org/10.1016/0022-3697\(61\)90054-3](https://doi.org/10.1016/0022-3697(61)90054-3).
- [105] C. Wagner, The growth of dispersed precipitates in solution, *Zeitschrift Für Electrochemie* 65 (1961) 581–597.
- [106] A.J. Ardell, The effect of volume fraction on particle coarsening: theoretical considerations, *Acta Metall.* 20 (1972) 61–71, [https://doi.org/10.1016/0001-6160\(72\)90114-9](https://doi.org/10.1016/0001-6160(72)90114-9).
- [107] D.J. Chellman, A.J. Ardell, The coarsening of γ' precipitates at large volume fractions, *Acta Metall.* 22 (1974) 577–588, [https://doi.org/10.1016/0001-6160\(74\)90155-2](https://doi.org/10.1016/0001-6160(74)90155-2).
- [108] C.K.L. Davies, P. Nash, R.N. Stevens, The effect of volume fraction of precipitate on oswald ripening, *Acta Metall.* 28 (1980) 179–189, [https://doi.org/10.1016/0001-6160\(80\)90067-X](https://doi.org/10.1016/0001-6160(80)90067-X).
- [109] H.A. Calderon, P.W. Voorhees, J.L. Murray, G. Kosterz, Ostwald ripening in concentrated alloys, *Acta Metall. Mater.* 42 (1994) 991–1000, [https://doi.org/10.1016/0956-7151\(94\)90293-3](https://doi.org/10.1016/0956-7151(94)90293-3).
- [110] A. Baldan, Review Progress in Ostwald ripening theories and their applications to nickel-base superalloys Part I: Ostwald ripening theories, *J. Mater. Sci.* 37 (2002) 2171–2202, <https://doi.org/10.1023/A:1015388912729>.
- [111] A. Baldan, Review Progress in Ostwald ripening theories and their applications to the γ' -precipitates in nickel-base superalloys Part II Nickel-base superalloys, *J. Mater. Sci.* 37 (2002) 2379–2405, <https://doi.org/10.1023/A:1015408116016>.
- [112] V. Vaithyanathan, L.Q. Chen, Coarsening of ordered intermetallic precipitates with coherency stress, *Acta Mater.* 50 (2002) 4061–4073, [https://doi.org/10.1016/S1359-6454\(02\)00204-5](https://doi.org/10.1016/S1359-6454(02)00204-5).
- [113] C.Z. Hargather, S.-L. Shang, Z.-K. Liu, A comprehensive first-principles study of solute elements in dilute Ni alloys: Diffusion coefficients and their implications to tailor creep rate, *Acta Mater.* 157 (2018) 126–141, <https://doi.org/10.1016/j.actamat.2018.07.020>.
- [114] J. Kou, K. Chen, S. Huang, C. Zhai, C.Y. Chiang, S. Wang, Z. Li, Y.D. Wang, Mapping stress heterogeneity in single-crystal superalloys by novel submicron-resolved X-ray diffraction, *Mater. Res. Lett.* 12 (2024) 450–458, <https://doi.org/10.1080/21663831.2024.2341932>.
- [115] A. Epishin, T. Link, U. Brückner, B. Fedelich, P. Portella, EFFECTS OF SEGREGATION IN NICKEL-BASE SUPERALLOYS: DENDRITIC STRESSES, 2004.
- [116] Y. Liu, Y. Li, Z. Yan, D. Yu, C. Liang, M. Xue, C. Geng, R. Su, S. Li, L. He, Y. dong Wang, Effects of dendritic segregation and local internal stress on microstructure evolution in a 3rd-generation single crystal superalloy during thermal exposure, *Mater. Des.* 257 (2025) 114507, <https://doi.org/10.1016/J.MATDES.2025.114507>.