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Assessment of the feasibility of determining the volume fraction and characteristics of the size, shape and distribution of γ' phase precipitates in nickel-based superalloys

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Abstract

This overview presents a comprehensive exploration of the research methods employed for the precise assessment of volume fraction and the detailed characterisation of the size, shape and distribution of γ' phase precipitates within Ni-based superalloys. These advanced materials exhibit exceptional mechanical properties due to the presence of γ' precipitates. The accurate quantification of precipitate parameters is crucial for understanding material behaviour and for the optimisation of alloy design. In this overview, a spectrum of techniques, including microscopy (SEM, TEM), diffraction (XRD), spectroscopy (EDS, EELS) and advanced imaging (3D-APT, STEM-HAADF, FIB-SEM) is discussed. Strengths, limitations and potential synergies among these methods are highlighted, offering researchers a comprehensive toolbox to advance their investigations of γ' phase precipitates in Ni-based superalloys.

Keywords: volume fraction, size, shape, distribution, γ' phase precipitates, Ni-based superalloys, research methods

1. Introduction

Nickel-based superalloys are a class of materials that exhibit exceptional mechanical strength, high temperature stability and resistance to corrosion, making them ideal for applications in aerospace, power generation and other high-temperature environments. Nickel-based superalloys have revolutionised various industries where critical components made of them operate under extreme conditions. Their unique combination of properties has had a transformative impact on efficiency, sustainability and technological progress across the globe. One crucial feature that contributes to their superior properties is the presence of precipitates, such as the gamma prime (γ') phase, within their microstructure. These precipitates are small, ordered particles that form during solidification and heat-treatment processes. The γ' phase, in particular, is known for its strengthening effect through the mechanism of precipitate hardening. The gamma prime phase is essential for nickel-based superalloys due to its ability to provide high strength, creep resistance, thermal stability and other desirable mechanical properties required for applications in extreme high-temperature conditions. The careful design and control of the formation of the gamma prime phase are critical in producing high-performance materials for advanced engineering applications.

The determination of the volume fraction and the characterisation of size and shape and the distribution of precipitates holds paramount significance in materials science and engineering, particularly in the realm of advanced alloys and composite materials. These precipitates, often referred to as strengthening or reinforcing phases, play a pivotal role in dictating the mechanical, thermal, and even chemical properties of materials. Accurate assessment and understanding of these microstructural features are essential for tailoring materials with desired performance attributes, enabling innovations in aerospace, automotive, energy and numerous other industries.

In this context, researchers and engineers employ a range of sophisticated methods and techniques to quantify the volume fraction and to characterise the morphological aspects of precipitates like phase γ' precipitates in Ni--based superalloys. These methods are designed to provide insights into the spatial distribution, size, shape, orientation and interfacial interactions of these precipitates within the host material (matrix). The knowledge gained through these analyses contributes to optimizing material processing routes, predicting mechanical behaviour and guiding the design of new materials with improved properties.

The methods for determining the volume fraction of phase γ' precipitates include both traditional and modern approaches. Traditional methods often involve labour-intensive manual measurements, such as point counting using microscopy or image-analysis techniques. These methods, while informative, may be limited by their time-consuming nature and potential human error. By contrast, modern methods harness the power of advanced imaging and computational tools. Techniques like electron microscopy (TEM), scanning electron microscopy (SEM), and synchrotron X-ray diffraction offer high--resolution imaging and diffraction capabilities that facilitate the automated analysis of large sample areas. Additionally, machine learning algorithms have been employed to enhance the accuracy and efficiency of phase quantification by training on large datasets of micrographs.

The characterisation of the size, shape and distribution of phase γ' precipitates is equally vital. The advent of three-dimensional imaging techniques, such as tomography and serial sectioning, has enabled researchers to obtain a comprehensive understanding of these microstructural features. By reconstructing three-dimensional volumes, researchers can assess the connectivity of precipitates, study their morphological evolution during processing and service, and make predictions about mechanical behaviour and long-term stability.

All of the methods considered in the work for the evaluation of volume fraction and the characterisation of the size, shape and distribution of precipitates in materials, especially phase γ' precipitates in Ni-based superalloys, represent critical steps in advancing materials science. The synergy between traditional methods and cutting-edge technologies has elevated our ability to comprehensively analyse microstructures and optimise material properties. As technological advancements continue to unfold, the field is poised to gain deeper insights into the role of precipitates in material behaviour, leading to innovations that will shape the future of various industries. In describing their importance for research, the author mentions their limitations and drawbacks.

2. Materials

This work is especially devoted to Ni-based superalloys. Nickel-based superalloys are a class of advanced materials primarily used in high-temperature applications such as gas turbine engines in aviation and power generation. These materials are crucial and irreplaceable in various industries due to their exceptional combination of mechanical strength, thermal stability and corrosion resistance at elevated temperatures. They are vital due to the following reasons:

- high-temperature applications (one of the primary reasons for the importance of nickel-based superalloys is their ability to maintain their mechanical properties at extreme temperatures, often exceeding 1000°C);
- efficiency and performance (the use of nickel-based superalloys allows gas turbines to operate at higher temperatures, which in turn improves their efficiency – higher operating temperatures lead to increased thermodynamic efficiency, resulting in reduced fuel consumption and lower emissions);
- creep and fatigue resistance (superalloys are specifically designed to resist creep and fatigue, which are deformation mechanisms that occur at high temperatures and under cyclic loading – superalloys are able to withstand these conditions without experiencing rapid degradation or failure over the long term);
- corrosion and oxidation resistance (in high-temperature and aggressive environments, such as those found in combustion gases and corrosive atmospheres, nickel-based superalloys exhibit remarkable resistance to corrosion and oxidation – the mentioned resistance stems from the protective oxide layers that forms on the alloy's surface, preventing further degradation and maintaining mechanical integrity);
- aerospace innovation (Ni-based superalloys have been a driving force behind advancements mainly in aerospace technology, but not limited to this);
- power generation (beyond aviation, nickel-based superalloys play a pivotal role in power generation industries);
- medical and industrial applications (Ni-based superalloys find applications in medical devices, such as orthopedic implants and dental prosthesis, due to their biocompatibility and corrosion resistance);
- material science and engineering (the research and development of nickel-based superalloys have pushed the boundaries of material science and engineering via innovations in alloy design, processing techniques and understanding of phase transformations).

The presence of the gamma prime (γ') phase plays a significant role in determining the unique mechanical and thermal properties of Ni-based superalloys. The precipitates of this phase are critical components in nickel-based superalloys due to the following reasons:

- strength and creep resistance (this phase is known for its excellent strength and stability at elevated temperatures due to the formation of coherent precipitates within the gamma (γ) matrix of the alloy);
- high-temperature stability (the gamma prime phase has a higher melting point than the surrounding gamma matrix, which helps maintain the structural integrity if the alloy and prevents premature failure at elevated temperatures);
- dislocation pinning (the coherent precipitates of the gamma prime phase act as barriers to dislocation movement within the crystal lattice and this effect contributes to the high strength and resistance of the material to plastic deformation at high temperatures);
- thermal expansion match (the thermal expansion coefficient of the gamma prime phase is often tailored to match that of the surrounding gamma matrix and in this way, it helps to reduce the generation of internal stresses during thermal cycling, which can lead to cracking and degradation of the material over time);
- phase stability (the stability of the gamma prime phase is influenced by its composition and processing conditions, which is why the controlled formation of the gamma prime phase through precise alloying and heat-treatment processes is required);
- fatigue resistance (the presence of the gamma prime phase enhances the fatigue resistance of nickel-based superalloys, which is crucial in applications subjected to cyclic loading, such as rotating turbine blades);
- oxidation resistance (the gamma prime phase can also contribute to improved oxidation resistance of the material at high temperatures due to the fact that it can act as a diffusion barrier, slowing down the diffusion of oxygen and other reactive species into the material) (Palmert, 2009).

Determining the volume fraction and characterising the size, shape and distribution of precipitates in materials, particularly phase γ' precipitates in superalloys, is crucial for multiple reasons:

- performance prediction (understanding the volume fraction of γ' precipitates is essential for predicting the mechanical, thermal and even chemical performance of superalloys – the presence and distribution of these precipitates significantly influence material properties, such as strength, hardness, creep resistance, fatigue life and thermal stability – accurate volume fraction measurements allow for more precise predictions of material behaviour in various operating conditions);
- material design and optimisation (the size, shape and distribution of precipitates play a vital role in tailoring material properties – by characterising these parameters, researchers and engineers can optimise alloy compositions, heat treatment processes and manufacturing techniques to achieve desired performance characteristics);
- microstructure-property relationships (by characterising the size, shape and distribution of γ' precipitates, researchers can establish quantitative relationships between microstructure and material behaviour, which enables more informed decision-making in material selection and design);
- failure prevention and design safety (inadequate or non-uniform distribution of precipitates can lead to localised deformation, stress concentrations and premature failure);
- process control and quality assurance (controlling the volume fraction and distribution of precipitates during manufacturing is critical for ensuring consistent material properties – by characterising precipitates, manufacturers can monitor and adjust processing parameters to maintain desired microstructural features and meet performance requirements);
- material certification (industries with stringent quality standards, such as aerospace and power generation, often require detailed material characterisation for certification);

- fundamental understanding (studying precipitates and their distribution contributes to the fundamental understanding of phase transformations, diffusion, nucleation and growth processes in materials – this knowledge advances the field of materials science);
- computational modelling validation (accurate characterisation data are essential for validating computational models that simulate material behaviour under various conditions).

To sum up, determining the volume fraction and characterising the size, shape and distribution of phase γ' precipitates is not only important but also highly beneficial for informed material design, performance prediction, failure prevention, process optimisation and advancing our understanding of materials at the microstructural level.

3. Research problem

What is the research problem with regard to the determination of the volume fraction and characterisation of size, shape and distribution of phase γ' precipitates in superalloys? The research problem arises due to the complexity of characterising these precipitates accurately (see Fig. 1). The main challenges include:

- volume fraction determination (precise determination of the volume fraction of γ' precipitates in the microstructure is essential for understanding the material's mechanical properties – accurately measuring the fraction of γ' phase in a sample requires advanced techniques that can differentiate between different phases and accurately quantify their presence);
- size and shape characterisation (the size and shape of precipitates significantly influence the properties of the given material – small changes in size and shape can have a profound impact on mechanical strength, creep resistance and other performance factors – characterizing these parameters necessitates advanced imaging techniques, such as transmission electron microscopy (TEM) or X-ray diffraction);
- distribution analysis (the spatial distribution of γ' precipitates within the alloy matrix can affect properties like fatigue resistance and thermal stability – quantifying the distribution, whether it's homogeneous, clustered, or dispersed, requires statistical analysis and advanced image processing methods);
- sample representation (achieving a representative sample for analysis is a challenge in itself – precipitates might not be uniformly distributed throughout the material, leading to potential sampling bias);
- influence of processing parameters (the formation of γ' precipitates is influenced by the processing conditions, such as heat treatment and cooling rates – understanding how these processing parameters impact the size, shape and distribution of the precipitates is essential for optimising material properties).

Solving this research problem involves a combination of experimental techniques, material characterisation tools and possibly computational simulations. There is a need to integrate various methods to accurately determine the volume fraction, characterise the size and shape of precipitates, analyse their distribution and establish correlations with material properties. This research is vital for advancing our understanding of superalloys and improving their performance for critical applications in demanding environments.

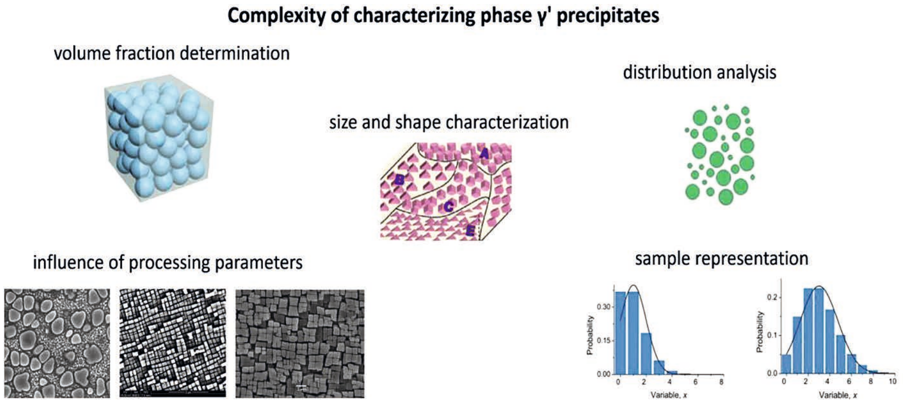

Fig. 1. The complexity of characterising phase γ' precipitates

4. Methods

There are several possible methods for determining the volume fraction and/or characterising the size, shape and distribution of phase γ' precipitates in superalloys (Kaufman, 2012; Leng, 2008; Sharma, 2018; De Graef, 2012).

Firstly, scanning electron microscopy (SEM) which enables high-resolution imaging of material surfaces at high magnification and can provide information about the morphology and the spatial distribution of the analysed precipitates, including even small phase γ' precipitates. Energy-dispersive X-ray spectroscopy (EDS) can be coupled with SEM to obtain elemental compositional information for matrix and precipitates (e.g. Smith, 2021).

Secondly, transmission electron microscopy (TEM) as a high-resolution imaging technique which provides detailed information about the size, shape and distribution of phase γ' precipitates along with nano-sized precipitates. Image analysis software can be used to quantify volume fractions and measure particle sizes (e.g. Rakoczy, 2020; Sun, 2021). In the case of superalloys, TEM is often employed to study the intricate microstructure of phase γ' precipitates. Techniques like selected area diffraction (SAD) can offer crystallographic information. High-resolution TEM (HRTEM) provides atomic-level resolution (Ghica, 2020).

STEM-HAADF stands for scanning transmission electron microscopy – high-angle annular dark-field imaging. It is a specialised imaging technique used in electron microscopy to obtain high-resolution images of materials at the nanoscale level. In STEM-HAADF imaging, a focused electron beam is scanned over the sample and the electrons that are scattered at high angles are collected and used to form an image. This technique provides information about the atomic arrangement and composition of a material. The high-angle scattering of electrons produces contrast in the image, with heavier elements appearing brighter and lighter elements appearing darker.

STEM-HAADF imaging is particularly useful for studying materials with variations in composition, such as the γ' phase precipitates in Ni-based superalloys. It enables researchers to visualise the distribution, size and shape of these precipitates at a very high spatial resolution, providing valuable insights into the microstructure and properties of the material.

Thirdly, X-ray diffraction (XRD) is a non-destructive technique used to identify crystalline phases present in a material. It is based on the fundamental principle of X-ray diffraction, which involves the interaction of X-rays with crystalline structures. When X-rays strike a crystalline material, they undergo scattering due to the arrangement of atoms in the crystal lattice. This scattering results in the generation of diffraction patterns, which provide information about the crystallographic orientation, lattice spacing and arrangement of atoms within the material (e.g. Chen, 2018). XRD can be used to quantitatively determine the phase composition and estimate the volume fraction of precipitates by analysing the diffraction pattern (based on the Rietveld refinement process –

Rundevski, 2021). By analysing the intensities and positions of diffraction peaks corresponding to the host matrix and the precipitates, one can quantify the relative proportions of the phases present. In the case of γ' phase precipitates in superalloys, XRD can offer information about their crystal structure and orientation. However, it may not provide detailed size and shape information about precipitates.

The next are small-angle scattering (SAS) techniques, such as small-angle X-ray scattering (SAXS) and small-angle neutron scattering (SANS), which provide insights into the size and spatial distribution of nano-sized particles. By analysing the scattering patterns, researchers can derive parameters such as the average size of the precipitates, their size distribution and their spatial clustering. In the example works (Haas, 2018; Wang, 2020) one can find these methods being reported for the study of phase γ' precipitates.

The further method is image analysis and stereology (IA+STER). Image analysis combined with stereology principles are used to quantify the volume fraction, size and shape of precipitates from two-dimensional micrographs (e.g. Escobar-Moreno, 2019). Image analysis involves the processing and quantification of images captured using microscopy or other imaging techniques. In the case of phase γ' precipitates in superalloys, the most commonly used approaches are techniques like scanning electron microscopy (SEM) and transmission electron microscopy (TEM) to capture images of the microstructure. Image analysis tools can then be employed to analyse these images. In the first step, image segmentation is applied. It is the process of identifying and separating different phases or features within an image. In the case of γ' precipitates, this could involve distinguishing between the γ matrix and the γ' precipitates. The next step is particle detection to detect individual γ' precipitates and provide information about their size, shape and spatial distribution. And finally a quantification is performed. Image analysis software measures parameters like particle size, aspect ratio (shape), orientation and distance between particles. These measurements help in characterising the microstructure. Image analysis and stereology techniques involve systematic sampling and mathematical modelling to extrapolate three-dimensional information from two-dimensional sections.

In recent years, 3D-imaging techniques have been of great interest in the field of research techniques. Advanced 3D-imaging techniques, such as X-ray micro-computed tomography (X-ray micro-CT) and focused ion beam-scanning electron microscopy (FIB-SEM), provide volumetric information about the microstructure of materials. These techniques enable the visualisation and quantification of precipitates in a three-dimensional context.

X-ray microtomography, also known as X-ray computed tomography (CT), is a versatile method for three-dimensional imaging of a wide range of materials. It can be suitable for larger precipitates and offers non-destructive imaging, enabling the analysis of larger sample volumes. X-ray microtomography is advantageous for visualising the distribution and connectivity of precipitates in larger volumes and provides a good compromise between resolution and sample size. It offers a combination of the 3D visualisation of internal microstructures, enabling the determination of the volume fraction, size, shape and distribution of precipitates. Example articles that discuss the application of X-ray microtomography to characterizing phase γ' precipitates in superalloys are Venter et al. (2016), Venter et al. (2017) and Chang et al. (2019).

Modern X-ray techniques, such as synchrotron X-ray diffraction and X-ray tomography, have revolutionised the characterisation of phase γ' precipitates. Synchrotron sources offer intense and tunable X-ray beams, enabling high--resolution diffraction studies that can reveal subtle structural details. Synchrotron XRD provides highly intense and collimated X-ray beams, enabling accurate phase identification and quantification even in complex microstructures. By analysing the diffraction patterns produced by the superalloy sample, researchers can identify the presence of different phases, such as the γ matrix and the γ' precipitates. The volume fraction of each phase can be estimated based on the intensities of their respective diffraction peaks. X-ray tomography, by contrast, enables three-dimensional imaging of microstructures, allowing researchers to visualise the distribution of precipitates within the material and study their connectivity and spatial relationships.

Electron tomography and focused ion beam scanning electron microscopy (FIB-SEM) have emerged as a powerful and versatile technique for the detailed characterisation of phase γ' precipitates within various materials, particularly in superalloys. Leveraging advanced electron microscopy and computational methods, electron tomography enables researchers to obtain three-dimensional insights into the volume fraction, size, shape and distribution of these precipitates at the nanoscale. Electron tomography is an extension of transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM), techniques that use a focused beam of electrons to probe the structure of thin samples. In electron tomography, a series of two-dimensional images (projections) are obtained as the sample is tilted or rotated. These images capture different perspectives of the sample, and by using sophisticated reconstruction algorithms, a three-dimensional volume is reconstructed. This volume provides information about the internal structure of the sample in three dimensions, enabling detailed analysis of microstructural features.

FIB-SEM combines high-resolution SEM imaging with the ability to remove material layer by layer using a focused ion beam. Electron tomography enables the determination of the volume fraction of phase γ' precipitates within a material (e.g. Ziętara, 2020). By segmenting the reconstructed volume to isolate the precipitates from the matrix, one can quantify the relative volume occupied by the precipitates. This information is crucial for understanding the material's composition and its subsequent mechanical and thermal properties. Electron tomography excels in characterising the size, shape and distribution of phase γ' precipitates. With its high spatial resolution, electron tomography can reveal nanoscale features that influence material properties. The three-dimensional reconstruction enables the precise measurement of precipitate dimensions, such as size, aspect ratio and orientation. Additionally, the distribution and spatial arrangement of precipitates throughout the material can be visualised, in which aids understanding of clustering, alignment and interconnectivity. Furthermore, electron tomography also provides crystallographic information about precipitates. By tilting the sample and collecting images at different angles, researchers can determine the crystallographic orientations of precipitates and their relationship to the surrounding matrix. This information is critical for understanding how the orientation relationship between precipitates and the matrix affects material behaviour.

Another useful research method the author would like to point out is atom probe tomography (APT). APT is a high-resolution technique that provides atomic-scale spatial information about the composition of materials. It can be particularly powerful for analysing very small precipitates and interfaces, such as those in superalloys. APT enables researchers to map the distribution of various elements within a sample with extremely high sensitivity and resolution. This technique is well suited for understanding the elemental composition, clustering and segregation of elements at the atomic level. APT provides atomic-scale 3D visualisation and compositional analysis. It is particularly useful for understanding the chemical distribution of precipitates. To summarise, atom probe tomography is an advanced technique that provides detailed three-dimensional compositional information at the atomic scale and can be used to precisely determine the composition, distribution and spatial arrangement of precipitates in materials like superalloys. Goodfellow et al. described the use of this method to study phase γ' precipitates in the superalloy (Goodfellow, 2018; Park, 2022).

Yet another method is electron backscatter diffraction (EBSD), which is used to determine crystallographic orientations and phase distributions within a material. It can be employed to study the orientation relationship between the matrix and precipitate phases as well as the distribution of precipitates. It can be also used to study grain boundaries and the arrangement of precipitates within grains (e.g. Wang, 2020).

Table 1 presents the main features and limitations of each of the research methods described above in order to facilitate an understanding of their possibilities.

Table 1. Characteristic features and limitations of the analysed research methods

	SEM	TEM	STEM-HAADF	XRD	SAXS and SANS	IA+STER	X-ray micro-CT	FIB-SEM
Characteristic features	Surface imaging with high depth of field, providing morphological information	High-resolution imaging of internal structures, lattice imaging, diffraction patterns	High-resolution imaging, contrast based on atomic number	Determining crystallography, phase identification	Probing nano to microscale structures, including precipitates	Quantitative analysis of 2D and 3D microstructures	3D imaging of internal structures	3D imaging of surfaces and subsurfaces
Requirements	Vacuum environment, conductive sample, electron beam	Ultra-thin sample sections, high vacuum, electron beam	Thin sample sections, high vacuum, electron beam	Polycrystalline samples, X-ray source	X-ray/neutron source, suitable sample environment	Micrographs, image analysis software	X-ray source, suitable sample preparation	Conductive sample, FIB-SEM instrument
Example of material	Ni-based superalloy containing γ’ precipitates	Thin TEM sections of Ni-based superalloys	TEM sections of Ni-based superalloys	Bulk Ni-based superalloy specimen	Solution-treated Ni-based superalloys	Micrographs of Ni-based superalloys	Bulk or sectioned Ni-based superalloys	Ni-based superalloy sample
Example of research	Quantifying γ’ particle size distribution on alloy surfaces	Determining γ’ precipitate sizes within the microstructure	Visualizing γ’ precipitate distribution at the atomic scale	Identifying phases, estimating volume fractions	Characterizing size distribution of γ’ precipitates	Estimating volume fraction, size, and shape of precipitates	Visualizing 3D distribution and connectivity of γ’ precipitates	Obtaining 3D microstructural information including γ’ precipitates
Limitations	Limited depth information, surface-sensitive, potential charging effects	Complex sample preparation, small field of view, potential electron beam damage	Similar to TEM, complex sample preparation	Bulk analysis, cannot provide detailed microstructural information	Sample-dependent, specialized equipment required	Relies on accurate image acquisition and processing	Spatial resolution, contrast for certain materials	Sample damage, limited penetration depth, imaging artifacts

Each of the presented methods has its advantages and limitations, and the choice of method depends on factors such as the size of the precipitates, required resolution, sample preparation capabilities, the required level of detail in the analysis and the availability of equipment. Often, researchers use a combination of techniques to gain a comprehensive understanding of precipitate characteristics.

5. Summary and conclusions

A breakdown of the advantages and limitations of the methods mentioned for determining the volume fraction and characterising the size, shape and distribution of phase γ' precipitates presented in this work is as follows:

- Scanning electron microscopy (SEM)
  Advantages:
  provides high-resolution surface imaging; compositional information through EDS; suitable for larger phase γ' precipitates

  Limitations:
  lacks true 3D information; surface-sensitive technique; sample preparation can affect results
- Transmission electron microscopy (TEM)
  Advantages:
  high-resolution imaging; detailed morphological and crystallographic information; suitable for nanoscale precipitates
  Limitations:
  complex sample preparation; requires thin samples; limited field of view
- X-ray diffraction (XRD)
  Advantages:
  provides phase identification; estimates volume fraction; non-destructive
  Limitations:
  limited size and shape information; averages over bulk sample; cannot distinguish between multiple phases
- X-ray structural analysis small- and wide-angle X-ray scattering (SAXS, WAXS)
  SAXS advantages:
  size range – suitable for analysing nanoscale features, making it useful for studying small precipitates like γ' in superalloys; provides information about the size distribution of particles, which is crucial for understanding the range of precipitate sizes present; non-destructive technique that can be applied to bulk samples, allowing for the analysis of the overall microstructure; can be used for in-situ studies, enabling the observation of changes in the microstructure under different conditions, such as temperature and stress
  SAXS limitations:
  interpretation of SAXS data requires sophisticated modelling and analysis techniques due to the complex scattering patterns produced by particles of various shapes and arrangements; limited to nanoscale – most effective for features on the nanometer scale, which might not be suitable for larger precipitates
  WAXS advantages:
  provide crystallographic information including lattice spacing and crystal orientation, which is valuable for phase identification and understanding the crystal structure of precipitates; well-suited for larger precipitates or crystallites that produce distinct diffraction peaks; can be used for quantitative analysis of crystallite size and lattice strain, providing information about the size and structure of precipitates
  WAXS limitations:
  limited to crystalline materials (the most effective for crystalline materials, it might not provide as much information for amorphous or poorly crystalline precipitates); bulk averaging (it provides an average over a relatively large sample volume, potentially masking localised variations in precipitate distribution); sample preparation (proper sample preparation is crucial for obtaining accurate results, powder samples are typically required, which might not be representative of the microstructure in some cases)
- Image analysis methods, combined with stereology principles
  Advantages:
  quantitative analysis (enables quantitative measurements of microstructural features which is crucial for accurate and objective characterisation, allowing researchers to obtain numerical data rather than relying on qualitative observations); representative sampling (selection of representative samples from a material, ensuring that the analysis accounts for the entire structure rather than being biased towards specific regions which leads to statistically robust results); efficiency (it can process a large number of images quickly, making it

possible to analyse a substantial amount of data in a relatively short time); non-destructive in some cases (most image analysis methods are non-destructive, allowing for the repeated analysis of the same sample or the analysis of valuable samples that can't be easily replaced)
Limitations:
sample preparation (the quality of results heavily relies on the proper preparation of samples – poor sectioning, staining artefacts, or inadequate image quality can introduce errors into the analysis); assumption of randomness (according to stereology principles the features of interest are randomly oriented in the sample, if the microstructure is not random, e.g. preferential alignment of precipitates, additional corrections may be necessary); complexity of 3D structures (image analysis methods tend to be two-dimensional, while techniques like serial sectioning can provide three-dimensional data, analysing complex 3D structures may be challenging); partial volume effect (when features are smaller than the imaging resolution, they may be partially visible in images, leading to inaccuracies in size and shape measurements); software and expertise (effective application requires familiarity with appropriate software and proper training. Interpretation of results also demands expertise in distinguishing true features from artefacts); boundary identification (identifying accurate feature boundaries, especially in cases of overlapping or irregularly shaped features, can be difficult and might introduce errors); limited to specific length scales (some stereological methods are more suitable for specific size ranges of features, choosing the appropriate method depends on the size of the features being analysed)

- X-ray microtomography, also known as X-ray computed tomography (CT)
  Advantages:
  non-destructive 3D imaging of the internal structure of materials, allowing analysis of the microstructure without altering or damaging the sample; full volume information (CT scanning captures information throughout the entire volume of the sample, unlike traditional 2D techniques that only provide information from surface sections which ensures that the analysis is representative of the entire material, including the distribution of γ' phase precipitates); visualisation of complex structures (it generates detailed three-dimensional reconstructions that enable researchers to visualise complex microstructures, such as the interconnected network of γ' phase precipitates within the alloy matrix, which aids in understanding the arrangement, connectivity, and interactions of the precipitates); quantitative analysis (it can provide quantitative information about the microstructure and with advanced image processing and segmentation techniques, it is possible to extract quantitative data on the volume fraction, size distribution, and shape of γ' phase precipitates); high throughput (it can be adapted for high-throughput imaging, allowing analysis of a large number of samples in a relatively short amount of time which is particularly useful for statistical analyses of precipitate distributions across different samples or conditions); in-situ studies (CT systems can be designed for in-situ studies, enabling the observation of changes in the microstructure and precipitate distribution under specific temperature, stress or other environmental conditions which provides insights into the dynamic behaviour of γ' phase precipitates); multi-scale imaging (it can capture features across a wide range of length scales, from millimetres to nanometers, making it suitable for studying both macroscopic sample structures and fine details like precipitate sizes and distributions); comparative studies (it enables to compare samples with different compositions, processing conditions, or heat treatments, allowing for the investigation of how these factors influence the γ' phase precipitate characteristics); non-destructive testing (it is also used for quality control and non-destructive testing in industries that

use Ni-based superalloys, aiding the identification of defects, voids and irregularities in manufactured components)
Limitations:
spatial resolution (which might not be sufficient to accurately capture the fine details of nanoscale precipitates, especially in complex microstructures with densely-packed precipitates and this limitation can lead to an underestimation of the volume fraction and the potential blurring of precipitate shapes); density contrast (it relies on the density contrast between different materials to distinguish them in the reconstructed images, in the case of γ' phase precipitates, which are often composed of similar elements as the matrix, the density contrast might not be strong enough to provide clear differentiation which can result in difficulties in accurately segmenting and identifying the precipitates from the matrix); beam hardening artifacts (X-ray beams used in microtomography are polychromatic, meaning they consist of a range of X-ray energies which can lead to an artifact known as "beam hardening," where higher-energy X-rays penetrate materials more effectively than lower-energy X-rays; this can distort the density measurements and affect the accuracy of the volume fraction determination, particularly in materials with varying compositions and thicknesses); sample preparation and orientation (achieving representative samples and proper orientation within the imaging setup is crucial for accurate microstructural characterisation, precipitates may have preferred orientations within the alloy matrix, and obtaining a representative sample cut can be challenging; incorrect orientations can lead to an inaccurate assessment of the precipitate distribution and shape); limited contrast for small precipitates (small γ' phase precipitates might not generate enough contrast in the X-ray images to be reliably detected and characterised, this is particularly problematic when dealing with precipitates below the microtomography's spatial resolution limits); limited quantitative analysis (quantitative analysis of precipitate size distribution and shape requires sophisticated image analysis techniques that might introduce additional uncertainties); beam damage (X-rays, especially at high energies, can potentially cause radiation damage to the material being imaged, particularly if the material is sensitive to radiation, which can alter the microstructure and affect the accuracy of the results)

- Synchrotron techniques
  Advantages:
  high resolution; provides structural and compositional information; suitable for various sample sizes
  Limitations:
  requires access to synchrotron facilities and complex data analysis
- Electron tomography and focused ion beam scanning electron microscopy (FIB-SEM)
  Advantages:
  provides 3D reconstruction; high-resolution imaging; suitable for nanoscale precipitates; advancements in electron microscope technology, detector systems, and reconstruction algorithms continue to improve the speed, resolution, and accessibility of electron tomography, combining electron tomography with other techniques, such as X-ray analysis; can provide complementary information for a more comprehensive understanding of microstructures
  Limitations:
  sample preparation is critical, as thin samples are required for electron transmission; precipitates near the sample surface might be overrepresented due to this limitation; data acquisition and reconstruction can be time-consuming and computationally intensive;

limited field of view, may introduce artefacts during milling, requires skilled operators

- 3D atom probe tomography (APT)
  Advantages:
  atomic-scale 3D visualisation; high compositional sensitivity; suitable for detailed compositional mapping
  Limitations:
  complex sample preparation; limited field of view; requires specialised equipment and expertise; limitations in terms of sample size; it may not provide a comprehensive three-dimensional view of the microstructure
- Electron backscatter diffraction (EBSD)
  Advantages:
  provides crystallographic information; can be combined with SEM imaging
  Limitations:
  surface-sensitive; limited to studying microstructures near the surface; requires special sample preparation.

In general, the choice of method depends on the specific research goals, the size of the precipitates, the required resolution, the sample characteristics and the available equipment. Often, researchers use multiple methods in combination in order to overcome the limitations of individual techniques and to gain a more comprehensive understanding of precipitate characteristics.

Among the different testing methods for characterising the size of γ' phase precipitates in superalloys, each have their own advantages and limitations. The choice of the most useful method depends on the specific research or engineering goals, the size range of the precipitates and the desired level of detail.

Each of the mentioned methods contributes unique information about the γ' phase precipitates in Ni-based superalloys (Table 1). Researchers often employ a combination of these techniques in order to gain a comprehensive understanding of the microstructure, enabling the accurate assessment of volume fraction and detailed characterisation of precipitate size, shape and distribution. In conclusion, the most useful method for characterising the size of γ' phase precipitates in superalloys depends on the specific requirements of the analysis. If atomic-scale composition and segregation information is crucial, atomic probe tomography might be the preferred choice. For high-resolution structural and morphological details at the nanoscale, electron tomography can be beneficial. For larger volumes and a balance between resolution and sample size, X-ray microtomography is a valuable option. Often, researchers might use a combination of these techniques in order to gain a comprehensive understanding of the microstructure and properties of superalloys.

References

Chen, K.-Ch., Chen, T.-Ch., Shiue, R.-K., Tsay, L.-W. (2018). Liquation cracking in the heat-affected zone of IN738 superalloy weld. *Metals* 8, 387, https://doi:10.3390/met8060387

De Graef, M., McHenry, M. E. (2012). *Structure of Materials: An introduction to crystallography, diffraction and symmetry*. 2nd ed. Cambridge University Press.

Escobar-Moreno, I., Aguirre, M. V., Andrés-López, E.-M., Viscasillas, M.J., Barba, D. (2019). Microstructural study of nickel-based superalloys through open-source software of image analysis. In *8 Th European Conference for Aeronautics and Space Sciences (EUCASS)*. 1 - 4 July 2019, Madrid, Spain. Published by the EUCASS Association. https://doi.org/10.13009/EUCASS2019-206

Ghica, C., Solís, C., Munke, J., Stark, A., Gehrmann, B., Bergner, M., Rösler, J., Gilles, R. (2020). HRTEM analysis of the high-temperature phases of the newly developed high-temperature Ni-base superalloy VDM 780 Premium. *Journal of Alloys and Compounds* 814, 152157, https://doi.org/10.1016/j.jallcom.2019.152157.

Goodfellow, A.J., Galindo-Nava, E.I., Christofidou, K.A., Jones, N.G., Martin, T., Bagot, P.A.J., Boyer, C.D., Hardy, M.C., Stone, H.J. (2018). Gamma Prime Precipitate Evolution During Aging of a Model Nickel-Based Superalloy. *Metallurgical and Materials Transactions* A 49, 718–728. https://doi.org/10.1007/s11661-017-4336-y.

Haas, S., Andersson, J., Fisk, M., Park, J.-S., Lienert, U. (2018). Correlation of precipitate evolution with Vickers hardness in Haynes® 282® superalloy: In-situ high-energy SAXS/WAXS investigation. Materials Science and Engineering: A 711, 250-258. https://doi.org/10.1016/j.msea.2017.11.035.

Kaufman E.N. (Ed.). (2012). *Characterization of Materials*. 2nd ed. Wiley Online Library.

Leng, Y. (2008). *Materials characterization: introduction to microscopic and spectroscopic methods*. John Wiley & Sons (Asia) Pte Ltd.

Palmert, F. (2009) *Oxidation and degradation of nickel-base alloys at high temperatures*. Master of Science Thesis, Stockholm, Sweden.

Park, J.-U., Jun, S.-Y., Lee, B.H., Jang, J.H., Lee, B.-S., Lee, H.-J., Lee, J.-H., Hong, H.-U. (2022). Alloy design of Ni-based superalloy with high γ′ volume fraction suitable for additive manufacturing and its deformation behavior. *Additive Manufacturing* 52, 102680, https://doi.org/10.1016/j.addma.2022.102680.

Rakoczy, Ł., Milkovič, O., Rutkowski, B., Cygan, R., Grudzień-Rakoczy, M., Kromka, F., Zielińska-Lipiec, A. (2020). Characterization of γ′ precipitates in cast Ni-based superalloy and their behaviour at high-homologous temperatures studied by TEM and in situ XRD. *Materials* 13, 2397. https://doi.org/10.3390/ma13102397.

Rundevski, T., Brown, C.M. (2021). The Rietveld Refinement Method: Half of a Century Anniversary. *Crystal Growth and Design* 21, 9, 4821–4822. https://doi.org/10.1021/acs.cgd.1c00854

Sharma, S.K. (Ed.). (2018). *Handbook of materials characterization*. Springer Cham Publisher.

Smith, T.M., Zarkevich, N.A., Egan, A.J., Stuckner, J., Gabb, T.P., Lawson, J.W., Mills, M.J.(2021). Utilizing local phase transformation strengthening for nickel-base superalloys. *Communications Materials* 2, 106, 1-9. https://doi.org/10.1038/s43246-021-00210-6.

Sun, F. (2021). Integrated TEM/transmission-EBSD for recrystallization analysis in nickel-based disc superalloy. Progress in Natural Science: *Materials International* 31, 1, 63-67. https://doi.org/10.1016/j.pnsc.2020.11.003.

Wang, Z., Muránsky, O., Zhu, H., Wei, T., Sokolova, A., Short, K., Wright, R.N. (2020). On the kinetics of gamma prime (γ’) precipitation and its strengthening mechanism in Alloy 617 during a long-term thermal aging. *Materialia* 11, 100682, https://doi.org/10.1016/j.mtla.2020.100682.

Ziętara, M, Kruk, A., Cempura, G., Gruszczyński, A., Czyrska-Filemonowicz, A. (2020). Fourth-generation single crystal nickel base superalloy for jet engine turbine blades application. 3D imaging and metrology of microstructure elements. *Materials Engineering SIGMA Publisher* 3, https://doi.org/10.15199/28.2020.3.1