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ABSTRACT

Nickel-base superalloys are used at high temperature applications in aerospace and power generation. The objective of present work is to gain a better understanding of microstructural evolution and deformation mechanisms in Nickel-base superalloys.

The microstructure of the Nickel base superalloy is basically composed of gamma matrix with eta, delta, gamma prime or gamma double prime/or both, carbides and nitrides. Cold working of IN 718 and Waspaloy to 50% reduction led to an increase in hardness. This hardening was related to the continuous increase in dislocation density in both alloys. Cold working of IN 718 to levels of 10% and higher also led to shearing of the gamma double prime precipitates present initially, leading to their dissolution and the redistribution of the alloying elements into the matrix. Shot peening of both alloys introduces near surface compressive residual stresses and a significant increase in the surface and near-surface hardness to a distance of ~200-400 μm in both alloys. Shot peening of both alloys followed by aging at 900°C quickly led to a large drop in hardness to near that of the bulk material. Aging shot peened the IN 718 at 700°C led to an increase in the hardness throughout the sample. Microstructural characterization revealed that this hardening is related to the formation of new precipitates of gamma prime or gamma double prime or both within the gamma matrix. Aging shot peened
Waspaloy at 700°C led to an increase or decrease at near surface region at short time, depending on the Almen intensity. Microstructural characterization shows that these changes are related to partial reduction in % cold work by recrystallization and/or new gamma prime precipitation, depending on the Almen intensity.

The hardening, microstructural evolution and stress rupture behavior of IN 740 were studied. Aging of the IN 740 alloy led to significant hardening due to the gamma prime precipitation. The gamma prime coarsening in aged and tensile tested and stress rupture tested IN740 alloy was observed to follow the LSW theory. Gamma prime coarsening was promoted by the applied stress and accelerated between 750 and 800°C. Applied stress was also found to significantly enhance the eta phase formation. Various models for strengthening in Ni-base superalloys were considered and their contributions to strength following aging were calculated utilizing the experimentally measured gamma prime sizes and volume fractions. Good agreement between the calculated and experimentally measured yield strengths was obtained. Based on these calculations and characterization of the deformation microstructure in the aged and tensile-tested samples, it was concluded that apart from solid solution strengthening, the main strengthening contributions from the gamma prime precipitates is associated with precipitate shearing involving either weak pair dislocation coupling or strong pair
dislocation coupling/ or both at 700°C for 100h, strong pair dislocation coupling following aging at 700°C for 1000, 3000h and 750°C for 100, 1000h, strong pair coupling or Orowan process/or both following aging at 750°C for 3000h and 800°C for 100h, and mainly Orowan process following aging at 800°C for 1000 and 3000h.
ABSTRACT

Nickel-base superalloys are metallic alloys that are used at high temperatures, sometimes in excess of 0.7 of their absolute melting temperatures. They exhibit good creep and oxidation resistance, owing to which they are used in a wide range of high temperature applications in aerospace and power generation.

The objective of present work is to gain a better understanding of microstructural evolution and deformation mechanisms in Nickel-base superalloys. Specifically, in this research, two types of Nickel base superalloys: 1) shot-peened IN718 and Waspaloy for aero engine applications, and 2) IN740 for ultra-supercritical (USC) steam boiler applications, were studied.

The microstructure of the non-cold worked IN718 alloy was basically composed of 20-μm size γ grains within which were present platelets of η-Ni₃Ti, round particles of δ-Ni₃Nb and ultrafine, nanoscale γ'' precipitates in the γ matrix, together with a low volume fraction of (Nb,Ti) carbides and (Ti,Nb) nitrides. The microstructure of the non-cold worked Waspaloy material was composed of the γ matrix grains of size 30-50-μm within which was present a low volume fraction of spheroidal, 60-200-nm diameter γ' precipitates, together with (Ti,Mo) carbides along the grain boundaries. Cold working of both alloys to 50% reduction led to an increase in hardness, with the (initial) rate of
and extent of hardening being greater in IN718 compared with Waspaloy. This hardening was related to the continuous increase in dislocation density in both alloys. Cold working of IN718 to levels of 10% and higher also led to shearing of the γ'' precipitates present initially, leading to their dissolution and the redistribution of the alloying elements into the matrix. Cold working also led to an increase in electrical conductivity in Waspaloy at all cold work levels to 50%, whereas IN718 showed a very small decrease or practically no change compared with the counterpart cold work-free materials. These changes in conductivity appear to be related to short range order in the γ matrix. Short-range order in the γ matrix of both Waspaloy and IN718 is associated with a lower conductivity than that of a fully random state. The increase in conductivity in Waspaloy, as well as the smaller change in IN718, with cold work has been explained on the basis that cold working and the passage of dislocations reduces the degree of SRO, thereby leading to a conductivity increase such as to overcompensate for or balance the decrease in conductivity expected from the increase in dislocation/defect density.

Mechanical surface treatment by shot peening of the IN718 and Waspaloy introduces near surface compressive residual stresses and a significant increase in the surface and near-surface hardness to a distance of ~200-400 μm in both alloys, with the
degree of hardening being greater at the higher Almen intensities. This hardening is
caused by the cold work/plastic strain introduced by shot peening, with the degree of
cold work and, in turn, hardness diminishing with distance from the surface.

Shot peening of the IN718 and Waspaloy followed by aging at 700 and 900°C
was performed, then microhardness measurement and microstructural characterization
were carried out. Aging of the both materials at 900°C quickly led to a large drop in
hardness to near that of the bulk material, which has been related to partial/total removal
of the initial cold work that was present. Aging shot peened the IN718 at 700°C led to
an increase in the hardness throughout the sample. Microstructural characterization
revealed that this hardening is related to the formation of new precipitates of $\gamma'$ or $\gamma''$ or
both within the $\gamma$ matrix. Aging shot peened Waspaloy at 700°C led to an increase or
decrease at near surface region at short time, depending on the Almen intensity.
Microstructural characterization shows that these change are related to partial reduction
in % cold work by recrystallization and/or new precipitation of $\gamma'$, depending on the
Almen intensity. Dislocations in shot peened samples in IN 718 and Waspaloy are of
the $b = 1/2<110>$ type lying on the (111) planes and a significant dislocation density is
still retained following aging at 700°C.
Since in ultra-supercritical (USC) condition a temperature higher than 640°C is encountered, it is likely that traditional ferritic alloys would be replaced by Ni-base superalloys. Inconel 740 alloy is a nickel base superalloy originally developed for use in ultra-supercritical (USC) steam boiler tubing applications in coal and other fossil-fired power plants, because of its attractive creep strength and corrosion resistance.

The hardening, microstructural evolution and stress rupture behavior of IN740 were studied. Aging the IN740 alloy led to significant hardening, which was associated with the precipitation of \( \gamma' \). The coarsening of \( \gamma' \) in aged and tensile tested and stress rupture tested IN740 alloy was observed to follow the \( d^2 - do^3 = kt \) kinetics predicted by the LSW theory, indicating diffusion controlled growth. The activation energy for \( \gamma' \) coarsening was determined to be 368 kJ/mole in the aged plus tensile tested samples, and 300 kJ/mole in the stress rupture tested samples, which is close to the value of 275 kJ/mole reported for diffusion of Al and Ti in Ni and those for \( \gamma' \) coarsening in binary and multicomponent Ni-base alloys. \( \gamma' \) precipitate coarsening was promoted by the applied stress and accelerated in the temperature range between 750 and 800°C. Applied stress was also found to significantly enhance the formation of \( \eta \)-Ni$_3$Ti.

Various models for strengthening in Ni-base superalloys, including solid solution strengthening, grain size strengthening, precipitate cutting processes involving...
weak-pair coupled and strong-pair coupled dislocations and Orowan bypass mechanisms were considered and their respective contributions to strength following aging were calculated utilizing the experimentally measured γ' sizes and volume fractions. Good agreement between the calculated and experimentally measured yield strengths was obtained. Based on these calculations and characterization of the deformation microstructure in the aged and tensile-tested samples, it was concluded that apart from solid solution strengthening, the main strengthening contributions from the γ' precipitates is associated with precipitate shearing involving either weak pair dislocation coupling or strong pair dislocation coupling or both at short aging times to 100h at 700°C when the γ' precipitates are small in size (<20 nm), strong pair dislocation coupling following aging at 700°C for 1000, 3000h and 750°C for 100, 1000h where the g' sizes are larger, strong pair coupling or Orowan process or both following aging at 750°C for 3000h and 800°C for 100h, and mainly Orowan process following aging at 800°C for 1000 and 3000h where the γ' sizes and interparticle spacings are even larger.
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CHAPTER 1: INTRODUCTION

Nickel-base superalloys are metallic alloys that are used at high temperatures, sometimes in or excess of 0.7 of their absolute melting temperatures. They exhibit good creep and oxidation resistance [1, 2], owing to which they are used in a wide range of high temperature applications in aerospace and power generation [1, 2]. Surface Treated IN 718 and Waspaloy by Shot Peening show good fatigue resistance. IN 740 for power plant shows good high temperature tensile and creep strengths [3].

The objective of present work is to gain a better understanding of microstructural evolution and deformation mechanisms in Nickel-base superalloys. Specially, in this research, two types of Nickel base superalloys: 1) shot-peened IN 718 and Waspaloy for aero engine applications, and 2) IN740 for ultra-supercritical (USC) steam boiler applications, were studied.

The intentional introduction of near-surface compressive residual stresses, using methods like shot peening, laser shock peening (LSP) and low-plasticity burnishing (LPB), is a well known practice for enhancing the resistance to fatigue crack nucleation and growth of turbine engine parts [4]. Nondestructive evaluation of residual stress gradients in such surface-enhanced materials has great significance for component life extension and their reliability in service, and, in this context, significant progress
has been made recently by the NDE Branch (AFRL/MLLP) towards the development of a quantitative apparent eddy current conductivity (AECC) method for residual stress measurement in nickel-base superalloys [5]. However, the difficulty in separating the residual stress (elastic) and cold work (plastic) contributions militates against the achievement of a complete analysis of the AECC results, and, furthermore, there is a lack of basic understanding of how intrinsic and extrinsic factors like microstructural changes brought about by surface and/or cold-work treatments affect the conductivity. It is thus clear that complementary research is needed to achieve better fundamental understanding of the effects of elastic and plastic strains (i.e., residual stress and cold work) on the electrical conductivity and microstructure of nickel-base superalloys, in order to develop accurate calibration techniques for quantitative nondestructive residual stress assessment in surface-treated components.

One basic question: we are seeking to answer related to the materials/microstructural factors responsible for the changes in electrical conductivity observed in surface-treated and/or cold-worked nickel-base superalloys. Recent published results indicate that an increase in AECC occurs in shot-peened nickel-base superalloys and is mainly due to an increase in the near-surface electrical conductivity of the material, but no conclusive evidence exists at this point to completely eliminate
the potential contributions of additional changes in magnetic susceptibility and microstructure. Many factors affect the electrical conductivity of materials, most notable ones being elements in solid solution, temperature, point defects, dislocations; presence of short-range and long-range order, second-phase precipitates; and electronic structure. Many Ni-base alloys display a phenomenon called the K-state effect, in which an anomalous increase in electrical conductivity occurs with an increase in the degree of cold work. This feature has been related to the occurrence of short-range order, which in some cases is responsible for lower electrical conductivity than a random solid solution due to the presence of unfilled d-electron shells and s-d electron scattering. Cold working is thought to decrease the degree of short-range order and hence resistivity to a level that more than compensates for the increase in resistivity caused by an increase in the density of defects. The intrinsic/extrinsic factors that affect the electrical conductivity of the Ni-base alloys of interest here are likely to be complex, since this will depend on the nature of both the γ matrix (composition) as well as the γ' precipitates (size, volume fraction, composition) that are present and how each is affected by surface-treatments and cold work. Therefore, as a first step, it is necessary to establish how the electrical conductivity of these superalloys changes as a result of
elastic and plastic strains and relate these to changes in microstructure to establish a basic understanding of the behavior of these materials subjected to these treatments.

The present work will combine measurements of electrical resistivity/conductivity with detailed atomic-scale characterization of microstructure and quantitative analysis in a study of surface-treated and cold worked Ni-base superalloys. Using these techniques, The study of surface-treated and cold worked Ni-base superalloys (IN 718 and Waspaloy) will provide information on what changes in microstructure and atomic configuration occur in these materials during shot peening and cold working and their relationships with the measured frequency-dependent apparent eddy current conductivity (AECC) and microhardness.

Currently, coal-fired power plants are generating more than one half of the electricity in America [6]. Sub-critical boilers (538°C, 17MPa) are now the workhorse with an efficiency of around 35%. The efficiency of utility fossil-fueled power plants is constrained by the operational limits of affordable materials for steam generators and steam turbines. In order to further increase the efficiency to 50-55 percent (based on the higher heating value (HHV) of the fuel), a program to evaluate existing alloys or identify, develop and demonstrate improved alloys for service at steam temperatures up to 760 °C is necessary. Ultimately, it may be desirable to increase steam temperatures
even further to get highest possible efficiency, and possibly reach 870 °C when the efficiency could jump to nearly 60% (HHV). Also, this efficiency jump is favored by environmental concerns since higher temperature will greatly reduce the emission of pollutants, such as CO₂ alone by more than 30% [6]. The heaviest section of coal power plants demands materials with high creep and thermal fatigue strength with excellent corrosion resistance. By deriving their strength mainly through the coherently precipitated γ’ particles, nickel-base superalloys can meet these requirements. However, beyond the intermediate temperature, rapid coarsening of the γ’ particles, often known as rafting [7] occurs, thus the strength deteriorates rapidly. The factors affecting the shape changes and splitting the γ’ precipitate particles during the coarsening have been reviewed by A. Baldan [8]. Attempts would be made to predict the life time of materials based on the microstructural development. Since it would be impractical that we put the sample under real service conditions (decades of service time and disaster risks involved), microstructural analysis for life prediction is one of the few choices [9].

Since in ultra-supercritical (USC) condition a temperature higher than 640°C is encountered, it is likely that traditional solid solution nickel-base superalloys would be replaced by precipitation hardening Ni-base superalloys [6]. Inconel 740 alloy is a nickel base superalloy originally developed for the European Thermie program for use
in ultra-supercritical (USC) steam boiler tubing applications in coal and other fossil-fired power plants, because of its attractive creep strength and corrosion resistance [10]. The samples received from ORNL are creep-ruptured. There is still not much published work on alloy 740, but it has already generated great interests as it has shown excellent resistance to corrosive attack and high strength at elevated temperatures up to 800°C [11]. A comparison of microstructural developments in these alloys would lead to a more complete understanding of nickel-base superalloys. TEM would be utilized to study the microstructure development in Inconel 740 alloy under different modes of creep tests, in terms of nano-scale $\gamma^\prime$ rafting, dislocation network in such conditions as higher temperature and longer time of exposure. The study makes it possible to predict phase transformations; this characterization tool will provide in return a fundamental understanding of these alloys in terms of microstructural behaviors.

Therefore, the study of IN740 focuses on changes in microstructure and mechanical properties over the range of 700-800°C. Specifically, to understand the deformation mechanisms under ambient as well as creep conditions, the microstructural analysis focused on the formation, distribution, size and volume fraction of $\gamma^\prime$ and their interaction with dislocations.
CHAPTER 2: LITERATURE REVIEW

2.1 Nickel and Nickel-Iron Base Superalloys

2.1.1 Superalloys

The term "superalloy" was first used shortly after World War II. Superalloys are nickel-, nickel-iron, and cobalt base alloys generally used at temperature above approximately 540°C (1000°F). Superalloys have been widely used in aircraft industrial, and marine/land based gas turbines, nuclear reactors, chemical/petroleum plants, orthopedic and dental production, and environmental protection application. They are particularly well suited for these demanding applications because of their ability to retain most of their strength even after long exposure times above 650°C (1,200°F) as well as good low-temperature ductility and excellent oxidation resistance. Superalloys are based on Group VIIIIB elements and usually consist of various combinations of Fe, Ni, Co, and Cr, as well as lesser amounts of W, Mo, Ta, Nb, Ti, and Al [12].

All three types of superalloys contain the austenitic (γ) face-centered cubic (fcc) structure as the matrix phase. Even though iron has a body-centered cubic structure and cobalt a hexagonal close-packed structure at room temperature, these alloys contain sufficient alloying additions that the fcc crystalline structure is stable at all temperatures. Superalloys are relatively heavy, with densities in the range of 7.8 to 9.4 g/cm³. Density
depends on alloying additions, with elements such as Al, Ti, and Cr reducing the density and elements such as W, Rh, and Ta increasing it [13].

**Ni-base superalloys:** the most important class of Ni-base superalloys is that strengthened by intermetallic compound precipitation in a fcc matrix. The strengthening precipitate is $\gamma'$, in Waspaloy or Udimet 700. Another class of Ni-base superalloys is represented by Hastelloy X, which is essentially a solid solution alloy but probably also derives some strengthening from carbide precipitation produced through working plus aging. A third class includes oxide dispersion strengthened (ODS) alloys such as INMA-754 or IN MA-6000E, which are strengthened by dispersions of inert particles such as yttria coupled in some cases with $\gamma'$ precipitation [14].

**Ni-Fe base superalloys:** the most important class of Ni-Fe base superalloys includes those alloys which are strengthened by intermetallic compound precipitation in a fcc matrix. The most common precipitate is $\gamma'$, A-286, Inconel 901, but in some alloys the Inconel 718 in the $\gamma''$ precipitate. Another class of Ni-Fe base superalloys is typical the CRM series, which are hardened by carbides, nitrides and carbonitrides; some W and Mo may be added to produce solid solution hardening. Other Ni-Fe base superalloys consist of modified stainless steels primarily strengthened by solid solution hardening [14].
**Cobalt base superalloys;** The Co base superalloys are invariably strengthened by a combination of carbides and solid solution hardeners. Unfortunately, Cobalt base superalloys can not be strengthened by precipitation hardening because no coherent, ordered precipitate, such as \( \gamma' \) in Nickel base superalloys, has been found for the Cobalt base alloys [14].

### 2.1.2 Nickel and Nickel-Iron Base Superalloys

Nickel base superalloys are the most complex and the most widely used for the hottest parts of turbine engines. They are available as either solid solution strengthened alloys for lower temperature applications or as precipitation hardened alloys for higher temperature use. Many nickel base superalloys contain 10 to 20 wt% Cr, a combined Al and Ti content up to 8 wt%, 5 to 15 wt% Co, and small amount of B, Zr, Mg and C. Other common additions are Mo, Nb, and W, all of which provide solid solution strengthening and form carbides. Cr and Al are added for improving surface stability of oxidation resistance through the formation of \( \text{Cr}_2\text{O}_3 \) and \( \text{Al}_2\text{O}_3 \), respectively. Typical alloys strengthened by \( \gamma' \) are the wrought alloys Waspaloy and Udimet 720 and the cast alloy Rene 80. Most wrought nickel base alloys contain between 20 to 45 vol % \( \gamma' \), while cast alloys can contain as much as 60 vol % \( \gamma' \). As the amount of \( \gamma' \) increase, the elevated temperature resistance increases; however, the workability of wrought alloys
decreases and eventually reaches a point where other fabrication methods, such as casting or powder metallurgy, must be used [14].

Nickel-Iron base superalloys evolved from austenitic stainless steels and are based on the principle of combining both solid solution hardening and precipitate hardening. The Nickel-Iron base superalloys have useful strengths to approximately 650°C (1200°F). The austenitic matrix is based on nickel and iron with at least 25 wt % Ni needed to stabilize the fcc phase. Other alloying elements, such as Cr, partition primarily to the austenitic phase to provide solid solution hardening. Most alloys contain 25 to 45 wt % Ni. Cr in the range of 15 to 28 wt % is added for oxidation resistance at elevated temperature, while 1 to 6 wt % Mo provides solid solution strengthening. The main elements that facilitate precipitate hardening are Ti, Al, and Nb. The strengthening precipitates are primarily γ′ (Ni₃Al), γ″, eta (Ni₃Ti), and δ (Ni₃Nb). Inconel 718 is the single most important wrought superalloy that is sometimes classified as a nickel base alloy and sometime as a nickel iron base alloy. It contains Nb and is therefore strengthened by primarily γ″ (Ni₃Nb). Alloys that contain Nb with Ti and/or Al are strengthened by the combination of γ″ and γ′. Inconel 718 is one of the strongest and most widely used superalloys because of moderate price, and ease of processing,
including weldability, but it rapidly loses strength in the range of 650 to 815°C (1200 to 1500°F) because of the instability of the γ” precipitate [14].

In broad terms, the elemental additions in Ni-base superalloys can be categorized as being i) γ formers (elements that tend to partition to the γ matrix, ii) γ’ formers (elements that partition to the γ’ precipitate, iii) carbide formers, and iv) elements that segregate to the grain boundaries. Elements which are considered γ formers are Group V, VI, and VII elements such as Co, Cr, Mo, W, Fe. The atomic diameters of these alloys are only 3-13% different than Ni (the primary matrix element). γ’ formers come from group III, IV, and V elements and include Al, Ti, Nb, Ta, Hf. The atomic diameters of these elements differ from Ni by 6-18%. The main carbide formers are Cr, Mo, W, Nb, Ta, Ti. The primary grain boundary elements are B, C, and Zr. Their atomic diameters are 21-27% different than Ni [12].

![Figure 1–Alloying Elements present in Ni base super alloys [15]](image-url)
The major phases in most nickel base superalloys are as follows:

**Gamma matrix (γ):** The austenitic matrix (γ) is capable of dissolving large percentages of solid solution hardeners, such as Co, Fe, Cr, Mo, and W. Additions of Al and Ti not only provide solid solution strengthening, they also form the basis for precipitation hardening. Solid solution strengthening effect is proportional to the difference in atom size between the matrix and the solute atom with a total concentration which is typically less than 10 atomic percent [13].

![Figure 2—Crystal structure of γ](image)

**Gamma prime (γ’):** the most important precipitate in nickel and nickel iron base superalloys is γ’, which is the fcc intermetallic compound, either in the form of Ni₃Al or Ni₃(Al, Ti). Other elements, such as Nb, Ta, and Cr, also enter into γ’. In nickel base superalloys, γ’ is typically Ni₃(Al, Ti), but if Co is added, it can substitute for some nickel as (Ni, Co)₃(Al, Ti). Since the γ’ precipitate has only approximately 0.1% lattice
mismatch with the \( \gamma \) matrix, \( \gamma' \) precipitates are both homogeneous and have a low surface energy, leading to extraordinary high temperature stability. The coherency between the precipitate and matrix is maintained to high temperature, and the precipitate coarsens very slowly, even at temperatures as high as 0.7 \( T_m \). Alloys with a high volume of \( \gamma' \) exhibit the rather anomalous behavior of becoming stronger with an increase in temperature up to approximately 700\(^\circ\)C(1300\(^\circ\)F). As the combined content of Al and/or Ti increases, the amount of \( \gamma' \) precipitate increases, resulting in better high temperature performance. The degree of lattice mismatch between the \( \gamma' \) precipitate and \( \gamma \) matrix determines precipitate morphology. The precipitate morphology is spherical for mismatch of 0 to 0.2 \%, cubical for mismatch 0.5 to 1\%, and platelike at mismatch above approximately 1.25\% [13].

![Crystal structure of \( \gamma' \)](image)

*Figure 3—Crystal structure of \( \gamma' \) [16]*

The \( \gamma' \) phase is based on the ordered Ni\(_3\)Al structure with nickel atoms at face centers and Al atoms (or atoms of other elements) at cube centers. The \( \gamma' \) phase is quite stable
with respect to temperature. It has a virtually constant yield strength at temperature as high as 900°C (1650°F), with the amount of work hardening increasing with temperature to a peak that occurs in the range 700°C to 900°C (1300 to 1650°F) depending on the composition. This temperature dependence of work hardening has been explained in terms of interaction between <110> and <100> dislocations [14].

**Gamma double prime (γ’):** Superalloys that contain considerable amounts of iron and about 2 to 6 % Nb are strengthened primarily by the so-called γ” precipitate, which has an ordered BCT (body centered tetragonal, D022) crystal structure based on the compound Ni3Nb. The most well-known and most widely used member of this group is INCONEL 718. The γ” precipitates are usually considerably finer than the γ’ precipitates in other superalloys and, when the alloy is heat treated for maximum strength, deformation occurs as a result of dislocations interacting with the coherency strain fields and eventually shearing the precipitate. The orientation relationships are [100]γ” || [100]γ ; [001]γ” || <001> γ. The γ’ usually forms at a higher temperature about 620°C, while the precipitation of γ” takes place at the lower temperature about 620°C. If the alloy is used above about 650°C (1200°F), the γ” coarsens very fast and some of the γ” may convert to δ phase, which is an ordered Ni3Nb phase having an orthorhombic crystal structure and generally a platelike morphology. As a result, the
alloy loses its strength and thus the applications of alloy 718 is limited to 650 °C [14]. However, some of the advanced powder processed alloys are capable of operating at peak temperature about 750°C.

![Crystal structure of γ''](image)

**Figure 4—Crystal structure of γ'' [17]**

**Carbides:** Carbon, added at levels of 0.05-0.2%, combines with reactive and refractory elements such as titanium, tantalum, and hafnium to form carbides (e.g., TiC, TaC, or HfC). During heat treatment and service, these begin to decompose and form lower carbides such as M_{23}C_6 and M_6C, which tend to form on the grain boundaries. These common carbides all have an fcc crystal structure. Results vary on whether carbides are detrimental or advantageous to superalloy properties. The general opinion is that in superalloys with grain boundaries, carbides are beneficial by increasing rupture strength at high temperature [12].
**Topologically Close-Packed Phases:** These are generally undesirable, brittle phases that can form during heat treatment or service. The cell structure of these phases has close-packed atoms in layers separated by relatively large interatomic distances. The layers of close packed atoms are displaced from one another by sandwiched larger atoms, developing a characteristic "topology." These compounds have been characterized as possessing a topologically close-packed (TCP) structure. Conversely, Ni$_3$Al ($\gamma'$) is close-packed in all directions and is called geometrically close-packed (GCP). TCPs ($\sigma$, $\mu$, Laves, etc.) usually form as plates (which appear as needles on a single-plane microstructure.) The plate-like structure negatively affects mechanical properties (ductility and creep-rupture.) $\sigma$ appears to be the most deleterious while strength retention has been observed in some alloys containing $\mu$ and Laves. TCPs are potentially damaging for two reasons: they tie up $\gamma$ and $\gamma'$ strengthening elements in a non-useful form, thus reducing creep strength, and they can act as crack initiators because of their brittle nature [12].
2.1.3 Precipitation in Ni-and Ni-Fe base superalloys

Precipitation in Ni-and Ni-Fe base superalloys generally occurs by a classical nucleation and growth mechanism. Prolonged heat treatment leads to the coarsening of precipitates. Three basic steps are considered to be involved in $\gamma'$ and/or $\gamma''$ precipitation during aging.

**Nucleation:** As solute $\gamma$ matrix is heated up and quenched, new precipitates will nucleate from the saturated $\gamma$-matrix. The Gibbs energy changes required for the nucleation of new spherical solid precipitate from matrix is

$$
\Delta G = \frac{4}{3} \pi \cdot r^3 \cdot (\Delta G_V + \Delta G_E) + 4\pi \cdot r^2 \cdot \sigma
$$

(1)

where $\Delta G_V$ is chemical free energy per unit volume, which is the driving force of nucleation, $\Delta G_E$ is elastic strain energy per unit volume, which is induced by the lattice misfit between matrix and precipitate, $\sigma$ is interface energy between matrix and precipitate.

**Growth:** The growth of the particle is driven by the reduction of the free energy, i.e., the free energy difference between new precipitate and matrix. The growth of the nucleus introduces long range diffusion of solute elements and redistribution of the atoms to satisfy the phase diagram. These reactions proceed relatively slowly, since the atoms must diffuse in the solid, but occur more rapidly at high temperature, where diffusion is
more rapid. During the growth, the interface between nucleus and matrix moves from the particle into matrix.

**Coarsening:** The precipitate coarsening involves competitive growth process in which the larger particles grow by consuming smaller one, while the volume fraction of precipitates in the system remains same even the total number of precipitates decreases. The driving force for coarsening is the reduction in interfacial energy of the system. The rate of coarsening is governed by the mass transfer rate from the shrinking particles to the growing ones through the $\gamma$-matrix. Research work has been conducted to examine the coarsening of the precipitates during high temperature exposures of the superalloys [18, 19]. It was observed that in general the precipitates grow at an almost constant volume fraction, following a cube law involving diffusion-controlled particle coarsening in agreement with the Lifshitz Slyozov-Wagner (LSW) theory [20, 21], namely:

$$d^3 - d_o^3 = kt \quad (2)$$

where $d$, $d_o$ are average and initial precipitate diameter, respectively, $t$ is time and $k$ is rate constant related to the precipitate-matrix interfacial energy, diffusivity, concentration of solute in equilibrium with a particle of infinite diameter, molar volume, and temperature.
2.1.4 Strengthening mechanisms in Ni- and Ni-Fe base superalloys

Ni- and Ni-Fe base superalloys are basically strengthened by solid solution and/or precipitation hardening. The solid solution strengthening is associated with inhibiting dislocation motion. The introduction of the solute atom into the matrix creates elastic strain in the crystal lattice to accommodate solute atom. The solute atoms cause lattice distortions that impede dislocation motion, increasing the yield stress of the material. Solute atoms have stress fields around them which can interact with those around dislocations. The presence of solute atoms imparts compressive or tensile stresses to the lattice, depending on solute size, which interfere with nearby dislocations, causing the solute atoms to act as potential barriers to dislocation propagation and/or multiplication.

Although some of the high-temperature strength of Ni-and Ni-Fe base superalloys can be attributed to solid-solution strengthening, the dominant strengthening mechanism is
considered as precipitation hardening by $\gamma'$ and/or $\gamma''$ since they prevent dislocation motion.

The $\gamma'$ precipitates can strengthen the alloys in two ways [22]:

First, coherency strain makes it difficult for dislocations to penetrate the precipitate. Since both the phases have a cubic lattice with similar lattice parameters, the $\gamma'$ precipitates in a cube-cube orientation relationship with the $\gamma$. This means that its cell edges are exactly parallel to corresponding edges of the $\gamma$ phase. Furthermore, because their lattice parameters are similar, the $\gamma'$ is coherent with the $\gamma$ when the precipitate size is small. Dislocations in the $\gamma$ matrix can not easily penetrate $\gamma'$, partly because the $\gamma'$ is an atomically ordered phase. The order interferes with dislocation motion and hence strengthens the alloy.

Second, when the dislocation interacts with the precipitates, the dislocation would cut through them or be looping/bowing around the precipitates. Sufficiently high stresses are required for dislocations to penetrate the particles since the process of dislocation shearing disrupts the stacking sequence of the ordered structure and results in the formation of an anti-phase boundary (APB). When dislocations cut the $\gamma'$, anti-phase boundary (APB) energy must be created because new surfaces (APB) of the precipitate would get exposed to the matrix and the precipitate/matrix interfacial energy
would increase. Thus, shearing of the precipitates often occurs via ‘weakly or strongly coupled’ dislocation pairs so that order is not destroyed [23]. With the magnitude of the APB energy being independent of the size of the $\gamma'$ precipitates, small precipitates (less than approximately 10–20 nm) are more easily sheared than large precipitates (~300 nm). For small precipitates the pair of dislocations may not lie within an individual precipitate (Figure 7(a)). This case is known as weak pair coupling (also referred as underaged condition). When the precipitates are larger, the trailing dislocation enters a precipitate before the leading dislocation exits (Figure 7(b)). This situation is known as strong pair coupling (also referred as peak aged condition). Figure 7(c) schematically compares the stress required to push a pair of dislocations through a precipitate for each mechanism as a function of precipitate size. The active mechanism changes from weak pair coupling to strong pair coupling as the $\gamma'$ size increases [24]. As particle sizes increase for a constant volume fraction, the mechanism of deformation also changes. Since the spacing between the particles also increases, it becomes more favorable for dislocations to loop or bow between the precipitates which results in Orowan loop.
Figure 6—This is a schematic illustrating how the dislocations can interact with a particle. It can either cut through the particle or bow around the particle and create a dislocation loop as it moves over the particle [22].

Figure 7—(a) Configuration of dislocations and precipitates during weak pair coupling, (b) configuration of dislocations and precipitates during strong pair coupling, and (c) relative strength due to each mechanism vs precipitate size [24]

During deformation, dislocations pile up at the matrix-precipitates interface if the resolved shear stresses are not sufficient to cause shearing of the γ' precipitates. As dislocations accumulate, an internal stress associated with the pile-up eventually enables shearing of the particle and the formation of an APB.
The strengthening by $\gamma'$ is related to particle size, APB energy and fault energy of $\gamma'$, $\gamma$ strength, $\gamma'$ strength, coherency strain, volume fraction of $\gamma'$, diffusivity in $\gamma$ and $\gamma'$, $\gamma$- $\gamma'$ modulus mismatch. The most direct correlations can be made with $V_f$ of $\gamma'$ and with $\gamma'$ particle size. Before the age hardening peak is reached during precipitation, the strength mechanism involves cutting of $\gamma'$ particle by dislocation and strength increase with $\gamma'$ size at constant $V_f$ of $\gamma'$. After the age hardening peak is reached, strength decrease with continuing particle growth because dislocation no longer cut $\gamma'$ particle but bypass them. This effect can be demonstrated for tensile or harness behavior in low $V_f$ of $\gamma'$ alloys (A-286, Inconel 901, Waspaloy) but is not as readily apparent in high $V_f$ of $\gamma'$ alloys such as Mar-M206, IN-100, etc [14].

![Figure 8](image)

*Figure 8—Dependence of room temperature hardness of a Ni-Cr-Al alloy on ageing treatment (22% Cr-2.8% Ti-3.1% Al) [14].*
2.1.4.1 Strengthening mechanisms with penetrable particle

Nembach [25] studied and summarized strengthening mechanism and CRSS model of penetrable and impenetrable particles. In the proposed following strengthening mechanism and CRSS models are summarized and the various hardening mechanisms are described. Five interactions between penetrable particles and dislocations will be distinguished. The particle-matrix interface is assumed to be free of dislocation. Thermal activation is disregarded.

Chemical Strengthening: when the dislocation cuts a coherent particle, an additional particle-matrix interface is created. The externally applied stress must supply the energy stored in the interface. Because the particle is coherent, the specific surface energy is low and the ensuing hardening effect is very small. An alternative name for this strengthening is “surface” strengthening. This particle-dislocation interaction is the energy storing type. Kelly and Fine [26] interpreted particle hardening of two Al base alloys (Al rich Al-Cu and Al rich Al-Ag alloys) as being due to chemical hardening. They derived estimates of the critical resolved shear stresses (CRSS) of these alloys from the energy difference $\Delta E$ between a copper and a silver atom in the particle and in Al-rich matrix. Brown and Ham [27] also interpreted the strengthening of Cu by Be-rich zones as being due to chemical strengthening. Harkness and Hren [28] also interpreted
the hardening of Al-Zn alloys as being due to chemical strengthening by Guinier-
Preston zones. However, Gerald [29] rejected their interpretation. So far chemical
strengthening has not been proven to be principal hardening mechanism in any system.

*Modulus Mismatch Strengthening:* The energy density in the strain field of a
dislocation is proportional to the shear modulus of material. If that of the matrix and the
particle differ from each other, the particle attracts or repels the dislocation, depending
on whether the modulus of the particle is smaller or larger than that of the matrix phase.
This interaction is elastic. Russel and Brown [30] interpreted the strengthening of Fe
rich Fe-Cu alloys as being due to modulus mismatch strengthening. Melander and
Persson [31] analyzed the data of Dunkeloh et al.’ [32] and interpreted the strength of
Al base Al-Zn-Mg alloys as being due to modulus mismatch. The strength of Fe rich
Fe-Cu alloys and Al rich Al-Zn-Mg alloys probably drives from the modulus mismatch
between particles and matrix. In Al alloys it can be applied only to the coherent Guinier-
Preston zones, but not to the impenetrable η-particles. In other systems, modulus
strengthening may not be major strengthening mechanism. If the principal particle-
dislocation interaction force peaks at the particle-matrix interface, then lattice mismatch
strengthening becomes dominant.
**Stacking Fault Mismatch Strengthening:** This mechanism is analogous to modulus mismatch strengthening. A difference in the specific fault energy between particle and matrix leads to an elastic particle-dislocation interaction. Hirsch and Kelly [33] derived estimates for the maximum interaction force that a particle exerts on a dissociated dislocation. However, Brown and Ham [27] criticized this proposition, because a particle which has a stacking fault energy mismatch is no diffuse obstacle. The most elaborate model of stacking fault energy mismatch strengthening has been proposed by Gerold and Hartmann [34] in the Al-Ag alloy system.

**Lattice Mismatch Strengthening:** If the lattice constant of the coherent particle differs from that of the matrix, the particle is surrounded by a stress field, via which it interacts elastically with dislocations. This mechanism is often referred to as “coherency” strengthening. There are two important reasons for lattice mismatch strengthening. First, from the comparison with other mechanisms, the lattice mismatch strengthening is strong. Second, in contrast with modulus and stacking fault energy mismatch, it is possible to calculate accurate interaction force between undissociated edge dislocation and a spherical particle that has a lattice mismatch. Since the interaction between a particle and a straight infinitely long screw dislocation vanishes, only edge dislocation have to be considered. Many precipitate hardening alloys are believed to be controlled
by lattice mismatch strengthening mechanism. Three major examples are Al-Zn, Cu-Fe, and Cu-Co system. All three systems have been found to be in qualitative or quantitative agreement with experimental data. Cu-Co, which has coherent Co rich particles, shows the best example for lattice mismatch strengthening and has been studied mostly extensively.

**Order Strengthening:** If the matrix and the particle have similar or even the same crystal structure, but only the particles are long-ranged-ordered, whereas the matrix is disordered or short rage ordered, a perfect matrix dislocation is only a partial dislocation in the particles and it creates anti-phase boundaries (APB) within particle. In such materials, dislocations glide in pairs; the trailing dislocation eliminates the anti-phase boundaries (APB). Order strengthening is the relevant mechanism in technical Nickel base superalloys, in which L1₂ long range ordered γ' particles are distributed in the nickel rich fcc matrix. Long range ordered particles are obstacles of the energy storing type, because the two dislocations of a pair interact rather independently of each other with the particles.

### 2.1.4.2 Strengthening mechanisms with impenetrable particles

If the particles are incoherent, dislocations circumvent or bypass them. The relevant process was suggested by Orowan [35]. The external shear stress $\tau_{\text{EXT}}$ increases
from left to right in figure 9. \( \tau_{\text{EXT}} \) causes the dislocation to bow out between the particles. The segment \( \Delta S_1 \) and \( \Delta S_2 \) have opposite sign and attract each other. If \( \tau_{\text{EXT}} \) is sufficiently high, \( \Delta S_1 \) contacts \( \Delta S_2 \) and both of them annihilate. The Orowan process can apply not only if the particles are incoherent, but also if the particles are coherent, but dislocations can not glide in them if they are not ductile, or if the particles are coherent and ductile but they are so widely spread overaged condition that the stress required to shear exceeds that to bypass them.

![Figure 9](image)

*Figure 9—A dislocation bypass impenetrable particles. The external stress increases from left to right. [26].*

2.1.4.3 Modeling of Critical Resolved Shear Stress (CRSS)

The critical resolved shear stress (CRSS) is defined as the resolved shear stress necessary to make dislocations glide over macroscopic distance. In particle hardened materials the external stress must be high enough to overcome the interaction between particles and dislocations. The models are concerned with the critical resolved shear stress (CRSS) of materials strengthened by particles. In a real materials, the total CRSS
\( \tau_f \) consists of particle CRSS \( \tau_p \) and solid solution matrix CRSS \( \tau_s \), because it is not possible to produce materials without solid solution matrix. However, the models in this chapter are dealing with CRSS \( \tau_p \) due to the difficulty in measuring CRSS \( \tau_s \). In many materials it is possible that particles grow in size with the duration time (t) of the heat treatment and to keep their total volume constant; they become large but less numerous. If the particles are sheared by dislocations, the CRSS increases with time until a maximum is reached at \( t = t_m \) (peak aged state). For \( t < t_m \) the material is said to be underaged. Further heat treatment leads to the CRSS decrease with time. For \( t > t_m \) the material is said to be overaged though the particles may still be coherent, they have become so widely spaced that it is easier for dislocations to circumvent than to shear them.

2.1.4.3.1 The CRSS Model of penetrable particles

In nickel base superalloys the dominant strengthening is precipitation hardening by \( \gamma' \) particles. Thus, order strengthening is key controlling mechanism of these alloys, in which L1₂-long rage ordered \( \gamma' \) particles are in the nickel rich fcc matrix. In this chapter all CRSS models are focused on the order strengthening mechanism of Nickel base superalloys.
Crystallography of the L12-phase: The elementary cell of the L12-structure is basically fcc. It consists of two sublattices. A site is the faces of the elementary cell and B site is the corners of the elementary cell. Thus the stoichiometric composition is A3B. Figure 10 shows three successive (111)-planes of the L12 structure. All 12 nearest neighbors of B atoms are A-atoms. The Burgers vector of a perfect dislocation in a fully ordered L12 materials is type a<101>, whereas a is the lattice constant. A perfect dislocation dissociates into two identical partials:

\[ a[10\bar{1}] = (\frac{a}{2})[10\bar{1}] + (\frac{a}{2})[10\bar{1}] \]  

These partials are perfect dislocations in the disordered fcc matrix. Each of two \((a/2)[10\bar{1}]\)-partials may dissociate further into two Shockley partials:

\[ a[10\bar{1}] = (\frac{a}{6})[11\bar{2}] + (\frac{a}{6})[2\bar{1}\bar{1}] + (\frac{a}{6})[11\bar{2}] + (\frac{a}{6})[2\bar{1}\bar{1}] \]  

The \((a/6)[11\bar{2}]\)-partial P1 is the leading one of a procession of four partials; the last one, P4 has the Burgers vector \((a/6)[2\bar{1}\bar{1}]\). Already P1 disturbs the long range order: P1 makes the B-atom B1 a nearest neighbor of another B-atom, namely B1. There is a complex stacking fault between P1 and P2, and the same holds for P3 and P4. Between P2 and P3 there is an anti-phase boundary; its specific energy will be referred to as \(\gamma\). \(\gamma\) of the L12-ordered \(\gamma\)-precipitates in nickel base superalloy is about 0.2J/m² [36], but can change with compromise of \(\gamma\) and \(\gamma\). P4 restores the L12-order. Let \(S_{ij}\) be the
separation of \( P_i \) from \( P_j \). \( S_{ij} \) is governed by the shear modulus and by the specific energy of the complex stacking fault (\( S_{12}, S_{34} \)), respectively, of the anti-phase boundary (\( S_{23} \)). \( S_{23} \) is much larger than \( S_{12} \) and \( S_{34} \). If \( L1_2 \)-long rage ordered precipitate are embedded in a disordered fcc matrix, \( S_{23} \) may exceed the inter-particle spacing whereas \( S_{12} \) and \( S_{34} \) are negligible. Therefore, the dissociation into Shockley partials according to Eq. (4) will not be considered.

\[
\begin{align*}
E_1, 1 \leq i \leq 3, \text{ of the } L1_2\text{-ordered intermetallic compound } A_3B. \text{ The radii of the circles decrease as } i \text{ increase. The dislocations glide between } E_1 \text{ and } E_2. \\
b = a[10\overline{1}], \quad b' = (a/2)[10\overline{1}], \text{ and the dashed arrows mark Burgers vectors of the type } \\
b^* = (a/6)[211].
\end{align*}
\]
Dislocation Configuration in materials strengthened by Long range ordered particles:

Nembach et al [37] observed the dislocation configuration of the tensile tested single crystal nickel base superalloy NIMONIC PE 16. The leading \((a/2)<110>\) - dislocation D1 is strongly scalloped, whereas the trailing one D2 is nearly straight or just follows the overall curvature of D1. Since it was not possible to image the long range ordered \(\gamma\)' particle and the dislocation simultaneously, they concluded that in the critical moment when D1 breaks free from the \(\gamma\)' particles, D2 may be considered as straight. In this case the ratio \(d_2/L_2\) follows

\[
d_2/L_2 = f \quad (5)
\]

Where \(d_i\) is the mean length of the dislocation \(D_i\) lying inside of a \(\gamma\)' particle, and \(L_i\) is the mean spacing of \(\gamma\)' particles along \(D_i\). \(d_i\) and \(L_i\) are meant for the critical configuration, and they are indicated in Figure11.

Figure 11—Critical configuration of the paired dislocation D1 and D2 in a material strengthened by long range ordered \(\gamma\)' particles, shown schematically. antiphase boundaries are shown hatched [37]
Pretorius and Ronnpangel’s [38] computer simulation concluded that the antiphase boundaries pull D2 forward so that it lies outside of all \( \gamma' \) particles. This means that Eq (5) should be replaced by

\[
d_2 / L_2 = 0 \quad (6)
\]

From these two equations, the actual configuration of D2, the ratio \( d_2/L_2 \) is written as

\[
d_2 / L_2 = \alpha_\gamma \cdot f \quad (7)
\]

With \( 0 \leq \alpha_\gamma \leq 1.0 \). \( \alpha_\gamma \) has to be derived from experimental data and \( f \) is volume fraction.

The subscript \( \gamma' \) of \( \alpha_\gamma \) reminds one of \( \gamma' \) strengthening. The mean number of \( n_P \) of pairs needed to cut the \( \gamma' \) particle in two is given by

\[
n_P = \frac{2r_r}{2b} = \frac{r_r}{b} \quad (8)
\]

where \( b \) is the length of the Burgers vector of D1 or D2 that is \( b = a/\sqrt{2} \) and \( r_r \) is the mean radius of the intersections of parallel planes with spherical particles.

**Modeling of the CRSS:** Though many pairs of dislocations may glide in the same plane, only a single pair will be considered in below; that is the elastic interaction between pairs will be disregarded. The resolved shear stress \( \tau_{\text{obst}\gamma} \) due to \( \gamma' \) particle follows [25]

Inside of the \( \gamma' \) particle: \( \tau_{\text{obst}\gamma} = \pm \gamma/b \)

Outside of \( \gamma' \) particle: \( \tau_{\text{obst}\gamma} = 0 \)
$\gamma$ is the specific energy of the antiphase boundaries. The plus/minus signs in the above equation apply to D1/D2, respectively. The straight line approximation yields the following for the force profile $F_g(y, -hZ)$ of a spherical $\gamma'$ particle of radius $d$ centered at $(0, 0, h)$: $\rho(h_Z^2 + y^2)^{1/2}$; that is particle of the dislocation lies inside of the $\gamma'$ particle:

$$F_g(y, -h_Z) = \pm 2(\rho^2 - h_Z^2 - y^2)^{1/2} \cdot \gamma \quad (9)$$

$\rho \leq (h_Z^2 + y^2)^{1/2}$; that is the entire the dislocation lies outside of the $\gamma'$ particle:

$$F_g(y, -h_Z) = 0 \quad (10)$$

The critical configuration of a dislocation pairs shearing coherent, spherical long range ordered precipitates is in Figure 11. In the critical moment following balance of force are;

For the leading dislocation D1: $\tau_p \cdot b \cdot L_1 + \tau_{mut} b \cdot L_1 - \gamma \cdot d_1 = 0$

For the trailing dislocation D2: $\tau_p \cdot b \cdot L_2 + \tau_{mut} b \cdot L_2 - \gamma \cdot d_2 = 0$

$\tau_p$ is the CRSS and $L_i$ is the average spacing of $\gamma'$ particles along $D_i$. $\tau_{mut}$ represents the repulsive elastic interaction between D1 and D2. The average forces exerted by $\gamma'$ particle on D1 and D2 are $-\gamma d_1$ and $\gamma d_2$, respectively. The two forces $\tau_p b L_1$ and $\tau_{mut} b L_1$ drive D1 forward. The corresponding forces $\tau_p b L_2$ and $-\tau_{mut} b L_2$ acting on D2 nearly cancel. Therefore D1 bows out strongly between the $\gamma'$ particles, whereas D2 is nearly straight or follows the overall curvature of D1. Elimination $\tau_{mut}$ from Eqs (11) yields
\[ \tau_p = \frac{\nu}{2b} \left( \frac{d_1 - d_2}{L_1 - L_2} \right) \] (11)

**Underaged State**

**1. Brown and Ham [27] Model**

\[ \tau_p = \frac{\gamma}{2b} \left\{ C_1 \left( \frac{4 \cdot \omega_C^3}{\pi \cdot \omega_q} \right)^{1/2} \left[ \frac{\gamma \cdot r \cdot f}{S} \right]^{1/2} - \alpha_{\gamma'} \cdot f \right\} \] (12)

Where \( \gamma, r \) and \( f \) are the specific anti-phase boundary energy, the average radius, and the volume fraction of the \( \gamma' \)-precipitates, respectively. Here the statistical factors \( C_1, \omega_r \), and \( \omega_q \) have been introduced. \( \omega_r \) and \( \omega_q \) depend on the distribution function of the particle radii.


\[ \tau_p = \frac{\gamma}{2b} \left\{ C_1 \left( \frac{4 \cdot \omega_C^3}{\pi \cdot \omega_q} \right)^{1/2} \left[ \frac{\gamma \cdot r \cdot f}{S} \right]^{1/2} + \frac{2 \cdot C_1 \cdot C_2 \cdot \xi \cdot \omega_q^2}{\pi \cdot \omega_q} - \alpha_{\gamma'} \right\} \cdot f \] (13)

Where \( C_2 \) is statistical factors, \( \xi \) is defined as \( w_y/r_n \), \( w_y \) is the interaction force between a \( \gamma' \) particle.

**3. Ardell [40] model**

\[ \tau_p = \frac{\gamma}{2b} \left\{ \left[ \frac{4 \cdot \omega_C^2}{\pi \cdot \omega_q} \right]^{1/2} \left[ 2 \cdot f \cdot y \cdot (1 + 36y^2)^{1/2} - \alpha_{\gamma'} \right] - \alpha_{\gamma'} \cdot f \right\} \] with \( y = \frac{\gamma \cdot r_n}{2S(\theta_d = 90^\circ)} \) (14)

**Peak-aged state**

\[ \tau_p = \frac{\gamma}{2b} \left\{ \left[ \frac{\omega_r}{(\pi \cdot \omega_q)^{1/2}} \right] \cdot f^{1/2} - \alpha_{\gamma'} \cdot f \right\} \] (15)
2.1.4.3.2 The CRSS Model of impenetrable particles

Orowan[34] estimates the CRSS $\tau_p$ as follow;

$$\tau_p = \frac{\mu_s \cdot b}{L_p} \quad (16)$$

where $\mu_s$ is the shear modulus of the matrix, b the length of the Burgers vector, and $L_p$ the free space between particles in figure 9. Bacon et al. [41] calculated the CRSS $\tau_p$ of the Orowan process on the basis of the dislocation self-stress. They are considered the self-interactions between different parts of a bent dislocation. For example the attraction between the segment $dS_1$ and $dS_2$ and between the segment $dS_2$ and $dS_3$ are considered.

The equations are as follows;

Screw dislocation:

$$\tau_p (\theta_{do} = 0^o) = \frac{\mu_s \cdot b}{2 \cdot \pi \cdot L_p} \cdot \frac{1}{1 - v} [\ln(D/R_i) + B] \quad (17a)$$

Edge dislocation:

$$\tau_p (\theta_{do} = 90^o) = \frac{\mu_s \cdot b}{2 \cdot \pi \cdot L_p} [\ln(D/R_i) + B] \quad (17b)$$

Where $\theta_{do}$ is the angle between the dislocation and its Burgers vector before bow out, $R_i$ is the dislocation’s inner cutoff radius and $v$ is Poisson’s ration, which was assumed to equal $1/3$. The numerical constant $B$ is about 0.65.

$D$ is a function of $L_p$ and $d_p$:

$$\frac{1}{D} = \frac{1}{L_p} + \frac{1}{d_p} \quad (18)$$
Where \(d_p\) is the particle’s diameter measured in the glide plane, \(L_p\) is the interparticle spacing.

Scattergood and Bacon [42] introduced the anisotropic shear modulus \(\mu_K\) and the anisotropic Poisson’s ration \(v_K\). The equations are as follow;

**Screw dislocation:**

\[
\tau_p(\theta_{do} = 0^\circ) = \frac{\mu_k \cdot b}{2 \pi L_p} \cdot \frac{1}{(1 - v_k)} \left[ \ln \left( \frac{D}{R_i} \right) + B \right] \quad (19a)
\]

**Edge dislocation:**

\[
\tau_p(\theta_{do} = 90^\circ) = \frac{\mu_k \cdot b}{2 \pi L_p} \left[ \ln \left( \frac{D}{R_i} \right) + B \right] \quad (19b)
\]

Bacon et al [41] also introduced the CRSS of a random array of particles in order to obtain the CRSS of real particle arrays. The equation is given by;

\[
\tau_p = 0.9 \cdot \frac{2 \cdot K_{Eg} \cdot b}{r \left( \frac{\pi \cdot \omega_k}{f} \right)^{\frac{3}{2}} - 2 \cdot \omega_r} \cdot \left\{ \ln \left( \frac{4 \cdot \omega_k \cdot r}{R_i} \right) \right\}^{\frac{3}{2}} \quad (20a)
\]

Where \(K_{Eg}\) is the geometric mean of the energy parameter of edge and screw dislocation and \(R_i\) is the inner cutoff radius, which is close to \(b\). \(K_{Eg}\) can be written by;

\[
K_{Eg} = [K_E(\theta_d = 0^\circ) \cdot K_E(\theta_d = 90^\circ)] \quad (20b)
\]

Where \(K_E = \frac{\mu}{4 \pi (1 - \nu)} [1 - \nu \cos^2 \theta_d]\) with poisson’s ratio \(\nu\) and shear modulus \(\mu\).
2.1.4.4 Strengthening mechanism with Solid Solution

The theory for solid-solution strengthening of the nickel matrix is based on a model proposed by Gypen and Deruyttere,[43] which assumes a superposition of strengthening of individual solutes that individually have differing potencies.[44-47] Because solute spacing is proportional to the square root of the concentration, the resultant strengthening is given by

$$\Delta \sigma = \sum_{i} \left( \frac{d\sigma}{dC_i} \cdot \sqrt{C_i} \right)$$ (21)

where $d\sigma/dC_i$ is a strengthening coefficient that reflects the strengthening potency of each alloying element.

2.1.4.5 Strengthening mechanism with Grain Size

The most commonly encountered strengthening mechanism is that associated with variations in grain size. Strengthening is well known to be inversely proportional to the grain size; this phenomenon has been attributed to the existence of grain boundaries as sources of dislocations and barriers to deformation. According to the Hall-Petch relation [48], grain size strengthening can be written by

$$\sigma = \sigma_o + K_y \cdot d_m^{-\frac{1}{2}}$$ (22)

Where $\sigma$ is the yield strength, $\sigma_0$ is the friction stress, $k_y$ is the Hall–Petch coefficient, and $d_m$ is the average grain size.
2.1.4.6 Superimposition

In general, Nickel base superalloys are strengthened by mainly solid solution and $\gamma'$ precipitates as well as grain size, it is necessary to consider how these strengthening increments should be superimposed. There are several possible methods, most general form is

$$\sigma_{Total} = \sum_i \Delta \sigma_i^k \quad (23)$$

Where $k$ can be taken between 1 to 2. The $k$ from literature for nickel base superalloy is 1.13 to 1.19 and 1 for theoretical calculation [24].

2.1.5 Mechanical Properties

The mechanical properties of Ni- and Ni-Fe base superalloys are greatly dependent on microstructure. In addition to the volume fraction, size, shape and morphology of the $\gamma'$ or $\gamma''$ play a major role in determining the strength of the alloy [14].

The shape and structural stability of the $\gamma'$ depend on the misfit parameter, $\delta$, which is defined as :

$$\delta = \frac{a_p - a_m}{a} = 2 \cdot \frac{a_p - a_m}{a_p + a_m} \quad (24)$$

Where $a_p$ is the lattice parameter of precipitate, $a_m$ is the lattice parameter of matrix and $a$ is the average lattice parameter. When either the particle size or the misfit parameter is small, the $\gamma'$ particles tend to assume spherical shape. For alloys having large misfit
parameters, continued aging results in cuboidal precipitates whose faces are parallel to {100} matrix planes. This effect can be explained simply by noting that surface energy effects dominate when particle are small, whereas strain energy effects dominate when particle are large. Because {100} planes are soft, elastic deformation is most easily accommodated when these planes are the matching planes. This misfit parameter depends on composition [49]. The study showed that elements such as Nb and Ti tend to increase the misfit parameter whereas Fe and Mo tend to decrease it. The misfit parameter is important because it plays a large role in determining deformation characteristics. When the misfit or coherency strain is large, deformation tends to occur by looping of precipitates by dislocation; when the misfit is small, shearing occurs. The deformation mode plays in an important role in determining strength, fracture toughness and fatigue properties. When large negative misfit is present at the precipitate-matrix interface, this tends to repel dislocations and slow down the rate of recovery at low temperatures. Compared to other superalloys containing similarly low levels of $\gamma'$, the unusually high strength and low precipitate coarsening kinetics of IN 718 are often attributed to the large coherency strains at $\gamma - \gamma''$ interfaces. As temperatures increases, however, the dominant deformation mechanisms change and microstructural stability becomes critical. At high temperatures ($T>\sim0.6\ TM$), dislocations are sufficiently
mobile that by-pass mechanisms, such as dislocation climb and bowing, may potentially be more energetically favorable than cutting of the precipitate. With the lower applied stresses typically associated with high-temperature deformation generally insufficient to result in shearing of the \( \gamma' \) and Orowan stresses providing resistance to bowing of the dislocation between the precipitates, dislocations are forced to climb. As climb is a thermally controlled process and a rate-limiting step, strain rates are correspondingly low when dislocations are not able to cut the \( \gamma' \) precipitates. Within the \( \gamma' \) precipitate, thermal activation of cross slip from the \{111\} octahedral to \{001\} cube planes may occur and contribute to the high-temperature strength of the alloy. With the deformation characteristics of these polycrystalline Ni-base superalloys highly sensitive to the APB energies of the precipitate and temperature, substantial increases in strength and creep resistance can be incurred by increasing the volume fraction of intermetallic precipitates. As the volume fraction of \( \gamma' \) increases to over 50%, optimum structural properties are attained when cuboidal \( \gamma' \) precipitate morphologies measuring approximately 0.5 \( \mu \text{m} \) in diameter are dispersed within the \( \gamma \) matrix. Under these conditions, the microstructure is highly resistant to deformation since the stresses required for dislocation bowing are approximately the same as the stresses required for dislocation cutting of the \( \gamma' \) precipitates. The small misfit between the \( \gamma \) and \( \gamma' \) lattices is
important for two reasons. Firstly, when combined with the cube-cube orientation relationship, it ensures a low $\gamma'/\gamma$ interfacial energy. The ordinary mechanism of precipitate coarsening is driven entirely by the minimisation of total interfacial energy. A coherent or semi-coherent interface therefore makes the microstructure stable, a property which is useful for elevated temperature applications [14, 50].

The morphological stability of $\gamma'$ has been studied as a function of applied stress, and it has been shown that the equilibrium microstructure depends on the sense of the applied stress and the misfit parameter. For example, in alloys for which the misfit parameter is positive, a strong along [001] will convert cube into plates lying on (001) [14].

2.2 Surface Treatment

It is well-known that surface properties play an important role in determining the performance of structural components, especially their fatigue resistance. In most cases, the failure of critical structural components originates in surface areas under tension, which leads to crack initiation and propagation. The existence of a near-surface compressive stresses can cancel out service-induced tensile stresses, thus preventing crack nucleation and growth and ultimately increasing the service life of the component. Therefore, Surface enhancement technologies such as shot peening (SP), laser shock
peening (LSP) and low plasticity burnishing (LPB) can provide substantial fatigue life improvement.

2.2.1.1 Shot Peening Process

Shot peening is a cold working process in which small spherical media called shot bombard the surface of a part. Figure 12 shows how residual compressive stresses produced by shot peening. During the shot peening process, each piece of shot that strikes the material acts as a tiny peening hammer, makes a small indentation or dimple on the surface. To create the dimple, the surface of the material must yield in tension. Below the surface, the material tries to restore its original shape, thereby producing below the dimple, a hemisphere of cold-worked material highly stressed in compression [51, 52].

Figure 12—Schematic representation of Residual stresses produced by shot peening.

2.2.1.2 Low Plasticity Burnishing (LPB) Process

In the LPB process, a smooth, free-rolling spherical ball is pressed against and rolled along the surface of material to be burnished. To ensure free rolling, the ball is
supported in a spherical-socket fluid bearing with sufficient fluid pressure and flow to
maintain for free rolling between the ball and the socket. The force with which the ball
is pressed against the surface is made large enough to deform the surface of the material
in tension, creating a compressive layer of residual stress. The process is shown
schematically in Figure 13[53].

![Figure 13– Low Plasticity Burnishing (LPB) schematic [53]](image)

By use of the positioning capability of a computer numerically controlled (CNC)
machine tool, the ball is moved along the surface in a raster or other suitable pattern to
cover the surface in a series of passes at a controlled separation chosen to obtain
maximum compression with minimum cold working [54].

### 2.2.1.3 Laser Shock Peening Process

The LSP process which is illustrated in Figure 14 involves irradiating a target
with laser pulse. The area of the critical component that needs to be Laser Shock Peened
is coated by the opaque overlaying material that is absorbing to laser radiation,
commonly black paint or tape. Transparent overlaying layer is usually water, which is
transparent to laser radiation but can act as a confinement medium for the plasma. The
laser pulse passes through the transparent overlay and strikes the opaque overlay
causing it to begin to vaporize. The vapor absorbs the remaining laser light and
produces rapidly expanding plasma by partially ionizing. Since the expanding plasma is
confined between the surface of material and the transparent overlay, the expansion
causes high-pressure shock wave to propagate into the material. The pressure of the
shock wave is greater than the yield strength of the material under shock conditions,
plastically deforming the material to a depth where the pressure is no longer higher than
the yield strength. The undeformed material attempts to restore the original shape of the
surface, causing compressive residual stress fields to be set up in the near surface region
of the material [54, 55].

Figure 14—Schematic of the Laser Shock Peening Process
2.2.2 Compressive Residual Stresses by Surface Treatment

Residual Stresses are stresses that remain after all manufacturing processes are completed, and with no external load applied. Residual Stresses can be either tensile or compressive. In most applications for low plasticity burnishing, laser shock peening and shot peening, the benefit obtained is the direct result of the residual compressive stresses produced.

This compressively stressed layer is extremely effective, in preventing premature failure under conditions of cyclic loading since the fatigue failure generally propagates from the free surface of a material. In most modes of long-term failure, the common condition is tensile stress. Tensile stresses attempt to stretch or pull the surface apart and may eventually lead to crack initiation. Because crack growth is slowed significantly in a compressive layer, increasing the depth of this layer increases crack resistance [56]. The distribution of residual stress below the surface treated material is illustrated in Figure 15 [51]. The maximum compressive residual stresses are produced just below the surface of material.
2.2.3. Compressive Residual Stress Measurement

When a metal is deformed elastically in such a manner that the strain is uniform over relatively large distance, the lattice plane spacing in the constituent grains change from their stress-free value to some new value corresponding to the magnitude of applied stress, this new spacing being essentially constant from one grain to another for any particular set of planes similarly oriented with respect to the stress. This uniform macro-strain causes a shift of the diffraction lines to new $2\theta$ positions. If the metal is deformed plastically, the lattice planes become distorted in such a way that the spacing of any particular (hkl) set varies from one grain to another. This non-uniform micro-strain causes a broadening of the corresponding diffraction line. Actually, both kinds of strain are usually superimposed in plastically deformed crystalline materials, and diffraction lines are both shifted and broadened because not only do the plane spacing vary from grain to grain but also mean value differ from that of the un-deformed metal.

Figure 15—Classic residual stress profile for surface treated material [51]
Shot peened material also show a shift of the diffraction line by residual stress and a broadening of the corresponding diffraction line by micro-strain and grain size due to cold working on the near surface produced by shot peening [57].

**Residual Stress Measurement** [57] – The biaxial surface stress field defined by the principal residual and/or applied stresses, $\sigma_1$ and $\sigma_2$, with no stress normal to surface is illustrated in Figure 16. The stress to be determined is, $\sigma_\phi$, lying in the plane of the surface at the angle, $\phi$, to the maximum principal stress, $\sigma_1$. The direction of measurement is determined by the plane of diffraction. The stress in any direction (for any angle, $\phi$) can be determined by rotating the specimen in the x-ray beam. If the stress is measured in at least three directions, the principal stresses and their orientation can be calculated.

![Figure 16–Plane Stress at a Free Surface [57]](image)

Consider the strain vector, $\varepsilon_{\psi\phi}$, lying in the plane defined by the surface normal and the stress, $\sigma_\phi$, to be determined. $\varepsilon_{\psi\phi}$ is at an angle $\psi$, to the surface normal, and can be expressed in terms of the stress of interest and the sum of the principal stresses as,
A typical metallic sample will consist of a large number of small grain or crystals, nominally randomly oriented. The crystal lattice consists of planes of atoms identified by their Miller Indices, \((hkl)\). The only Crystals which diffract x-ray are those which are properly oriented relative to the incident and diffracted x-ray beam to satisfy Bragg’s Law,

\[
n\lambda = 2d \cdot \sin \theta \quad (26)
\]

Where \(\lambda\) is the x-ray wavelength, \(n\) is an integer, \(\theta\) is the diffraction angle, and \(d\) is the lattice spacing. XRD can be used to selectively measure the lattice spacing of only those crystals of a selected phase which have a specific orientation relative to the sample surface by measuring \(\theta\) and calculating \(d\) from Equation 22. The lattice spacing can be determined for any orientation, \(\psi\), relative to the sample surface by merely rotation the specimen. It can be seen intuitively that if is a tensile stress, the spacing between lattices planes parallel to the surface will be reduced by a Poisson’s ration contraction, while the spacing of planes tilted into the direction of the tensile stress will be expanded. If we express the strain in terms of the crystal lattice spacing

\[
\epsilon_{\phi\psi} = \frac{1 + \nu}{E} \cdot \sigma_{\phi} \sin^2 \Psi - \left(\frac{\nu}{E}\right) \cdot (\sigma_{\psi} + \sigma_{\zeta}) \quad (25)
\]
Where \( d_0 \) is the stress free lattice spacing, our “strain gage” become the lattice spacing measured in the direction \( \theta, \psi \). Substitution Equation 23 into 21 and rearranging, the lattice spacing measured in any orientation can be expressed as a function of the stresses present in the sample and the elastic constants in the (hkl) crystallographic direction used for stress measurement.

\[
d(\phi, \Psi) = \left(1 + \frac{v}{E}\right)_{hkl} \cdot \sigma_{\phi} \cdot d_0 \sin^2 \Psi - \left(\frac{v}{E}\right) \cdot d_0 \cdot (\sigma_1 + \sigma_2) + d_0 \quad (28)
\]

**Line broadening and Cold Work** - When metallic material is cold worked by shot peening, the crystals are severely plastically deformed. The non-uniformity of this plastic deformation with depth causes the compressive macroscopic residual stresses produced by shot peening. As the crystal are deformed, lattice defect and dislocation tangles develop, producing micro-strain (strain over the dimensions on the order of the crystal lattice) and a reduction in the crystallite size (the perfect region within the crystals which are free of defects). Both increase in micro-strain and the reduction in crystallite size cause broadening of the diffraction peak used for measuring the macroscopic residual stress. This line broadening information can be used to quantify the degree to which the material has been plastically deformed by the shot peening process [57]. The percent cold work produced by shot peening can be calculated by using the half peak-breadth. It can be calculated from the width of the peak used for residual stress in term
of comparison between shot peened and cold worked sample, which percent cold work can be known.

### 2.2.4 Comparison of Compressive Residual Stress Distribution

Compressive residual stress distribution and cold work distribution by LPB, LSP and shot peening in IN 718 are illustrated in Figure 17 [58]. Low plasticity burnishing (LPB) produces deep depth and magnitude of compressive residual stress, a smooth, mirror-like surface and minimal cold working(<5%). LSP also produces comparable deep depth and magnitude of compressive residual stresses with less cold work. Single shock LSP can produce high compression with less than 1% cold work. To achieve maximum depths of compression, multiple laser shock cycles are required. Because the cold work is accumulative, multiple LSP shock cycles used achieve depths of compression exceeding 1 mm may produce an accumulation of 5-7% cold work. Shot peening produces from 10% to 50% cold work, more than grinding, machining, or other common surface finishing processes. Cold work is accumulative, and repeated applications of shot peening can produce even more than 50% cold work. Both the depth and degree of cold working increase with peening intensity, with the most severe cold working at the surface. Surface compression often decreases during shot peening of
work hardening materials as the yield strength of the surface increases with continued cold working [58, 59].

Therefore, Compressive residual stresses distribution in LSP and LPB is much higher magnitude and deeper relative to Shot Peening, and Cold work distribution in LSP and LPB is much less than Shot Peening.

![PERPENDICULAR RESIDUAL STRESS DISTRIBUTION](image)

![PERCENT COLD WORK DISTRIBUTION](image)

*Figure 17–Subsurface residual stress and cold work distributions produced by shot peening (8A), LSP (3X) and two levels of LPB in IN718 [58].*
2.2.5 Shot peening effects on the Microstructure

Shot peening is basically mechanical surface treatments so that plastic deformation in near surface regions leads to the formation of compressive residual stresses and severe microstructural alterations. Therefore, many materials will also increase in surface hardness due to the cold working effect of shot peening. The most important result of cold working, which accompanies this increase in the number of defects, is strain hardening, which is the increase in a metal with deformation. The mechanism of strain hardening certainly involves interactions among dislocations, the densities of which have been markedly increased.

Surface hardness for shot peened 7075 and A356 aluminum alloys are shown in Figure 18, shot peening results in twice the surface hardness increase as compared with laser peening [55].

![Figure 18](image)

*Figure 18—Vickers hardness measurements with a 25g load on A356-T6 and 7075-T7351 aluminum alloys treated by laser peening and shot peening respectively [55]*
Figure 19—TEM micrographs of deep rolled Ti–6Al–4V directly at the surface (left) and in the subsurface region in a depth of roughly 5 μm (right) [59]

In the near surface region, the microstructure is highly deformed due to cold work by shot peening. Figure 19 is illustrating microstructure change that average α grain size reduction of Ti–6Al–4V alloy was reported from 10μm to about 50nm after deep rolling directly at the surface (Figure 19, left). The subsurface exhibits high dislocation densities (Fig. 18, right) [59].

Shot peening can induce compressive residual stress fields and work-hardened microstructure in the surface layer, which then influences the precipitation process in Inconel 718. During aging, different microstructures may then form between the shot peened layer and the matrix [60].

Dayong Cai et al [60] studied shot peened Inconel 718. They preceded shot peening process in two step way to obtain a sufficient shot peening layer. After that, aging treatments were carried out at 700°C.
They found the residual stress at the surface is compressive, and the residual stress value increases with depth to a peak value and then decreases. Thermal relaxation effects on the residual stress after shot peening are shown in Figure 20(b). As aging time increases, the residual stress gradually decreases and almost disappears after 24 h. They also studied precipitation kinetic of $\gamma''$ phase shown in figure 21. During the beginning stage of aging, the amount of $\gamma''$ in the shot-peened layer is almost the same as that in the matrix. As aging proceeds, the difference in the amounts of the $\gamma''$ phase between the skin and the core increases remarkably. A near-equilibrium state of the precipitation process then tends to be reached, first for the skin. Meanwhile the amount of the $\gamma''$ phase in the core increases slowly. It shows that high degree of cold work introduced by shot peening may accelerate precipitation of the $\gamma''$ phase.
Figure 21—Precipitation kinetics of $\gamma''$ phase in shot-peened Inconel 718 during aging at 700 °C [60]

It is well known that the shot peened layer shows high density of dislocation in the deformed microstructure. W.C. Liu et al [61, 62] studied the influence of cold work on the precipitation kinetic of $\gamma''$. They found that the precipitation of the $\gamma''$ phase was greatly promoted at different temperature after shot peening. They interpreted that cold rolling creates not only dislocation but also excess vacancies. The latter can be attracted to Nb atoms to segregate to vacancy absorbing defects. Since local Nb segregation is increased, it can be expected the cold working may promote $\gamma''$ precipitation in the shot peened layer.

A. Turnbull et al [63] studied shot peening effect on the fatigue crack resistance of waspaloy. They found shot peening increase the hardness of the surface and near-surface layer and the affected layer is approximately 1mm. the hardness measured at the
surface is lower than the maximum hardness. Since dislocations generated in layer close to the surface are annihilated by their passage to the free surface with a subsequent relaxation of strain, the maximum hardness occurs about 50mm below. Increasing shot peening intensity increases the hardness and depth of the peened layer.

2.2.6 Comparison of Thermal Relaxation

The rate and amount of stress relaxation for 10A, 30% cold worked surface of shot peened Inconel 718 are shown in Figures 22(a) for exposures to 525°C. The surface residual stress produced by shot peening has been shown to relax rapidly in the first 60 minutes and then relax with time and temperature. The residual stress distributions shown in Figures 22(b) reveal virtually no thermal stress relaxation in the LSP processed Inconel 718, which was cold worked only 3 to 6%, at any depth or exposure time at temperature 525°C[59].
Residual stress and percent cold work depth profiles for 8A, 57 % cold worked surface of shot peened and LPB processed (<5% cold work) IN718 are shown in Figure 23 before and after exposure to 525°C and 600°C for 100 hr. The residual stresses from shot peening relax almost completely at the near surface where the material is highly cold worked. However, deeper and less cold worked inside region still retain compressive residual stress after exposure. The residual stress for LPB process show almost same depth and a little less magnitude [53].
Therefore, thermal stability is dependent on percent cold work and attributed to a reduced dislocation density and correspondingly limited dislocation annihilation at elevated temperatures, thus eliminating a mechanism for relaxation.

![Figure 23](image.png)

**Figure 23**—Thermal stresses relaxation in shot peened and low plasticity burnishing Inconel718 at 525°C and 600°C for 100hrs [53].

### 2.2.7 Comparison of fatigue properties

The bending fatigue properties of notched 7075-T7351 aluminum alloy specimens for shot peening and laser peening are compared in Figure 24(a). Shot peening provided an 11% increase in the fatigue strength at $10^7$ cycles, while laser shock peening provided a 22% increase, as compared with the untreated specimens. This improvement was explained by the greater depth and magnitude of the residual compressive stress fields induced by laser peening as compared with shot peening [55].
The S-N curves for the shot peened and low level LPB processed IN 718 after exposure to 600°C for 10 hours are shown in Figure 24(b). After 600°C for 10h, low LPB provides substantial improvement in fatigue life relative to 10A shot peening, especially at higher stress levels. The low LPB fatigue life at 800 MPa (116 ksi) is five times the 10A shot peened life [58]. These results indicate that LSP and LPB containing high magnitude and depth of compressive residual stresses, thus less dislocation density produced by cold work, improve fatigue life relative to shot peening.

Figure 24—(a) $S_{\text{max}}$–$N$ curves for untreated, shot-peened and laser-peened 7075-T7351 alloys (b) Inconel 718, 4 point bend, R=0, 30Hz, RT [58].
CHAPTER 3: MATERIALS AND EXPERIMENTAL PROCEDURE

3.1 MATERIALS

3.1.1 INCONEL 718

Inconel 718 is hardened by the precipitation of secondary phases (e.g. gamma prime and gamma double-prime) into the metal matrix. Inconel 718 contains significant amounts of iron, niobium, and molybdenum along with lesser amounts of aluminum and titanium. It combines corrosion resistance and high strength with outstanding weldability, including resistance to postweld cracking. The alloy has excellent creep-rupture strength at temperatures up to 700°C. Used in gas turbines, rocket motors, spacecraft, nuclear reactors, pumps, and tooling.

Table 1 – Nominal Chemical Compositions (in Wt.%). of IN718

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Ni</th>
<th>Fe</th>
<th>Cr</th>
<th>Ti</th>
<th>Nb</th>
<th>Mo</th>
<th>Co</th>
<th>Al</th>
<th>Si</th>
<th>C</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td>IN 718</td>
<td>52.5</td>
<td>19.0</td>
<td>19.0</td>
<td>0.09</td>
<td>5.3</td>
<td>3.1</td>
<td>–</td>
<td>0.6</td>
<td>0.21</td>
<td>0.04</td>
<td>0.004</td>
</tr>
</tbody>
</table>

3.1.2 WASPALOY

Waspaloy is a nickel base, age hardenable superalloy with excellent high temperature strength and good corrosion resistance, notably to oxidation, at service temperatures up to 650°C for critical rotating applications, and up to 870°C for other, less demanding, applications. The alloy’s high-temperature strength is derived from its
solid solution strengthening elements, molybdenum, cobalt and chromium, and its age hardening elements, aluminum and titanium. Its strength and stability ranges are higher than those typically available for alloy 718. Waspaloy is used for gas turbine engine components that call for considerable strength and corrosion resistance at high operating temperatures. Current and potential applications include compressor and rotor discs, shafts, spacers, seals, rings and casings, fasteners and other miscellaneous engine hardware, airframe assemblies and missile systems. The alloy’s oxidation resistance is good under conditions of frequent thermal cycling, and in continuous exposure to temperatures up to 1038°C.

Table 2—Nominal Chemical Compositions (in Wt.% of Waspaloy.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Ni</th>
<th>Fe</th>
<th>Cr</th>
<th>Ti</th>
<th>Nb</th>
<th>Mo</th>
<th>Co</th>
<th>Al</th>
<th>Si</th>
<th>C</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Waspaloy</td>
<td>54.0</td>
<td>2.0</td>
<td>19.5</td>
<td>3.0</td>
<td>–</td>
<td>4.25</td>
<td>13.5</td>
<td>1.4</td>
<td>0.75</td>
<td>0.07</td>
<td>0.006</td>
</tr>
</tbody>
</table>

3.1.3 INCONEL 740

Inconel 740 is a nickel-chromium-cobalt alloy developed by Special Metals Corporation. Inconel 740 primarily derives its strength from the precipitation of high volume fraction of \(\gamma'\) precipitates and is additionally solid solution strengthened by the addition of Co. It exhibits excellent resistance to high temperature corrosion due to the effects of chromium. With its high content of nickel and low level of iron, alloy 740
resists high temperature attack by halides as well. Potential applications include advanced power production boiler tubes and diesel engine exhaust valves. In ultra-supercritical (USC) power plants, steam conditions up to 37.5 MPa and 700°C will be encountered and this sets a higher temperature requirement for the hottest sections to be approaching 740-770°C. This alloy was developed to resist coal ash corrosion up to 700°C, and to resist creep-rupture for 100,000 h at 100 MPa (for component life >20 years). The chemical composition of Inconel 740 is shown in Table 3.

| Table 3—Nominal Chemical Compositions (in Wt.%) of IN 740 |
|-----------------|------|------|------|------|------|------|------|------|------|------|
|                | Ni   | Fe   | Cr   | Ti   | Nb   | Mo   | Co   | Al   | Si   | Mn   | C    |
| IN 740         | Balance | 0.7 | 25   | 1.8  | 2    | 0.5  | 20   | 0.9  | 0.5  | 0.3  | 0.03 |

3.2 SAMPLE PREPARATION

The Nickel-base superalloys IN718 and Waspaloy were obtained from plate material as pieces measuring approximately 25 mm x 30 mm x (6-10) mm thick that had been cold-rolled from 0 to 50% reduction in thickness. Three sets of thin strips measuring ~25 mm long x ~5 mm wide x ~0.5 mm thick were sectioned along both orientations (parallel-short and perpendicular-long to rolling direction) of the larger pieces using an Isomet 5000 precision diamond saw for electrical conductivity/resistivity and microhardness measurements and microstructure analysis.
Shot-peened samples of both alloys were also studied. For this purpose, sections were prepared from plates of both alloys that had been shot-peened to Almen intensities of 4A, 8A, 12A and 16A for hardness measurements, annealing treatments and microstructure analysis. Small sections cut from the shot-peened material of both alloys were encapsulated in evacuated and Argon back-filled quartz tubes and then annealed at 900 and 700°C for times from 1 to 25h and subsequently quenched in water while breaking the capsules.

Room temperature electrical resistivity measurements of the cold worked (CW) materials of both alloys were made by mechanically placing the thin, sectioned sample across a pair of calibrated knife edges, and connecting the ends to a precisely controlled current source. With a controlled current of ~3A passing, the voltage drop across the knife edges were measured using an accurate digital multimeter; the resistivity/conductivity was then obtained using the standard expression:

\[
\text{Resistivity } \rho = \frac{V}{I} \times \frac{A}{L} \quad \text{Conductivity } \sigma = \frac{1}{\rho} \quad (29)
\]

where, \( A \) is the sample cross-sectional area (width \( \times \) thickness), \( L (=10 \text{ mm}) \) is the sample length between the knife edges, \( V \) is the voltage drop across \( L \) and \( I \) is the current passed through the sample. Microhardness measurements of the cold worked, as well as shot peened materials were made on cut cross-section samples that had been
mounted in epoxy and then polished using a Leco tester under a load of 1kg held for 10 s. At least ten measurements were made on each sample and the average computed

The microstructures of the same cold-worked and shot-peened samples were characterized principally by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The cold-worked samples were mounted in epoxy parallel to their broad faces, whereas the shot-peened sections (annealed) were mounted in cross-section. Both sets of samples were polished and etched using a reagent composed of 50 ml HCl + 2 ml H₂O₂. Both unetched and etched samples were observed in an FEI XL-30 ESEM utilizing secondary electron (SE) and back scatter electron (BSE) imaging, and photographs digitally recorded at appropriate magnifications. Energy dispersive x-ray spectroscopy (EDS) analysis was performed to ascertain the chemical compositions of phases present in the samples using an EDAX Genesis 4000 thin-window EDS system attached to the XL-30 SEM. Orientation imaging microscopy (OIM)/electron backscatter diffraction (EBSD) observations of polished sections were also conducted on a few samples, using a TSL 4000 system attached to the XL-30 SEM, to ascertain texture. Thin foils for transmission electron microscopy (TEM) were prepared by punching 3-mm discs from the cold-worked sections polished to a thickness of ~150 µm, followed by twin-jet electro polishing of the 3-mm discs to perforation in a
Fischione unit using a solution of 10% perchloric acid in ethanol at -30°C. The foils were observed in a FEI CM20 TEM operated at 200 kV and photographs recorded under bright field (BF), dark field (DF) and selected area diffraction (SAD) modes.

Inconel 740 were solution-annealed at 1150°C (under this temperature only minor amount of carbides remain in the alloy in addition to the matrix; both the γ’ and η phases are completely dissolved) for 45 minutes in air and then water quenched. Subsequently aging of samples was performed at 3 different temperatures of interest: 700, 750 and 800°C for 100, 500, 1000 and 3000 hours, with those aged for 100, 1000 and 3000 hours being in the form of standard tensile bars, from a series of mechanical tests that were performed at Oak Ridge National Laboratory after the aging. Thus Stress Rupture tests for 700, 750, and 800°C were also performed at Oak Ridge National Laboratory. The samples conditions are listed in Table 4.

The samples that have undergone tensile testing and Stress rupture tests, a thin layer of the fractured tip containing the fracture surface was cut off, and subsequent slices of materials from the necked regions were sectioned for thin foils of Transmission Electron Microscopy (TEM) study. The illustration of these sampling areas is sketched in Figure 25.
### Table 4—Stress-Rupture Testing of Inconel 740

Stress Rupture Testing of INCONEL740 aged 16 hrs, 800°C, Ar

<table>
<thead>
<tr>
<th>Spec #</th>
<th>Product Form (Heat #)</th>
<th>Solution Annealing Temp.(°C)</th>
<th>TEST #</th>
<th>Stress (MPa)</th>
<th>Temp. (°C)</th>
<th>Life (hrs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>R740-04</td>
<td>Tube (CLH4633)</td>
<td>1190</td>
<td>30472</td>
<td>480</td>
<td>700</td>
<td>392.6</td>
</tr>
<tr>
<td>R740-05</td>
<td>Tube (CLH4633)</td>
<td>1190</td>
<td>30492</td>
<td>420</td>
<td>700</td>
<td>1082.2</td>
</tr>
<tr>
<td>R740-06</td>
<td>Tube (CLH4633)</td>
<td>1190</td>
<td>30507</td>
<td>340</td>
<td>700</td>
<td>5227.4</td>
</tr>
<tr>
<td>R740-07</td>
<td>Tube (CLH4633)</td>
<td>1190</td>
<td>30464</td>
<td>370</td>
<td>750</td>
<td>289.3</td>
</tr>
<tr>
<td>R740-08</td>
<td>Tube (CLH4633)</td>
<td>1190</td>
<td>30467</td>
<td>300</td>
<td>750</td>
<td>984.5</td>
</tr>
<tr>
<td>R740-09</td>
<td>Tube (CLH4633)</td>
<td>1190</td>
<td>30509</td>
<td>220</td>
<td>750</td>
<td>7201.6</td>
</tr>
<tr>
<td>R740-10</td>
<td>Tube (CLH4633)</td>
<td>1190</td>
<td>30449</td>
<td>250</td>
<td>800</td>
<td>279.2</td>
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<tr>
<td>R740-11</td>
<td>Tube (CLH4633)</td>
<td>1190</td>
<td>30439</td>
<td>180</td>
<td>800</td>
<td>1491.1</td>
</tr>
<tr>
<td>R740-12</td>
<td>Tube (CLH4633)</td>
<td>1190</td>
<td>30476</td>
<td>130</td>
<td>800</td>
<td>6882.7</td>
</tr>
</tbody>
</table>

**Figure 25—Illustration of sample preparation for tested bars**

Slice used for TEM thin foil
Thin foils for transmission electron microscopy (TEM) were prepared by punching 3-mm discs from sections polished to a thickness of ~150 μm, followed by twin-jet electro polishing of the 3-mm discs to perforation in a Fischione unit using a solution of 10% perchloric acid in ethanol at -30°C. The foils were observed in a FEI CM20 TEM operated at 200 kV and photographs recorded under bright field (BF), dark field (DF) and selected area diffraction (SAD) modes.
CHAPTER 4: RESULTS AND DISCUSSION

4.1 Shot peened Nickel-Base Superalloys

The results of this study are presented in the following of sub-sections in the form of plots, tables and micrographs. The results of the cold-worked samples are covered first, followed by those from the shot-peened samples, and then discussed in the last sub-section.

4.1.1 Electrical Resistivity/Conductivity of Cold-Worked Samples

The results of electrical resistivity and conductivity of non-cold worked and cold worked (10-50%) IN718 and Waspaloy samples are shown in Table 5 and plotted in Figure 26. As can be seen, the changes in resistivity/conductivity with % cold work are not so distinct in IN718, although an apparent tendency toward a slight decrease in conductivity with increasing amount of cold work is discernible. On the other hand, in Waspaloy, cold working leads to a small increase in electrical conductivity (~10% at 50% reduction).
Table 5—Average room temperature electrical resistivity and conductivity as a function of % cold work of IN718 and Waspaloy.

**IN718**

<table>
<thead>
<tr>
<th>% Cold Work</th>
<th>Resistivity (µ-Ω-cm)</th>
<th>Conductivity (Ω-cm⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Absolute</td>
<td>Normalized</td>
</tr>
<tr>
<td>0.0</td>
<td>120.13</td>
<td>1.00</td>
</tr>
<tr>
<td>10.0</td>
<td>122.67</td>
<td>1.02</td>
</tr>
<tr>
<td>20.0</td>
<td>120.41</td>
<td>1.00</td>
</tr>
<tr>
<td>30.0</td>
<td>127.03</td>
<td>1.06</td>
</tr>
<tr>
<td>40.0</td>
<td>122.12</td>
<td>1.02</td>
</tr>
<tr>
<td>50.0</td>
<td>121.93</td>
<td>1.01</td>
</tr>
</tbody>
</table>

**Waspaloy**

<table>
<thead>
<tr>
<th>% Cold Work</th>
<th>Resistivity (µ-Ω-cm)</th>
<th>Conductivity (Ω-cm⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Absolute</td>
<td>Normalized</td>
</tr>
<tr>
<td>0.0</td>
<td>127.77</td>
<td>1.00</td>
</tr>
<tr>
<td>10.0</td>
<td>122.49</td>
<td>0.96</td>
</tr>
<tr>
<td>20.0</td>
<td>117.48</td>
<td>0.92</td>
</tr>
<tr>
<td>30.0</td>
<td>117.09</td>
<td>0.92</td>
</tr>
<tr>
<td>40.0</td>
<td>120.42</td>
<td>0.94</td>
</tr>
<tr>
<td>50.0</td>
<td>117.02</td>
<td>0.92</td>
</tr>
</tbody>
</table>
Figure 26—Room temperature electrical resistivity and conductivity (normalized) as a function of cold work of IN718 and Waspaloy.
4.1.2 Microhardness of Cold-Worked Samples

As mentioned in section 3, non-cold worked and cold-worked samples that had been sectioned along two orientations, parallel-short and perpendicular-long, were prepared metallographically and at least ten hardness measurements were made on each sample using a Leco tester and the average computed. The results are shown in Figure 27 (a,b) for IN718 and Waspaloy. As can be seen, there was a small difference in the hardness at each cold work level between the long and short orientations, with the individual values differing by about 2-5% from the average. In addition, in IN718, the hardness rises from the initial value of 218 HV quickly ~328 HV at the 10% cold work level and then increases somewhat more gradually at higher levels of cold work to ~408 HV at the 50% level. The Waspaloy samples also show a continuous increase in hardness with % cold work from the initial non-cold worked value of ~318 HV to ~443 HV at the 50% level; this increase is somewhat more gradual compared with IN718.
Figure 27—Variation of microhardness with % cold work of IN 718 and Waspaloy.
4.1.3 Microstructure of As-received, Non-Cold-Worked Samples

Depending on processing and heat treatment conditions, the microstructure of IN718 can exhibit four major types of precipitates in the $\gamma$ matrix, namely: (i) $\gamma'$ having a composition $\text{Ni}_3(\text{Ti,Al})$ and a cubic ($L1_2$) crystal structure, (ii) $\gamma''$ having a composition $\text{Ni}_3(\text{Al,Ti,Nb})$ and a bct ($D0_{22}$) crystal structure, (iii) $\eta$-$\text{Ni}_3\text{Ti}$ with a hexagonal structure and (iv) $\delta$ having a composition $\text{Ni}_3\text{Nb}$ and an orthorhombic ($D0_a$) crystal structure. In addition, carbides are also present to a minor extent. The microstructure of Waspaloy after typical aging treatments is composed of $\gamma'$ precipitates in a $\gamma$ matrix, together with a small volume fraction of carbides.

Low magnification SE micrographs, EBSD/OIM results and higher magnification SE micrographs with relevant EDS spectra recorded from the non-cold-worked IN718 and Waspaloy samples are shown in sequence for each alloy in Figures 28-30 and 31-32, respectively. The low magnification SE micrographs in Figures 28 and 31 reveal that the IN718 had an average grain size of ~20 $\mu$m, whereas that of Waspaloy was larger, being about 30-50 $\mu$m. The grain sizes observed in the OIM images, Figures 29 and 32 are generally consistent with those measured in the companion SE images, with that of the Waspaloy again being coarser than that of the IN 718. The grain size numbers obtained from the OIM analysis, which are presented in the
tables and plots, however, appear to be much smaller than those in the SEM and OIM images, presumably because the software also includes twins and other microstructural features, which have much smaller dimensions than the grain size. Analysis of the pole figures presented in Figures 29 and 32 indicated the presence of mostly \{001\}<100> and \{001\}<110> texture elements in the IN 718 material and a small deviation from these in the Waspaloy sample.

The low (Figure 28) and high magnification (Figure 30(a-c)) SE images from IN718 also reveal the presence of second-phase particles in the form of Widmanstatten platelets in several orientations within a grain, which were determined by TEM to be $\eta$-Ni$_3$Ti, as well as bright blocky/round particles within grains and along the grain boundaries, which were identified by EDS to be $\delta$-Ni$_3$Nb (Figure 30(d)). Etch pits in the form of more or less cuboidal/triangular-shaped, dark gray contrast features can also be seen within the grains (Figure 30(a,c)). The high magnification SE images of Waspaloy (Figure 33(a-c)) reveal the presence of a few coarse second-phase particles (carbides) within grains and along grain boundaries, which were determined by EDS analysis (Figure 33(d)) to be (Ti,Mo) carbides; in addition, fine, spheroidal, 60-200 nm diameter $\gamma'$-Ni$_3$(Al,Ti) precipitates, appearing in grayish contrast within the grains, are also present.
Figure 28—Low magnification SE micrographs with grain size markers of etched non-cold worked IN718 sample.
Figure 29—OIM map, inverse pole figure, grain size and pole figure/texture data from non-cold worked IN718
Figure 30—Higher magnification SE micrographs (a-c) of etched Non-Cold Worked IN718 sample and (d) typical EDS spectrum from blocky and/or grain boundary precipitates, suggesting these are $\delta$-Ni$_3$Nb.
Figure 31—Low magnification SE micrographs with grain size markers of etched Non-Cold Worked Waspaloy sample.
Figure 32—OIM map, inverse pole figure, grain size and pole figure/texture data from non-cold worked Waspaloy sample.
Figure 33—(a-c) Higher magnification SE micrographs showing microstructure of etched Non-Cold Worked Waspaloy sample. Coarse grain boundary and intragrain particles can be seen. (d) EDS spectra from indicated particle in “a,” “b” indicating these are (Ti,Mo)C. Spheroidal γ' – [Ni₃(Al,Ti)] precipitates in dark gray contrast can also be seen in “b” and “c.”
TEM micrographs of the 0% CW IN718 sample, Figure 34(a,b), revealed the presence of platelets of $\eta$-Ni$_3$Ti in several orientations, as well as discrete particles of $\delta$-Ni$_3$Nb. SAD patterns recorded from the $\gamma$ matrix region in between the platelets and particles reveals superlattice reflections at the \{100\} and \{1\ ½ 0\} positions (Figure 34(c,d)), which are associated with the presence of $\gamma''$ precipitates. Figure 35 shows another example of $\delta$-Ni$_3$Nb (Figure 35(a)), as confirmed by the [011] selected area diffraction (SAD) pattern (Figure 35(b)), together with superlattice reflections at \{100\} and \{0-11\} positions associated with the presence of $\gamma''$ precipitates. A dark field (DF) image obtained from the indicated $\delta$ and $\gamma''$ superlattice reflections, Figure 35(c), reveals the plates of $\delta$-Ni$_3$Nb and ultrafine, nanoscale precipitates of $\gamma''$. The chemical compositions of various phases ($\gamma$, $\eta$-Ni$_3$Ti platelets and $\delta$ particles) present in the microstructure, determined by standardless analysis of EDS spectra, is shown in Table 6. The results reveal that the $\delta$ particles are highly enriched in Nb (~25 at.%), whereas the $\eta$ platelets contain modest levels of Nb, Al and Ti and high concentrations of Cr and Fe.
Figure 34—BF TEM micrographs showing microstructure of 0% CW IN718 sample. (a) Plate-shaped \(\eta\)-Ni\(_3\)Ti precipitates in several orientations and (b) coarse particles of \(\delta\)-Ni\(_3\)Nb can be seen. (c) [001] and (d) [011] SAD patterns from the \(\gamma\) matrix regions revealing superlattice reflections at the \{100\} and \{1\(\frac{1}{2}\)0\} positions indicating the presence of ultrafine \(\gamma''\) precipitates.

Table 6—Typical Chemical Compositions (in At.%) of Phases in Non-Cold Worked IN718.

<table>
<thead>
<tr>
<th>Phase</th>
<th>Al</th>
<th>Nb</th>
<th>Ti</th>
<th>Cr</th>
<th>Fe</th>
<th>Co</th>
<th>Ni</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matrix (\gamma)</td>
<td>1.80</td>
<td>2.90</td>
<td>1.20</td>
<td>22.50</td>
<td>20.50</td>
<td>0.80</td>
<td>47.90</td>
<td>2.40</td>
</tr>
<tr>
<td>Plate (\eta)-Ni(_3)Ti</td>
<td>1.20</td>
<td>7.30</td>
<td>1.90</td>
<td>19.10</td>
<td>17.10</td>
<td>0.80</td>
<td>50.70</td>
<td>2.00</td>
</tr>
<tr>
<td>Round (\delta)</td>
<td>0.00</td>
<td>24.60</td>
<td>3.40</td>
<td>02.20</td>
<td>03.50</td>
<td>0.30</td>
<td>64.80</td>
<td>1.30</td>
</tr>
</tbody>
</table>
Figure 35—TEM micrographs showing microstructure of 0% CW IN718 sample. (a) BF micrograph showing δ-Ni₃Nb, and γʺ precipitates in several orientations; (b) [011] SAD pattern showing δ-Ni₃Nb and γʺ superlattice reflections; and (c) DF micrograph from encircled reflections in “b” revealing the δ phase, and nanoscale γʺ precipitates.
A TEM micrograph (Figure 36(a)) recorded from the 0% CW Waspaloy sample revealed the presence of fine, spheroidal, ~200-nm diameter bright precipitates of $\gamma'$, together with round, dark particles, which were determined to be an artifact originating from electropolishing. The $\gamma'$ precipitates produce superlattice reflections at the \{100\} positions in the SAD pattern (Figure 36(b)). Analysis of EDS spectra acquired from the various phases, Table 7, revealed that the bright $\gamma'$ precipitates contain high amounts of Ni and moderate levels of Al and Ti, whereas the matrix $\gamma$ has lower levels of Al and Ti and higher levels of Mo, Cr and Co.

Figure 36—(a) BF TEM micrograph and (b) [001] SAD pattern showing microstructure of 0% CW Waspaloy sample. Spheroidal $\gamma'$ (light) precipitates are present in (a), which produce the superlattice reflections at the \{100\} and \{110\} positions in (b). The dark particles are artifacts from electropolishing.

Table 7—Typical Chemical Compositions (in At.%) of Phases in Waspaloy.

<table>
<thead>
<tr>
<th>Phase</th>
<th>Al</th>
<th>Mo</th>
<th>Ti</th>
<th>Cr</th>
<th>Co</th>
<th>Ni</th>
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</thead>
<tbody>
<tr>
<td>$\gamma$</td>
<td>2.40</td>
<td>4.50</td>
<td>3.20</td>
<td>23.90</td>
<td>12.30</td>
<td>53.70</td>
</tr>
<tr>
<td>$\gamma'$</td>
<td>7.20</td>
<td>1.10</td>
<td>12.80</td>
<td>5.20</td>
<td>6.30</td>
<td>67.40</td>
</tr>
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</table>
4.1.4 Microstructure of Cold Worked Samples

The microstructures of the 10, 20, 30, 40 and 50% cold worked IN718 and Waspaloy samples were observed by SEM and TEM. The results are presented below in sequence for each alloy.

BSE/SE micrographs and related EDS spectra from the 10, 20, 30, 40 and 50% CW IN718 samples are shown in the series of micrographs in Figures 36 through 39, respectively. The cold worked samples generally showed more or less the same microstructure, except that the blocky $\delta$-Ni$_3$Nb precipitates became more uniformly distributed and their volume fraction appeared to be higher and the thin, intragrain $\eta$-Ni$_3$Ti platelets showed evidence for deformation (bending) and occasional fragmentation, with the degree of bending increasing with an increase in the amount of cold work (Figures 37-41). Coarse (Nb,Ti)C and(Ti,Nb)(N,C) particles, identified by EDS (Figures 37(c), 37(d), respectively) were also observed. The typical chemical compositions of $\eta$-Ni$_3$Ti, $\eta$-Ni$_3$Nb and the $\gamma$ matrix present in the SE images of the cold-worked IN718 samples are shown in Tables 8-10, respectively.
Figure 37—(a,b) BSE micrographs of unetched, 10% cold worked IN718 sample and (c), (d) EDS spectra identifying bright, coarse particle near the middle of the micrograph in “a” to be (Nb,Ti)C and the dark particles in “a” to be (Ti,Nb)(N,C).

Figure 38—(a) Low magnification and (b) higher magnification BSE micrograph of 20% Cold-Worked IN 718.
Figure 39—SE micrographs showing microstructure of 30% CW IN718 sample.

Figure 40—(a) Low magnification and (b) higher magnification BSE micrograph of 40% Cold-Worked IN 718.

Figure 41—(a) Low magnification and (b) higher magnification BSE micrograph of 50% Cold-Worked IN 718.
Table 8—Typical Chemical Composition (At %) of \( \eta - \text{Ni}_3(Ti,Nb) \) precipitates in Cold Worked IN718

<table>
<thead>
<tr>
<th>% C.W</th>
<th>Al</th>
<th>Nb</th>
<th>Ti</th>
<th>Cr</th>
<th>Fe</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>2.27</td>
<td>6.60</td>
<td>0.91</td>
<td>16.49</td>
<td>17.02</td>
<td>56.71</td>
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<tr>
<td>20</td>
<td>2.66</td>
<td>6.86</td>
<td>2.68</td>
<td>14.04</td>
<td>13.07</td>
<td>60.69</td>
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<tr>
<td>30</td>
<td>1.29</td>
<td>5.79</td>
<td>1.97</td>
<td>16.98</td>
<td>16.52</td>
<td>57.45</td>
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<tr>
<td>40</td>
<td>1.58</td>
<td>6.26</td>
<td>1.87</td>
<td>17.36</td>
<td>17.15</td>
<td>55.77</td>
</tr>
<tr>
<td>50</td>
<td>1.99</td>
<td>6.85</td>
<td>2.55</td>
<td>15.27</td>
<td>14.69</td>
<td>58.64</td>
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</table>

Table 9—Typical Chemical Composition (At %) of \( \delta - \text{Ni}_3\text{Nb} \) phase in Cold Worked IN 718.

<table>
<thead>
<tr>
<th>% C.W</th>
<th>Al</th>
<th>Nb</th>
<th>Ti</th>
<th>Cr</th>
<th>Fe</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>1.41</td>
<td>17.44</td>
<td>2.34</td>
<td>8.09</td>
<td>7.98</td>
<td>62.73</td>
</tr>
<tr>
<td>20</td>
<td>2.08</td>
<td>16.39</td>
<td>3.76</td>
<td>4.80</td>
<td>5.38</td>
<td>67.59</td>
</tr>
<tr>
<td>30</td>
<td>1.51</td>
<td>17.75</td>
<td>3.68</td>
<td>4.21</td>
<td>5.81</td>
<td>67.04</td>
</tr>
<tr>
<td>40</td>
<td>0.92</td>
<td>17.68</td>
<td>3.63</td>
<td>5.24</td>
<td>6.50</td>
<td>66.04</td>
</tr>
<tr>
<td>50</td>
<td>0.32</td>
<td>18.77</td>
<td>3.47</td>
<td>4.55</td>
<td>4.60</td>
<td>68.28</td>
</tr>
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Table 10—Typical Chemical Composition (At %) of \( \gamma \) matrix in IN 718 obtained from 40% Cold Worked Sample.

<table>
<thead>
<tr>
<th>% C.W</th>
<th>Al</th>
<th>Nb</th>
<th>Ti</th>
<th>Cr</th>
<th>Fe</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>1.17</td>
<td>2.70</td>
<td>1.33</td>
<td>22.05</td>
<td>18.72</td>
<td>54.04</td>
</tr>
</tbody>
</table>
TEM observations of the 10% CW IN 718 sample revealed the presence of many dislocations, along with eta-Ni$_3$Ti plates that have been sheared by dislocations such that they appear bent (Figure 42(a,b)). No superlattice $\gamma''$ reflections could be seen in the SAD patterns recorded (Figure 42(c)), from the $\gamma$ matrix in this sample (unlike the situation in the non-cold worked sample, Figure 35(b)), suggesting that the shearing of these precipitates by dislocations leads to their dissolution and redistribution of alloying elements into the $\gamma$ matrix. This feature, in turn, appears to be partially responsible for the observed decrease in conductivity at the 10% cold work level [64]. TEM observations of the 20%, 30%, 40% and 50% cold worked samples, Figures 42 to 46, respectively, essentially revealed the same feature, i.e. dissolution of the $\gamma''$ precipitates into the $\gamma$ matrix, as indicated by the absence of reflections from these precipitates in the SAD patterns. The only other change observed was the increase in the density of dislocations and bending and break-up of the $\eta$-Ni$_3$Ti platelets with increasing amounts of cold work over this range.
Figure 42—TEM micrographs showing microstructure of 10% CW IN718 sample. (a), BF micrograph showing a bent/sheared \( \eta \)-Ni\(_3\)Ti platelet and many dislocations; (b) corresponding DF micrograph from \( \eta \)a reflection; and (c) \{011\} SAD pattern showing \( \eta \)-Ni\(_3\)Ti reflections, but no \( \gamma'' \) superlattice reflections.
Figure 43—TEM micrographs showing microstructure of 20% CW IN718 sample. (a) BF micrograph showing many dislocations; (b) [001] SAD pattern of γ matrix showing absence of γ' superlattice reflections.

Figure 44—TEM micrographs showing microstructure of 30% CW IN718 sample. (a) BF micrograph showing many dislocations in γ matrix; (b) BF micrograph showing a large δ particle; inset [011] SAD pattern of γ matrix, in which absence of γ' superlattice reflections can be noted.
Figure 45—TEM micrographs showing microstructure of 40% CW IN718 sample. (a) BF micrograph showing bent and broken platelets of η-Ni₃Ti γᵉ precipitates; (b), (c) BF, DF micrograph pair showing a high dislocation density in the γ matrix; and (c) [001] SAD pattern of γ matrix, in which absence of γᵉ superlattice reflections can be noted.
Figure 46—(a) BF and (b) weak beam dark field (WBDF) TEM micrographs showing a high dislocation density in the 50% CW IN 718 sample; (c) [001], (d) [011] SAD patterns showing absence of γ′ superlattice reflections; (e) BF micrograph showing sheared η-Ni₃Ti platelets.
BSE images of the unetched Waspaloy samples recorded at low magnification were essentially featureless and it was difficult to discern the presence of second phases within grains and changes in microstructure induced by cold work. The SE images of the cold-worked samples of Waspaloy (Figures 47-51) revealed the presence of (Ti,Nb)-carbides along the grain boundaries, coarse Ti-nitrides and fine, spheroidal $\gamma'$ precipitates within the grains. Generally, the SE images did not reveal substantial changes in the microstructure with increasing amounts of cold work, although some grain elongation could be seen at the higher levels. TEM observations of the cold worked Waspaloy samples revealed an increase in the dislocation density, often in planar arrays, with increasing amounts of cold work to fairly high levels in the 50% CW sample (Figures 52-55). The $\gamma'$ precipitates were still present in the cold worked samples, i.e., there was no dissolution, and a few dislocations were occasionally present within these precipitates.
Figure 47—SE micrographs of 10% CW Waspaloy sample. Note carbides along grain boundaries in (a) and fine γ’ precipitates and large Ti-nitride particle in (b).

Figure 48—SE micrographs of 20% CW Waspaloy sample.

Figure 49—SE micrographs of 30% CW Waspaloy sample.
Figure 50—SE micrographs of 40% CW Waspaloy sample.

Figure 51—SE micrographs of 50% CW Waspaloy sample.
Figure 52—TEM micrographs showing microstructure of 20% CW Waspaloy sample. (a) and (b) BF micrographs showing a few γ precipitates and many dislocations in γ matrix.

Figure 53—TEM micrographs showing microstructure of 30% CW Waspaloy sample. (a) and (b) BF micrographs showing a few γ precipitates and high dislocation density in γ matrix.
Figure 54—BF TEM micrographs showing microstructure of 40% CW Waspaloy sample. Note the high dislocation density in $\gamma$ matrix.

Figure 55—BF TEM micrographs showing microstructure of 50% CW Waspaloy sample. Note a few $\gamma$ precipitates and the high dislocation density in $\gamma$ matrix.
4.1.5 Microhardness of Shot-Peened Samples and Effect of Annealing

The change in microhardness as a function of distance from the surface of the samples of IN718 and Waspaloy that had been shot-peened to various Almen intensity levels (4A, 8A, 12A and 16A) were measured. The measurements were made on polished, cross-section samples. The results, Figures 56 and 57, show that shot peening leads to a significant increase in the near-surface hardness in both IN718 and Waspaloy, with the degree of hardening being greater, the greater the Almen intensity. The hardness is highest at the surface and then decreases with distance from the surface to a near-constant value around ~220-240 HV beyond ~175 µm from the surface for Almen 4A and 8A and beyond ~300 µm for Almen 12A and 16A. The initial decrease in hardness with distance from the surface is more pronounced in the IN718 samples compared with Waspaloy where the change is more gradual. The magnitude of the hardness increase in the near-surface regions with Almen intensity is also more pronounced in IN718 compared with Waspaloy, although the hardness of the latter at each Almen level is greater (Figure 57). The hardening is associated with the cold work/plastic strain introduced by shot peening, with the degree of cold work and, in turn, hardness diminishing with distance from the surface. The magnitude of this cold work and its penetration through the depth is greater at the higher 12A and 16A Almen
intensities. Comparison of the data in Figures 56 and 57 with those in Figure 57 showing the variation in hardness with % cold work indicates that the % cold work in the near-surface regions in the Almen 16A shot peened samples of both alloys approaches 70%. Furthermore, the data in Figures 56 and 57 and comments made above thereof suggests that the work hardening rate is greater in IN718 compared with Waspaloy, which is also consistent with the observed variations in hardness with % cold work in Figure 57.

![Variation of near-surface microhardness with Almen intensity of shot-peened IN718 and Waspalloy samples.](image)

Figure 56—Variation of near-surface microhardness with Almen intensity of shot-peened IN718 and Waspalloy samples.
Figure 57—Variation of microhardness with distance from surface in (a) IN718 and (b) Waspalloy samples shot-peened with various Almen intensity.
The effect of annealing treatments on the near-surface and through-the-depth microhardness of shot-peened samples was also studied. Cut samples of shot-peened IN718 and Waspaloy (various Almen intensities: 4A, 8A, 12A and 16A) were encapsulated in quartz under Argon and annealed individually at two different temperatures 900 and 700°C for 1, 5, 10 and 24h and water quenched and the microhardness measured on polished, cross-section samples.

The results of the variation of microhardness as a function of distance from the surface of Almen 4A, 8A and 12A, 16A IN718 samples annealed for various times at 900°C are shown in Figures 58(a,b) and 59(a,b), respectively. The results show that there is a large drop in the near-surface hardness within 1h of annealing in all the four samples compared with the as-shot peened condition upon annealing for longer times, the hardness attains a low and more or less constant value throughout the material (near-surface and through-thickness). These results show that the residual stresses and cold work present in the initial shot-peened material are lost pretty quickly on annealing, which is not surprising given that the annealing temperature of 900°C is quite high.

The results of hardness versus depth following annealing for times from 1-25h at 700°C of the IN718 samples initially shot-peened to Almen intensities of 4A, 8A and 12A, 16A are shown in the plots in Figures 60(a,b) and 61(a,b), respectively, whereas
plots showing the combined effect of Almen intensity and aging for 1 and 25h are displayed in Figure 62. As can be seen, the hardness of the near-surface and through-the-depth regions at each distance rises within 1 hour to values that are about 25-30% higher than those of the as-shot peened, un-annealed samples at the same distance (Figure 56). As in the case of the as-shot-peened samples, the hardness is highest at the surface and then decreases continuously with depth to a near constant value around ~300 HV at a distance of ~200-350 μm from the surface, depending on the Almen intensity (4A, 8A and 12A, 16A, respectively). This would suggest that the residual stress and cold work present in the initial shot-peened materials are not totally relaxed after the 700°C exposure and that additional changes in microstructure, namely, new precipitation of $\gamma'$ or $\gamma''$ has occurred during the annealing, leading to a higher hardness throughout. The fact that the hardness of the annealed sample far into the depth, where the effects due shot-peening should not be present, is higher than that in the un-annealed samples would suggest that new precipitation that presumably has occurred during the 700°C, 1h aging, makes an significant contribution to the overall hardness. With further increase in aging time (5, 10h), the hardness of both the near-surface and through-the-depth regions continues to increase over that of the 1h aging condition, with the difference between the values of the near-surface regions and the bulk becoming smaller,
such that after 25h, they are nearly the same and the highest amongst all the aging times. It should be noted, however, that the hardness of the near-surface regions are still slightly than those in the bulk in the Almen 12A and 16A samples after 25 h, which suggests that some of the high cold work present initially is still retained. A last point to note is that the hardness is the highest after 25h aging and the values are nearly the same (~430 HV) irrespective of the initial shot-peening intensity level (Figure 62(b)). This would suggest that most of the cold working produced by the shot-peening has been removed by this time (except in 12A and 16A, where some persists in the near-surface regions) and the high hardness throughout is caused by microstructural changes, i.e. new precipitation of $\gamma'$ or $\gamma''$ or both.
Figure 58—Variation of microhardness with distance from surface in shot-peened IN718 annealed for various times at 900°C; (a) Almen 4A and (b) Almen 8A.
Figure 59—Variation of microhardness with distance from surface in shot-peened IN718 annealed for various times at 900°C; (a) Almen 12A and (b) Almen 16A.
Figure 60—Variation of microhardness with distance from surface in shot-peened IN718 annealed for times from 1 to 25h at 700°C; (a) Almen 4A and (b) Almen 8A.
Figure 61—Variation of microhardness with distance from surface in shot-peened IN718 annealed for times from 1 to 25h at 700°C; (a) Almen 12A and (b) Almen 16A.
Figure 62—Variation of microhardness with distance from surface in IN718 shot-peened to various intensities (4A to 16A) and then annealed at 700°C for (a) 1h and (b) 25h. The hardness variation of the as-shot-peened 16A sample is included for comparison.
The changes in hardness with depth in the Waspaloy samples with annealing time at 700°C, shown in Figures 63 and 64, were somewhat different from the counterpart IN718 samples and also varied depending on the initial Almen intensity level. In the case of the Almen 4A and 8A samples, annealing at 700°C leads to an increase in the hardness at the surface and the near-surface regions to a depth of ~150 m at all aging times, with the values after 25h generally being higher than those at shorter times (Figure 63). This would suggest that additional precipitation of $\gamma'$ may have occurred during aging and that some of the cold work initially present is still retained and both of these give rise to the higher hardness in the surface/near-surface regions. On the other hand, in the Almen 12A and 16A samples, Figure 64, the hardness at the surface and near-surface regions to a depth of ~400 μm decreases with aging time at 700°C, with the values at all times being lower than those in the as-peened condition at equivalent distances, suggesting that some of the cold work present in the as-peened condition is lost upon annealing. Furthermore, the values after aging for 25h at the equivalent distances are higher than those at shorter times, which suggests that while some of the cold work present in the initial as-peened samples is still retained, additional precipitation of $\gamma'$ may have occurred at the longer aging times, leading to an increase in hardness over that at shorter times.
Figure 63—Variation of microhardness with distance from surface in shot-peened Waspaloy annealed for times from 1 to 25h at 700°C; (a) Almen 4A and (b) Almen 8A.
Figure 64—Variation of microhardness with distance from surface in shot-peened Waspaloy annealed for times from 1 to 25h at 700°C; (a) Almen 12A and (b) Almen 16A.
4.1.6 Microstructure of Shot-Peened and Annealed Samples

A limited set of Optical and SEM observations of the microstructure of the shot-peened samples of IN718 and Waspaloy that had been annealed at 700°C for 1h and 25h were made and are presented below.

The results for IN718 corresponding to the four Almen intensity levels for these two aging times are shown in the series of Optical and SEM micrographs in Figures 65-66 and 67-68, respectively. The observations of the 1h- and 25h-aged samples, Figures 65-68, revealed that the grain size in the near-surface regions was large and of similar size as that of the as-shot peened and non-cold worked samples, i.e., there was no evidence for recrystallization of the cold-worked structure present in the near-surface regions or formation of new grains. The presence of γ-Ni₃Ti platelets as part of a discontinuous reaction emanating from the grain boundaries could be seen in all the samples. Furthermore, high magnification SE micrographs revealed the presence of the ultrafine, nanoscale, disc/plate-shaped precipitates, presumably γ″, within the grains in the Almen 4A and 8A samples aged for 1 (Figure 65(c,f)) and 25h (Figure 66(c,f)). In the Almen 12A and 16A samples, ultrafine, nanoscale precipitates were also present within the grains, but these appeared spheroidal (Figures 67(c,f) and 68(c,f)) and presumably are γ′. It appears that the higher amount of initial cold work present in the
near-surface regions due to higher Almen intensity may have promoted the formation of $\gamma'$ rather than $\gamma''$. The possibility that the $\gamma''$ precipitates are still present, but too fine to be resolved by SEM, cannot, however, be excluded. Apart from these features, the presence of fine, round, bright particles, whose identity has not been determined and could be $\eta$-Ni$_3$Ti or $\delta$-Ni$_3$Nb, could also be noted in the near-surface regions of the Almen 12A and 16A samples aged for 25h (Figure 67(c,f)), Finally, it is clear that the increase in hardness over that of the as-shot peened condition with aging time in all the Almen 4A-16A samples is related to the precipitation of nanoscale $\gamma''$ and/or $\gamma'$ precipitates.
Figure 65—Optical and SEM micrographs of near-surface region of shot-peened IN718 annealed for 1h at 700°C. (a) is optical and (b), (c) are low and high magnification images of Almen 4A sample, whereas (d) is optical and (e), (f) are the counterpart images from the Almen 8A sample.
Figure 66—Optical and SEM micrographs of near-surface region of shot-peened IN718 annealed for 1h at 700°C. (a) is optical and (b), (c) are low and high magnification images of Almen 12A sample, whereas (d) is optical and (e), (f) are the counterpart images from the Almen 16A sample.
Figure 67—Optical and SEM micrographs of near-surface region of shot-peened IN718 annealed for 25h at 700°C. (a) is optical and (b), (c) are low and high magnification images of Almen 4A sample, whereas (d) is optical and (e), (f) are the counterpart images from the Almen 8A sample.
Figure 68–Optical and SEM micrographs of near-surface region of shot-peened IN718 annealed for 25h at 700°C. (a) is optical and (b), (c) are low and high magnification images of Almen 12A sample, whereas (d) is optical and (e), (f) are the counterpart images from the Almen 16A sample.
The results for Waspaloy corresponding to the four Almen intensity samples aged for 1h and 25h at 700°C are shown in the series of Optical and SEM micrographs in Figures 69-70 and 71, respectively. The observations of the 1h- and 25h-aged Almen 4A and 8A samples, Figures 69 and 71(a-c), revealed that the grain size in the near-surface regions was generally large (except very close to the surface) and of similar size as that of the as-shot peened and non-cold worked samples, i.e., there was no evidence for recrystallization of the cold-worked structure present in the near-surface regions or formation of new grains. On the other hand, the Almen 12A and 16A samples aged for 1h, Figure 70(a,d), clearly revealed the presence of finer, recrystallized grains in the near-surface regions, which have formed due to the higher degree of cold work present in these regions prior to annealing. Higher magnification observations of the Almen 4A and 8A 1h and 25h-aged samples, Figures 69(c,f) and 70(c), revealed the apparent presence of very fine, spheroidal precipitates, presumably \( \gamma' \), as well as precipitates along grain boundaries, presumably carbides. These precipitates could not easily discerned in the Almen 12A and 16A samples, Figures 70(c,f) and 71(f), presumably because they are too fine to be resolved. However, the precipitates along the grain boundaries could be seen (Figure 70(c,f)).
Figure 69—Optical and SEM micrographs of near-surface region of shot-peened Waspaloy annealed for 1h at 700°C. (a) is optical and (b), (c) are low and high magnification images of Almen 4A sample, whereas (d) is optical and (e), (f) are the counterpart images from the Almen 8A sample.
Figure 70—SE micrographs of near-surface region of shot-peened Waspaloy annealed for 1 hour at 700°C. (a) is optical and (b), (c) are low and high magnification images of Almen 12A sample, whereas (d) is optical and (e), (f) are the counterpart images from the Almen 16A sample.
Figure 71—Optical and SEM micrographs of near-surface region of shot-peened Waspaloy annealed for 25h at 700°C. (a), (b) are low and high magnification images of Almen 4A sample; (c) Almen 8A; (d) Almen 12A; (e), (f) are low and high magnification images from the Almen 16A sample.
TEM observations of the shot peened IN 718 sample revealed the presence of many dislocations (Figure 72). It appears that the lower Almen intensity sample shows relatively less dislocation density compared with the higher Almen intensity sample. Figure 72 (d) shows that shot peened IN 718 has a \(\gamma''\) precipitate. Figure 73 (a), (b) and (c) are TEM BF micrographs of near-surface region of shot-peened IN 718 of 4A, 8A and 16A sample, respectively, annealed for 25h at 700°C. These show that initial cold work is still retained after annealing. Thus precipitates in 4A and 8A samples show disc shape, but precipitates in 16A sample show a spherical shape. Cold rolling and post annealing effects have reported [61] that as cold rolling reduction increases, the \(\gamma''\) decrease whereas \(\delta\) phase increases; i.e., the precipitation of \(\gamma''\) is more prevalent than the precipitation of \(\delta\) phase for lower cold rolled materials, while the precipitation of \(\delta\) phase is more favorable for higher cold rolled materials. This suggests that since \(\delta\) phase consumes Nb during \(\gamma'' \rightarrow \delta\), higher Almen intensity may promote \(\gamma'\) rather than \(\gamma''\), while lower Almen intensity may promote \(\gamma''\) rather than \(\gamma'\). TEM BF micrograph (Figure 73 (a)) shows \(\gamma''\) reflection. TEM BF micrographs (Figures 73 (c) and (d)) of near-surface region of 16A sample and un-peened region of sample respectively annealed for 25h at 700°C are \(\gamma'\). The precipitate size of un-peened and 16A sample aged at 700°C for 25h are 17 nm and 16 nm respectively. TEM DF micrographs of near-
surface region of 16A samples (Figure 73 (e) and (f)) show very fine new precipitates. This indicates that shot peening may not affect precipitate size (growth), but promote new precipitation. The analysis of dislocations was carried out in Figure 74. The same area is imaged under a series of reflections, and the dislocations marked D1, D2. Both dislocation are visible with the (200), (1-11), and (-200) reflections, but invisible with (020) and (-1-11). Based on the invisibilities and $g \cdot b = 0$ criterion, the Burger vectors of D1 and D2 dislocation were determined to be $b = 1/2[101]$. The line directions of dislocations were determined by stereographic trace analysis to be $U_{\gamma} \approx [121]$ and [212] for D1 and D2, respectively, which established dislocation D1 to be mixed lying on the (-111) plane and D2 to be screw lying on the (-111) plane.
Figure 72 – TEM BF micrographs of near-surface region of shot-peened INCONEL 718 (a) 4 Almen Intensity (b) 8 Almen Intensity (c) 16 Almen Intensity (d) SAD pattern from 16 Almen Intensity sample at the [001]
Figure 73– TEM BF micrographs of near-surface region of shot-peened INCONEL 718 annealed for 25h at 700°C. (a) Almen 4A sample; (b) Almen 8A sample (c) Almen 16A (d) high magnification images of un-peened sample annealed 25h at 700°C. (e) and (f) are DF image of Almen 16A annealed at 700°C for 1h and 25h respectively.
Figure 74—TEM BF micrographs of near-surface region of shot-peened INCONEL 718 unaged Almen 4A sample. (a) dislocation D1 and D2 is invisible (b) dislocation D1 and D2 is visible (c) dislocation D1 and D2 is visible (d) dislocation D1 and D2 is visible (e) dislocation D1 and D2 is visible
TEM observations of the shot peened Waspaloy sample revealed the presence of many dislocations. The dislocation density has been increased with the increase of shot peening intensity as can be seen in Figure 75. Figure 76 (a) and (b) are TEM BF micrographs of near-surface region of shot-peened Waspaloy of 4A sample and 16A sample, respectively, annealed for 25h at 700°C. These show that initial cold work still have been retained after heat treatment. Thus, figure 76(b) shows fine γ’ precipitates. This indicates the new γ’ precipitates were precipitated.

The analysis of dislocation was carried out in Figure 77. The same area is imaged under a series of reflections, and the dislocations marked D1, D2 and D3. Both D1 and D2 dislocation are visible with the (0-20), (1-11), and (-1-11) reflection, but invisible with (200) and (-200). D3 dislocation is visible with the (200), (-1-11), and (-200) reflection, but invisible with (0-20) and (1-11). Based on the invisibilities and $g \cdot b = 0$ criterion, the Burger vectors of $b=1/2[0\bar{1}1]$ for D1 and D2, and $b=1/2[-101]$ for D3, dislocations have been determined. The line directions of dislocations were determined by stereographic trace analysis is to be $U_r=[211]$, [-301] and [-103] for D1, D2 and D3, respectively, which establishes dislocations D1 and D2 to be edge lying on the (111) plane and D3 to be screw lying on the (111) plane.
Figure 75—TEM BF micrographs of near-surface region of shot-peened WASPALOY (a) 4 Almen Intensity (b) 8 Almen Intensity (c) 16 Almen Intensity (d) SAD pattern at the [001]

Figure 76—TEM BF micrographs of near-surface region of shot-peened WASPALOY annealed for 25h at 700°C. (a) Almen 4A sample; (b) Almen 16A sample
Figure 77—TEM BF micrographs of near-surface region of shot-peened INCONEL 718 annealed for 25h at 700°C. (a) is high magnification images of Almen 4A sample; (b) is low magnification images of Almen 16A sample; (c) and (d) are high magnification images of unpeened sample and Almen 16A sample respectively.
4.1.7 General Discussion

This investigation has led to a number of new findings relating to the properties (electrical conductivity, hardness) and microstructure of cold-worked and surface-treated IN718 and Waspaloy, which are discussed in detail below.

The microstructure of the non-cold worked IN718 alloy was found to be basically composed of 20-μm size γ grains within which were present platelets of η-Ni₃Ti, round particles of δ-Ni₃Nb and ultrafine, nanoscale γ″ precipitates in the γ matrix, together with a low volume fraction of (Nb,Ti) carbides and (Ti,Nb) nitrides (Figures 28-30, 34, 35). That of the non-cold worked Waspaloy material was composed of a low volume fraction of spheroidal, 60-200-nm diameter γ′ precipitates in the γ matrix grains of size 30-50 μm, together with (Ti,Mo) carbides along the grain boundaries (Figures 31-33, 36). Cold working led to an increase in hardness of both alloys, with the (initial) rate of and extent of hardening being greater in IN718 compared with Waspaloy and the values after 50% reduction being, respectively, nearly twice versus a third higher than that of the counterpart cold work-free materials (Figure 27). This increase can be attributed to the continuous increase in dislocation density in the γ matrix seen from the TEM observations (Figures 42-46 and 52-55). In the IN718 material, cold working of 10% and beyond also led to shearing of the γ″ precipitates present initially (Figure 42),
leading to their dissolution and the redistribution of the alloying elements into the matrix. Nevertheless, the hardness rises, apparently, because the increase in dislocation density with cold work produces more resistance to subsequent dislocation motion during deformation than the decreased resistance that would be offered by the absence of these precipitates. The shearing and break-up of the $\eta$-Ni$_3$Ti precipitates that is also observed (Figures 37-46) likely exposes additional free volume for movement of dislocations in the $\gamma$ matrix, hence leading to a further increase in dislocation density and hardness. The Waspaloy materials did not display gross changes in microstructure with cold work (Figures 47-51), except for the continual increase in dislocation density in the $\gamma$ matrix (Figure 52-55), which is responsible for the hardness increase.

Cold working also led to an increase in electrical conductivity in Waspaloy at all cold work levels to 50%, whereas IN718 showed a very small decrease or practically no change compared with the counterpart cold work-free materials (Figure 26). Previously, it was reported using AECC measurements [64], that the electrical conductivity of the same IN718 and Waspaloy used in the present study initially decreases with cold work to $\sim$10% and then increases at higher levels of cold work to values greater than that of the non-cold worked material. The different methods used to measure conductivity maybe responsible for the slightly different results between the previous study [64] and
the current work; in the method used herein, the measured conductivity is sensitive to
the sample dimensions (width x thickness) and small variations in these dimensions
could have contributed to errors in the measured conductivity and hence the slightly
different results.

Explanations for the changes in electrical conductivity of both alloys with cold
working can be given based on the microstructural observations made of the cold
worked materials and by consideration of results from previous work on related alloys.
Electrical resistance in metallic materials arises due to electron scattering and many
factors affect the electrical resistivity/conductivity, most notable ones being elements in
solid solution, temperature, point defects, dislocations; presence of short-range and
long-range order, second-phase precipitates; and electronic structure. The
intrinsic/extrinsic factors that affect the electrical conductivity of the Ni-base alloys
studied here are likely to be complex, since this will depend on the nature of both the $\gamma$
matrix (composition, presence of short-range order) as well as the $\gamma'/\gamma''$ precipitates (size,
volume fraction, composition) that are present and how each is affected by cold work.
In general, solute atoms in solution decrease conductivity by increasing electron
scattering, as does cold working by producing an increased density of defects
(dislocations, vacancies). On the other hand, precipitation of ordered phases generally
increases conductivity by decreasing the solute concentration and hence scattering centers from the solid solution matrix in which they form. The situation is, however, more complicated when short-range order (SRO), i.e., a local departure from randomness, is present in the solid solution matrix and under these circumstances cold working may increase or decrease conductivity depending on the alloy system. Many Ni-base alloys display a phenomenon called the K-state effect, in which an anomalous increase in electrical conductivity occurs with an increase in the degree of cold work [65-68]. This feature has been related to the occurrence of SRO, which in some cases is responsible for lower electrical conductivity than a random solid solution that has been proposed to be due to the presence of lattice distortions [69, 70], unfilled d-electron shells and s-d electron scattering [65, 69-74]. Cold working is thought to decrease the degree of short-range order and hence increase conductivity to a level that more than compensates for the decrease in conductivity caused by an increase in the density of defects (dislocations, etc). The γ matrix solid solution in both IN718 and Waspaloy is likely to contain SRO, since the counterpart binary Ni-based solid solutions with the major elements like Al, Co, Fe, Mo and Nb, which are also present in the two superalloys in appreciable amounts, are well known to display SRO [65]
It is clear from the results obtained herein and in the previous study using AECC measurements [64] that Waspaloy shows an anomalous increase in conductivity with cold work (Figure 26(b)). Since the TEM observations show that the $\gamma'$ precipitates present in the alloy do not undergo any major change with cold working, it is apparent that changes produced in the $\gamma$ matrix by the passage of dislocations is responsible for the increase in conductivity. It might also be surmised from the discussion in the previous paragraph, that SRO is likely to be present in the $\gamma$ matrix solid solution in Waspaloy and is associated with a lower conductivity than that of a full random state. On this basis, it is proposed that cold working and the passage of dislocations increases conductivity by reducing the degree of SRO and that this reduction in SRO and its associated contribution to the conductivity increase overcompensates for the decrease in conductivity expected from the increase in dislocation/defect density. The observation herein that the conductivity does not change appreciably with cold work in IN718 suggests that SRO is likely to be present in the $\gamma$ solid solution matrix of this alloy also, since the increase in dislocation density that occurs with cold working should otherwise have led to the normal decrease in conductivity. The small decrease in conductivity at the 10% cold work level observed herein and previously in IN718 [64] can be related to the dissolution of $\gamma''$ observed by TEM (Figure 42) and consequent release of solute
elements into the $\gamma$ matrix, which, in turn, would promote more SRO and lower conductivity. The subsequent increase [64] or practically no change [this study] in conductivity at higher cold work levels can be explained in a similar way mentioned above for Waspaloy, namely, that the passage of more dislocations reduces the degree of SRO (and increases conductivity) to such an extent as to overcompensate for or balance the decrease in conductivity expected from the increased dislocation density.

The results have also shown that shot peening leads to a significant increase in the surface near-surface hardness to a distance of $\sim$200-400 $\mu$m in both IN718 and Waspaloy, with the degree of hardening being greater at the higher 12A and 16A Almen intensities (Figures 56 and 57). This hardening is caused by the cold work/plastic strain introduced by shot peening, with the degree of cold work and, in turn, hardness diminishing with distance from the surface. The large drop in the surface/near-surface hardness to values near that of the bulk upon annealing the shot-peened IN718 and Waspaloy materials at 900°C (Figures 58 and 59) is caused by the removal of the initial cold work that was present. Interestingly, aging the IN718 shot-peened samples at 700°C leads to a progressive increase in the hardness throughout the sample cross-section over that in the as-peened condition, with the values in the near-surface and bulk regions becoming nearly the same and attaining the highest value after 25h (Figures 60-
62). The microstructural observations reveal that this hardening is caused by formation of new precipitates of ultrafine/nanoscale $\gamma''$ (in Almen 4A, 8A) or $\gamma'$ or both (in Almen 12A, 16A) and growth of those within the $\gamma$ matrix (Figures 65-68). TEM observations (Figure 72-74) indicate that plastically deformed microstructure by shot peening still have been retained at 700°C and dislocations are Screw and Mixed type.

On the other hand, aging the shot-peened Waspaloy samples at 700°C led to either an increase (Almen 4A, 8A) or a reduction (Almen 12A, 16A) of hardness in the surface/near-surface regions compared with that of as-peened condition at short times, followed by a small increase at longer times to 25h, but to a level still above (Almen 4A, 8A) or below (Almen 12A, 16A) those in the as-peened condition (Figure 63 and 64). Microstructural observations revealed that the increase in the near-surface hardness with aging time in the Almen 4A and 8A samples is related to retention of the initial cold work that is present, together with new precipitation of $\gamma'$ precipitates. On the other hand, the initial decrease in the near-surface hardness observed in the Almen 12A and 16A samples compared with the as-peened condition is associated with the partial reduction in the degree of cold work by recrystallization in the near-surface (Figure 70(a,c)), whereas the subsequent increase in hardness at longer times is caused by the additional precipitation and growth of fine $\gamma'$ in Figure 76 (b). The observation that the hardness in
the near-surface regions of the annealed samples, though lower than that at the equivalent locations in the as-peened, un-annealed samples, is still higher than those in the bulk (Figures 63 and 64), indicates that a modest fraction of the initial cold work is still retained upon annealing for times as long as 25h at 700°C. Finally, TEM observations indicate that plastically deformed microstructure is still retained upon annealing at 700°C for 25h and dislocations have $b=1/2<110>$ with a mostly edge and mixed character and lie on $\{111\}$ planes.
4.2 Characterization of Inconel 740

The results of this study are presented in the following of sub-sections in the form of micrographs, plots, and tables. The results of the aged standard and Modified IN 740 samples are covered first, followed by Standard IN 740 from the Tensile tested samples, and Standard IN 740 from the Stress-Ruptured samples, and then discussed in the last sub-section.

The IN740 Ni-base superalloy is a potential boiler material for ultra supercritical steam power systems undergoes microstructural changes during aging of this alloy between 700 to 800°C and corresponding changes in Creep and Tensile mechanical properties. Aging leads to rapid and substantial increase in strength, whose magnitude depends strongly on the aging temperature and time. This strengthening is related to the formation of $\gamma'$ precipitates.

4.2.1 Microhardness and Tensile Test in aged Inconel 740

The microhardness with aging time at 700 and 800°C is shown in Figure 78. Rapid age hardening at both 700 and 800°C was observed, but 700°C aged sample maintains high hardness at long times [75]. Thus, hardening response is a little better in
modified alloy. Since this hardening behavior was mainly related to the precipitation of $\gamma'$, it is necessary to investigate microstructure by TEM.

![Microhardness plot](image)

*Figure 78—Microhardness of Standard and Modified IN 740 as a function of ageing time at 700 and 800°C for up to 3000 hours [75]*

Mechanical test data on three temperature aged samples were supplied by Oak Ridge National Laboratory, and the plots of ultimate tensile strength (UTS) and yield strength versus aging time are shown in Figure 79. Samples aged at 700 and 750°C showed similar responses in UTS variation, but a sharp drop of UTS following aging at 800°C very obvious. It is interesting to notice that the Young’s modulus was quite similar (Figure 79 (a)) in the 700 and 800°C aged conditions, but an abrupt increase at shorter time for 750°C aging condition was recorded, followed by rapid drop at longer aging times to values similar to those at 700 and 800°C aged samples. This could be a direct result of rapid $\gamma'$ precipitation hardening occurred at early time of aging of 750°C [75]
4.2.2 Aged Standard and Modified Inconel 740

TEM observations reveal that both Standard and Modified IN 740 exhibit in an increase of $\gamma'$ size with an increase of aging time and aging temperature (Figure 80-81). Figure 80 (e) is SAD pattern at [001] zone which indicate the precipitate is $\gamma'$.

The gamma prime size is plotted as function of aging time (Figure 82). In comparison with Standard IN 740, at 700°C Modified IN 740 show same trend as function of aging time, but slightly larger size. At 800°C, Modified IN 740 starts almost same size, but the size of gamma prime become bigger than Standard IN 740.
Figure 80—Standard IN 740 Aged at (a) 700°C for 100hrs (b) 700°C for 500hrs (c) 800°C for 100hrs (d) 800°C for 500hrs (e) [001] zone SAD Pattern aged at 700°C for 100hrs
Figure 81—Modified IN 740 Aged at (a) 700°C for 100hrs (b) 700°C for 500hrs (c) 800°C for 100hrs (d) 800°C for 500hrs

Figure 82—Gamma prime size of Standard and Modified IN 740
4.2.3 Tensile Tested Standard Inconel 740 after Heat Treatment

Standard IN 740 was heat treated at 700, 750, and 800°C for 100, 1000, and 3000hrs.

After heat treatment, tensile test was carried out. Gage section was cut, and TEM investigation was carried out.

TEM observations reveal that the gamma prime size increases with either increasing aging temperature or aging time (Figure 83). The analysis of dislocations in the aged + tensile tested sample was carried out in Figure 84. The same area is imaged under a series of reflections, and the dislocations marked D1 and D2. D1 dislocation is visible with the (-200) and (-11-1) reflections, but invisible with (020) and (11-1). D2 dislocation is visible with the (020), (11-1), and (-11-1) reflection, but invisible with (-200). Based on the invisibilities and the $\mathbf{g} \cdot \mathbf{b} = 0$ criterion, The Burger vector of D1 and D2 dislocations was determined to $\mathbf{b} = 1/2[101]$ and $\mathbf{b} = 1/2[0-11]$, respectively. The line directions were determined by stereographic trace analysis to $\mathbf{U}_T \approx [121]$ and $[1-23]$ for D1 and D2 respectively, which established dislocation D1 to be edge lying on the (-111) plane and D2 to be mixed lying on the (-111) plane.
Figure 83—Tensile Tested Standard IN 740 after aged at (a) 700°C for 100hrs (b) 700°C for 1000hrs (c) 700°C for 3000hrs (d) 750°C for 100hrs (e) 750°C for 1000hrs (f) 750°C for 3000hrs (g) 800°C for 100hrs (h) 800°C for 1000hrs (i) 800°C for 3000hrs
Figure 84—TEM BF micrographs of Tensile Tested Standard IN 740 after aging for 3000h at 800°C. (a) g=020, dislocation D1 is invisible and D2 is visible (b) g=-200, dislocation D1 is visible and D2 is invisible (c) g=11-1, dislocation D1 is invisible and D2 is visible (d) g=-11-1, dislocation D1 is visible and D2 is visible (e) g=-200, dislocation D1 is visible and D2 is invisible.

The Lifshitz-Slyosov-Wagner (LSW) theory predicts the mean linear dimension of the precipitate particles to increase by the cube root of the time [20, 21]. The size of the γ' precipitates, from TEM micrographs, was measured for all the aging conditions and the average size is shown plotted as a function of time at the three different aging temperatures in Figure 85. The cube of average γ' is plotted as function of time in Figure 86. This shows a really good linear fit at Temperature 700, 750, and 800°C, which is indication that the growth kinetics follows the predictions of the Lifshitz-Slyozov-Wagner (LSW) theory [20, 21, 76] of precipitate coarsening:

\[ d^3 - d_o^3 = kt \]  

where \( d, d_o \) = average and initial precipitate diameter, respectively, \( t = \) time and \( k = \) rate constant related to the precipitate-matrix interfacial energy, diffusivity, concentration of solute in equilibrium with a particle of infinite diameter, molar volume, and temperature.
The slope of the $d^3$ vs. $t$ plot in Figure 86 gives the coarsening rate constant $k$ at each temperature, which is

$$k = k_o \exp(-Q/RT) \tag{2}$$

where $k_o = \text{constant}$, $Q = \text{activation energy (KJ/mole)}$, $R = \text{gas constant (8.314 J/mole} \cdot \text{K)}$, $T = \text{temperature (°K)}$. From equation (1) and (2), equation (3) is drawn:

$$\ln\left(\frac{d^3 - d_0^3}{t}\right) = -\frac{Q}{RT} + \ln k_o \tag{3}$$

Figure 87 shows the plot of $\ln k$ versus $10000/T$. This shows a good linear fit and from the slope, the activation energy, $Q$, for precipitate coarsening was calculated to be about 368 kJ/mole. This value is a little higher than that reported for volume diffusion of both Al and Ti in Ni (270 and 257 kJ/mole, respectively [77]) and coarsening of $\gamma'$ in binary Ni-Al and Ni-Ti alloys (270 [78] and 282 kJ/mole [79]), as well as other multicomponent Ni-base superalloys [80-83].
Figure 85—Gamma prime size of Tensile Tested Standard IN 740 samples after aging at 700, 750 and 800°C

Figure 86—γ' coarsening of Tensile Tested Standard IN 740 Temperature at 700°C, 750°C and 800°C
$y = -4.4214x + 48.546$
$R^2 = 0.9966$
$Q = 368 \text{ KJ/mol}$

Figure 87—Activation Energy for γ coarsening of Tensile Tested Standard IN 740 after aging
4.2.4 Stress-Rupture Tested Standard Inconel 740

Standard IN 740 was Stress rupture tested at 700, 750, and 800°C under different applied stresses and the rupture times obtained as shown in Table 4; the corresponding stress versus the Larson-Miller parameter plot is shown in Figure 88. The gage and shoulder slices were sectioned from the ruptured specimens at high stresses/short. SEM and TEM investigation were carried out (Figure 89 and 90). SEM observations reveal that samples at 700°C show either or no η-Ni₃Ti phase in both gage and shoulder, whereas rupture samples tested at 750 and 800°C show more η-Ni₃Ti phases with a decrease in stress, which leads to lower ruptures, there is extensive precipitation of η-Ni₃Ti, especially at 800°C and in the gage sections. TEM observations reveal that samples at 750 and 800°C show similar increase in the average γ’ size, but sample at 700°C show very slow increase. Since rapid γ’ precipitation had occurred, samples at 750 and 800°C show a sharp increase in a shorter aging time, and then a linearly increase (Figure 92). Under same stress/temperature sections condition the γ’ size in the gage larger than that in the shoulder section, and the size difference between the gage and shoulder become greater when temperature is increased. These SEM and TEM results indicate that an applied stress accelerates η-Ni₃Ti precipitation and coarsening of γ’.
\[ y = 10^{-5}x^2 - 0.6578x + 9203.6 \]

\[ R^2 = 0.9969 \]

**Figure 88—IN 740 Stress rupture properties**
Figure 89–Stress-Rupture Tested Standard IN 740 temperature at 700°C (a) 392.6 hrs–Gage (b) 392.6 hr – Shoulder, temperature at 750°C (c) 984.5 hrs– Gage (d) 984.5 hrs – Shoulder and (e) 7201.6 hrs– Gage (f) 7201.6 hrs – Shoulder, temperature at 800°C (g) 279.2 hrs– Gage (h) 279.2 hrs – Shoulder and (i) 1491.7 hrs– Gage (j) 1491.7 hrs – Shoulder.
Figure 90—Stress-Rupture Tested Standard IN 740 temperature at 700°C (a) 392.6 hrs—Gage (b) 392.6 hr – Shoulder (c) 5227.4 hrs— Gage (d) 5227.4 hrs – Shoulder. Temperature at 750°C (e) 7201.6 hrs– Gage (f) 7201.6 hrs – Shoulder. Temperature at 800°C (g) 1491.1 hrs– Gage (h) 1491.1 hrs – Shoulder

The dislocations in the stress-rupture samples were analyzed and typical results are shown in Figure 91. The same area is imaged under a series of reflections, the dislocation marked D1 is visible with the (0-20), (1-11), and (-1-11) reflection, but invisible with (2-00). Based on these and $g$·$b$ = 0 criterion, The Burger vector of dislocation was established to be $b$ = $1/2[0-11]$. Stereographic trace analysis established the line directions to be [-211], based on which established to be of edge character and lying on the (111) plane.
Figure 91—TEM BF micrographs of Stress-Rupture Tested Standard IN 740 at 750°C for 289.3hrs.
(a) $g=-200$, dislocation $D$ is invisible (b) $g=0-20$, dislocation $D1$ is visible (c) $g=-1-11$, dislocation $D1$ is visible (d) $g=1-11$, dislocation $D1$ is visible (e) $g=000$, dislocation $D1$ is invisible.
The average particle size of $\gamma'$ precipitates, from TEM micrographs, was measured for all the aging conditions is shown plotted as time at each temperature in Figure 92. Cube of the $\gamma'$ size value was plotted versus aging time and as shown in Figure 93. As was observed with curve fitting, a good linear fit for the data of $d^3$ vs. $t$ could be obtained at all these test temperature conditions, indicating the coarsening of $\gamma'$ precipitates under these conditions was following the predictions of Lifshitz-Slyozov-Wagner (LSW) coarsening law:

Figure 94 shows the plot of $\ln k$ versus $10000/T$. A good linear fit obtained and from the slope, the activation energy, $Q$, for precipitate coarsening was calculated to be about 312 kJ/mole and 296 kJ/mole for the gage and shoulder sections, respectively. This value is a little higher than that for volume diffusion of both Al and Ti in Ni (270 and 257 kJ/mole, respectively [77]) and coarsening of $\gamma'$ in binary Ni-Al and Ni-Ti alloys (270 [78] and 282 kJ/mole [79]), as well as other multicomponent Ni-base superalloys [80-82].
Figure 92—Gamma Prime size of Stress-Rupture Tested Standard IN 740 temperatures at 700°C, 750°C and 800°C
Figure 93—\(\gamma'\) coarsening of Stress-Rupture Tested Standard IN 740 temperatures at 700\(^\circ\)C, 750\(^\circ\)C and 800\(^\circ\)C

Figure 94—Activation Energy for \(\gamma'\) coarsening of Stress-Rupture Tested Standard IN 740
4.2.5 Strengthening mechanism in aged and tensile tested Inconel 740

Tensile test results on the Inconel 740 alloy (Figure 79) reveal that aging leads to substantial strengthening and it is apparent that multiple strengthening modes may be active. Possible operative mechanisms include solid-solution strengthening, grain size hardening, and shearing of precipitates. In addition, depending on the size of the $\gamma'$ precipitates, the mechanism limiting the dislocation motion may be weak pair dislocation coupling shearing, strong pair dislocation coupling shearing or bowing between precipitates Orowan bypass mechanism [24].

4.2.5.1 Grain Size Strengthening

According to the Hall-Petch [48], the equation is given by;

$$\sigma = \sigma_0 + K_y \cdot d_m^{-\frac{1}{2}}$$

Where $\sigma$ is the yield strength, $\sigma_0$ is the friction stress, $K_y$ is the Hall–Petch coefficient, and $d_m$ is the average grain size. The Hall–Petch constant $K_y$ for pure Ni and a range of superalloys, a value of 750 MPa $\mu$m $\frac{1}{2}$ [83–85], was used. The average grain size is 350 $\mu$m and the increase in strength, $\Delta\sigma$, due to grain size, works out to 40 MPa.

4.2.5.2 Solid Solution Strengthening

The equation proposed by Gypen and Deruyttere[43] is given by;

$$\Delta\sigma = \sum_i \left( -\frac{d\sigma}{dC_i} \cdot \sqrt{C_i} \right)$$
where $\frac{d\sigma}{\sqrt{dC_i}}$, a strengthening coefficient that reflects the strengthening potency of each alloying element and $C$ is the concentration.

Chemical compositions of IN 740 (At.%) and solid solution strengthening constant for alloying elements to the $\gamma$ matrix are listed table 11 and 12, respectively. The result of solid solution strengthening before aging is 616.5 MPa. Since some alloying elements such as Ti, Nb, Al, Si, and C are going to be removed on following precipitate during aging treatment, it is assumed that 50% of those alloying elements will remain in the $\gamma$ matrix and constant through all the aging conditions. Thus, the contribution due to solid solution strengthening after aging is estimated to be 450 MPa.

*Table 11–Nominal Chemical Compositions (in At.% of IN 740

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Ni</th>
<th>Fe</th>
<th>Cr</th>
<th>Ti</th>
<th>Nb</th>
<th>Mo</th>
<th>Co</th>
<th>Al</th>
<th>Si</th>
<th>Mn</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>IN 740</td>
<td>Balance</td>
<td>0.7</td>
<td>27</td>
<td>2</td>
<td>1.2</td>
<td>0.3</td>
<td>19</td>
<td>1.9</td>
<td>1</td>
<td>0.3</td>
<td>0.1</td>
</tr>
</tbody>
</table>

*Table 12–Solid Solution Strengthening Constant for Alloying Elements to the $\gamma$ matrix ($d\sigma / dC_i^{1/2}$) [86]

<table>
<thead>
<tr>
<th>Constant</th>
<th>Fe</th>
<th>Cr</th>
<th>Ti</th>
<th>Nb</th>
<th>Mo</th>
<th>Co</th>
<th>Al</th>
<th>Si</th>
<th>Mn</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>153</td>
<td>337</td>
<td>775</td>
<td>1183</td>
<td>1015</td>
<td>39.4</td>
<td>225</td>
<td>275</td>
<td>448</td>
<td>1061</td>
</tr>
</tbody>
</table>

(MPa/At pct$^{1/2}$)
4.2.5.3 CRSS Model of Particle Strengthening

In the \( \gamma \) matrix, perfect dislocations have Burgers vectors of the type \( b = \frac{a}{2} <110> \) whereas in the \( \gamma' \) precipitates, perfect superlattice dislocations have Burgers vectors of the type \( a <110> \), where \( a \) is the lattice constant. When the \( \gamma' \) precipitates are sheared, two \( b = \frac{a}{2} <110> \) dislocations of the \( \gamma \) matrix form a pair and glide together. The leading dislocation D1 creates an anti phase boundary (APB) fault in the \( \gamma' \) precipitates that is eliminated by the trailing dislocation D2. Since the predominant glide planes are \{111\}, the APB energies on \{111\} planes are considered.

Some assumption have been made for the modeling of CRSS

1. The coherent \( \gamma' \)-particles are spherical (for some conditions where \( \gamma' \) appeared more cuboidal, the \( r \) size was obtained by using the equal volume theory, i.e. converting the cuboidal precipitates to the equivalent spheres by maintaining the same volume)

2. Ordering Strengthening is considered; Chemical, Modulus mismatch, Stacking Fault Energy Mismatch, and Lattice Mismatch strengthening are disregarded.

3. The glide of dislocation is planar; Cross-slip or climb is neglected for the modeling of CRSS.

4. All numerical values for the model are based on the Nickel base superalloy
When fine under-aged dispersions of $\gamma'$ particles are considered to be cut by weakly pair-coupled dislocations, the CRSS depends on the volume fraction $f$ of $\gamma'$ particles, their average radius $r$, and APB energy. Haasen and Labusch [39] proposed the CRSS equation given by

$$\tau_p = \frac{\gamma}{2b} \left\{ C_1 \left( \frac{4 \cdot \omega_r^3}{\pi \cdot \omega_q} \right)^{\frac{1}{2}} \cdot \left[ \frac{\gamma \cdot r \cdot f}{S} \right]^{\frac{1}{2}} + \left[ \frac{2 \cdot C_1 \cdot C_2 \cdot \xi \cdot \omega_r}{\pi \cdot \omega_q} - \alpha \cdot \eta \right] \cdot f \right\}$$

With $C_1=0.94$, $C_2=0.82$, $\omega_r=0.82$, and $\omega_q=0.75$, which are the LSW-distribution function of particle radii; $b$ is the magnitude of the Burgers vector of dislocation D1 equal to 0.254nm; $S$ is the geometric mean of the line tensions of edge and screw dislocations which is approximated by the value of $2 \times 10^{-9}$N; $\xi$ is a parameter related to the range of the interaction force between D1 and a $\gamma'$ precipitate approximately equal to 0.5; and $\alpha$ is a parameter between 0 and 1 and is related to the critical configuration of dislocation D2, which is defined in the critical configuration of the trailing dislocation D2: $\alpha = \frac{d_2}{f \cdot L_2}$. From the literature data on nickel based superalloys, the $d_2/L_2$ value is estimated as 0.2. A typical value of the antiphase boundary energy is $\gamma=0.26$ J/m$^2$. 

NIMONIC PE16 [25].
When large peak-aged dispersions of $\gamma^\prime$ particles are considered to be cut by strong pair-coupled dislocations, the CRSS depends on the volume fraction $f$ of $\gamma^\prime$ particles and APB energy. The CRSS equation given by

$$\tau_p = \frac{\gamma}{2b} \left\{\frac{\alpha f}{(\pi \cdot \omega_q)^{1/2}} \cdot \frac{1}{f} \cdot \alpha_{\gamma^\prime} \cdot f\right\}$$

With $\gamma = 0.26$ J/m$^2$, $\alpha = 1$, $\omega_q = 0.82$, $\omega_q = 0.75$, and $b = 0.254$ nm.

When large over-aged dispersions of $\gamma^\prime$ particles are considered to be bypass by dislocations, the CRSS depends on the volume fraction $f$ of $\gamma^\prime$ particles and their average radius $r$. Bacon et al [41] calculated the CRSS of the Orowan process as:

$$\tau_p = 0.9 \cdot \frac{2 \cdot K_{ Eg} \cdot b}{r} \cdot \frac{\{\ln(4 \cdot \omega_q \cdot r / R_i)^{1/2} \}}{\{\ln[r(\pi \cdot \omega_q / f)^{1/2} / R_i)^{1/2}]\}}$$

Where $K_{ Eg}$ is the geometric mean of the energy parameter of edge and screw dislocation and $R_i$ is the inner cutoff radius, which is close to $b$. $K_{ Eg}$ can be written by

$$K_{ Eg} = [K_E (\theta_d = 0^\circ) \cdot K_E (\theta_d = 90^\circ)]$$

Where $K_E = \frac{\mu}{4 \cdot \pi (1 - \nu)} [1 - \nu \cos^2 \theta_d]$ with poisson’s ratio $\nu = 0.359$ and shear modulus $\mu = 65.9$ GPa.

The calculated shear stress was converted to normal stress using the Schmid’s law $\sigma = M \cdot \tau$, where the Taylor Factor $M$ was taken to 2.45 (assuming $\{111\}<110>$ slip and average grain orientation with $<001>$).
4.2.5.4 Prediction of Yield Strength

The contribution to the strength due to each mechanism is summed as follows

\[ \sigma_{Total} = \sum_i \Delta \sigma_i^k \]

Where \( k \) can be taken between 1 to 2. The \( k \) from literature for nickel base superalloy is 1.13 to 1.19 and 1 for theoretical calculation [24]. In this study \( k = 1 \) was chosen.

Since Inconel 740 is strengthened by mainly solid solution and \( \gamma' \) precipitation, yield strength can be predicted by adding up the strength due to solid solution and \( \gamma' \) precipitation. The results are listed in Table 13 and plotted as function of aging time at temperature 700, 750, and 800°C in Figure 95.
Table 13 – Calculation results of strengthening mechanisms by using CRSS Model equations as a function of \( f_v \) and \( r \) of \( \gamma' \) precipitates. The results are converted to yield strength to compare with the measured yield strength of these conditions.

<table>
<thead>
<tr>
<th>Aging Conditions</th>
<th>( f_v )</th>
<th>( r ) (nm)</th>
<th>( \sigma ) (under) (MPa)</th>
<th>( \sigma ) (peak) (MPa)</th>
<th>( \sigma ) (orowan) (MPa)</th>
<th>Total (under) (MPa)</th>
<th>Total (peak) (MPa)</th>
<th>Total (orowan) (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>700°C-100h</td>
<td>0.136</td>
<td>12.65</td>
<td>326.52</td>
<td>329.63</td>
<td>1116.54</td>
<td>816.5</td>
<td>819.6</td>
<td>1606.5</td>
</tr>
<tr>
<td>700°C-1000h</td>
<td>0.147</td>
<td>25.11</td>
<td>580.59</td>
<td>335.56</td>
<td>684.67</td>
<td>1070.6</td>
<td>825.6</td>
<td>1174.7</td>
</tr>
<tr>
<td>700°C-3000h</td>
<td>0.16</td>
<td>41.13</td>
<td>849.21</td>
<td>341.62</td>
<td>490.19</td>
<td>1339.2</td>
<td>831.6</td>
<td>980.2</td>
</tr>
<tr>
<td>750°C-100h</td>
<td>0.14</td>
<td>22.15</td>
<td>512.58</td>
<td>331.88</td>
<td>727.24</td>
<td>1002.6</td>
<td>821.9</td>
<td>1217.2</td>
</tr>
<tr>
<td>750°C-1000h</td>
<td>0.153</td>
<td>46.92</td>
<td>894.26</td>
<td>338.47</td>
<td>421.38</td>
<td>1384.3</td>
<td>828.5</td>
<td>911.4</td>
</tr>
<tr>
<td>750°C-3000h</td>
<td>0.145</td>
<td>80.05</td>
<td>1191.6</td>
<td>334.54</td>
<td>255.36</td>
<td>1681.6</td>
<td>824.5</td>
<td>745.4</td>
</tr>
<tr>
<td>800°C-100h</td>
<td>0.135</td>
<td>57.88</td>
<td>881.4</td>
<td>329.06</td>
<td>316.20</td>
<td>1371.4</td>
<td>819.1</td>
<td>806.2</td>
</tr>
<tr>
<td>800°C-1000h</td>
<td>0.13</td>
<td>100.40</td>
<td>1301.89</td>
<td>326.06</td>
<td>191.44</td>
<td>1791.9</td>
<td>816.1</td>
<td>681.4</td>
</tr>
<tr>
<td>800°C-3000h</td>
<td>0.12</td>
<td>169.37</td>
<td>1716.58</td>
<td>321.56</td>
<td>116.37</td>
<td>2206.6</td>
<td>811.5</td>
<td>606.4</td>
</tr>
</tbody>
</table>
Experimetal Yield Strength
Weak Coupled Dislocation Model
Strong Coupled Dislocation Model
Orowan Model

(a)

(b)
Figure 95—Experimental Yield strength and Predicted Yield strength by adding grain size, solid solution, and particle strengthening, which is using CRSS model of IN 740 (a) 700°C (b) 750°C (c) 800°C.

Figure 96—Tensile Tested Standard IN 740 after aged at (a) 700°C for 100h (b) 750°C for 3000h (c) 800°C for 100h (d) 800°C for 3000h.
The results for the as-solutionized condition, predicted yield strength by adding grain size ($\approx 40$ MPa) and solid solution strengthening ($\approx 616.5$ MPa) to be $656.5$ MPa, which is close to experimental yield strength ($\approx 597$ MPa).

For aging conditions, the predicted yield strength is obtained effect by adding grain size ($\approx 40$ MPa), solid solution strengthening ($\approx 450$ MPa), and particle strengthening.

In the results for the $700^\circ$C aging condition (Figure 95 (a)), the predicted yield strength by strong coupled dislocation model reveals that it is close to experimental data but predicted yield strengths by weak coupled dislocation model and Orowan model did not show good match with the experimental yield strength. Since predicted yield strength by Orowan model did not show good match with the experimental data, Orowan model can not be applicable. Since particle size is less than approximately 10 to 20nm, weak coupled dislocation model can also be applicable; it may also be applicable aged at $700^\circ$C for 100h so that aging conditions at $700^\circ$C for 100 may be under-aged or peak-aged condition/ mixed condition, aging conditions aged at $700^\circ$C for 1000 and 3000h are peak-aged conditions.

In the results for $750^\circ$C aging condition, the predicted yield strengths by strong coupled dislocation model compare with closely with the experimental yield strength, whereas the predicted yield strengths by weak coupled dislocation model did not show
good match. Furthermore, the Predicted yield strengths by Orowan model at 100 and 1000h did not show good match with experimental yield strength but it is showing a reasonable value at 3000h so that either weak coupled dislocation or Orowan model may be applicable. TEM micrograph (Figure 96(b)) also shows that some dislocations are cutting γ′ precipitate, some are looping γ′ precipitate, which suggest that aging condition 700°C for 3000h is mixed condition.

In the results for 800°C aging condition, the weak coupled dislocation model is not applicable because it did not show good match with the experimental yield strength. For 100h aging time, weak coupled dislocation model and Orowan model show reasonable values so that weak coupled dislocation or Orowan model can be applicable. For 1000h and 3000h aging condition, Orowan model is solely applicable. TEM micrographs (Figure 96 (c) and (d)) shows that dislocations are cutting and looping γ′ precipitates, suggesting that aging condition 800°C for 100h is mixed condition, but Figure 96 (d) corresponding to the 800°C for 3000h, which shows that dislocations are bypassing the γ′ precipitates, suggesting that it is in an over-aged condition.
4.2.6 General Discussion

The hardening responses with aging time at different temperatures (Figure 78) can be correlated well with the changes in $\gamma'$ precipitate size in Figure 82. The finer $\gamma'$ size at 700°C would imply a smaller interparticle spacing and hence greater difficulty for dislocations to bypass the precipitates. This could explain the higher strength of samples aged at this temperature at all times compared with the 800°C-aged samples. The lower hardness of the 800°C-aged samples relative to those aged at 700 can be best explained on the basis of a larger interparticle spacing associated with larger $\gamma'$ precipitate size, so that dislocations are able to bypass the precipitates more easily. The sample at 700°C shows initial hardness increase in a shorter time due to rapid $\gamma'$ precipitation, but sample at 800°C show gradually increase because coarsening is faster.

$\gamma'$ precipitates in tensile tested and Stress-Rupture tested IN 740 obey LSW $d^3 - d_0^3 = kt$ coarsening behavior; Activation energy, $Q$, for coarsening $\sim$368 kJ/mole for Tensile tested sample, 312 and 296 kJ/mole for gage and shoulder, respectively. There are some conflicts between the observed microstructure and the LSW assumptions. One of the assumptions for LSW theory is that the inter-particle distances between the particles should be infinitely large compared with the particle size, which means that there is no interaction among the particles. The volume fraction of the dispersed phase
is infinitesimally small (i.e. infinitely dilute system), while at these aging conditions, high volume fraction of $\gamma'$ precipitates were observed and at some certain stage their inter-particle spacing appeared to be comparable to the particle size. As observed in TEM micrographs, the good linear fit under these aging conditions suggested the coarsening kinetics was following the diffusion-controlled growth. Similar $\gamma'$ precipitate coarsening behavior was also observed by Kusabiraki [87] at even higher temperatures in a nickel base superalloy with close chemical composition to that of alloy 740 in weight percent of Ni, Cr and Co.

There was a difference in average size of $\gamma'$ precipitates between the 700 and 750°C, but the difference in average size of $\gamma'$ precipitates between 750 and 800°C was noticeably higher than that from 700 and 750°C. The average size of $\gamma'$ precipitates (Figure 85 and 92) from 750 and 800°C suggested that during the early stage of aging, the coarsening rate of $\gamma'$ precipitates were slightly greater than that from the later stage of aging. Based on the comparisons of $\gamma'$ size variations between the 800°C and the 700/750°C, it could be predicted that acceleration of $\gamma'$ particle coarsening occurs in the temperature range between 750 and 800°C. There is an average size difference of $\gamma'$ precipitates between the Gage and Shoulder, and the difference from those is bigger at
higher temperature. These imply that $\gamma'$ precipitate coarsening was promoted by the applied stress and a higher temperature.

In general, the calculated yield strength shows good match with experimental yield strength, with deviation between predicted and experimental yield strength from 10 to 100MPa. There are some possible reasons to cause the deviation between predicted and experimental yield strength. The first reason is the microstructure characterizations such as volume fraction, precipitate size, and grain size, the second reason is the assumptions, which lead to slight overestimation of calculated yield strengths. In addition to predicting overall yield strength, it is also able to evaluate dominant strengthening mechanism in all aging conditions. For 700°C, increment of yield strength due to mainly strong pair coupling dislocation except short aging time where weak pair dislocation coupling prevails, is identified. For 750°C, the dominant strengthening mechanism is strong pair dislocation coupling except longer aging time, which may be mixed condition. For 800°C, except short aging time, which may be mixed condition, Orowan process is the dominant mechanism.
CHAPTER 5: CONCLUSIONS

The major conclusions arising from this study relating to the characterization of surface-treated and cold-worked nickel base superalloys In718 and Waspaloy are as follows:

1. The microstructure of the non-cold worked IN718 alloy was found to be basically composed of 20-μm size γ grains, having mostly {001}<100> and {001}<110> texture elements, within which were present platelets of η-Ni₃Ti, round particles of δ-Ni₃Nb and ultrafine, nanoscale γ′’ precipitates in the γ matrix, together with a low volume fraction of (Nb,Ti) carbides and (Ti,Nb) nitrides (Figures 28-30, 34, 35).

2. The microstructure of the non-cold worked Waspaloy material was composed of γ′ precipitates 30-50-μm in the γ matrix grains with texture elements near mostly {001}<100> and {001}<110> texture elements, within which was present a low volume fraction of spheroidal, 60-200-nm diameter γ′ precipitates, together with (Ti,Mo) carbides along the grain boundaries.

3. Cold working led to an increase in hardness of both alloys, with the (initial) rate of and extent of hardening being greater in IN718 compared with Waspaloy. This
hardening was related to the continuous increase in dislocation density in both alloys.

4. Cold working of IN718 to levels of 10% and higher also led to shearing of the $\gamma''$ precipitates present initially, leading to their dissolution and the redistribution of the alloying elements into the matrix.

5. Cold working also led to an increase in electrical conductivity in Waspaloy at all cold work levels to 50%, whereas IN718 showed a very small decrease or practically no change compared with the counterpart cold work-free materials.

6. It was surmised that short-range order is present in the $\gamma$ matrix of both Waspaloy and IN718 and is associated with a lower conductivity than that of a fully random state. The increase in conductivity in Waspaloy, as well as the smaller change in IN718, with cold work has been explained on the basis that cold working and the passage of dislocations reduces the degree of SRO, thereby leading to a conductivity increase such as to overcompensate for or balance the decrease in conductivity expected from the increase in dislocation/defect density.

7. Shot peening leads to a significant increase in the surface and near-surface hardness to a distance of ~200-400 $\mu$m in both IN718 and Waspaloy, with the degree of
hardening being greater at the higher Almen intensities. This hardening is caused by the cold work/plastic strain introduced by shot peening, with the degree of cold work and, in turn, hardness diminishing with distance from the surface.

8. Annealing of the shot-peened IN718 and Waspaloy materials at 900°C quickly led to a large drop in hardness to near that of the bulk material, which has been related to partial/total removal of the initial cold work that was present.

9. Aging the IN718 shot-peened samples at 700°C led to a progressive increase in the hardness throughout the sample cross-section over that in the as-peened condition, with the values in the near-surface and bulk regions becoming nearly the same and attaining the highest value after 25h. This hardening was related to the formation of new precipitates of ultrafine/nanoscale γ'' or γ' or both within the γ matrix.

10. Aging the shot-peened Waspaloy samples at 700°C led to either an increase or a reduction of hardness in the surface/near-surface regions compared with that of as-peened condition at short times, depending on the initial Almen intensity (low 4A, 8A vs high 12A, 16A), followed by a small increase at longer times to 25h, but to a level still above (Almen 4A, 8A) or below (Almen 12A, 16A) those in the as-peened condition. These changes were related to the partial reduction in % cold work by recrystallization and/or new precipitation of γ' precipitates, depending on the initial
Almen intensity level. A modest fraction of the initial cold work was still retained after 25h at 700°C in all the shot-peened samples and especially those peened at higher intensities.

11. Dislocation analysis revealed in the IN 718 and Wasploy samples the dislocation have b=1/2<110> of mixed character and lying on {111} planes.

The major conclusions arising from this study relating to the characterization of nickel base superalloys IN 740 are as follows:

1. The average γ’ size in 700°C is much finer; the hardening was noticeably less at 800°C than at 700°C.

2. The dislocation in the deformed samples have b=1/2<110> lying on the {111} planes. Within the γ’ precipitates, the dislocations are split into partials of type 1/2<110> on {111} planes connected by an APB.

3. γ’ precipitate coarsening in Tensile tested and Stress Rupture Tested IN 740 was observed to follow the $d^3 - d_o^3 = kt$ kinetics predicted by the LSW theory, indicating diffusion controlled growth. The activation energy for γ’ coarsening was determined to be 368 kJ/mole for aged + Tensile tested sample, 312 and 296 kJ/mole for gage and shoulder sections of the stress-ruptured samples, respectively,
which is higher than that reported for volume diffusion of Al and Ti in Ni and those observed for $\gamma'$ coarsening in binary and multi-component nickel-base superalloys.

4. For aging condition at 700$^\circ$C for 100h, $\gamma'$ precipitate may be under-aged or peak-aged/mixed condition but aging conditions 700$^\circ$C for 1000 and 3000h is peak-aged conditions. Aging conditions at 750$^\circ$C for 100 and 1000h, $\gamma'$ precipitates are peak-aged condition but aging condition at 750$^\circ$C for 3000h, $\gamma'$ precipitate may be peak-aged or over-aged/mixed condition. Aging condition at 800$^\circ$C for 100h, $\gamma'$ precipitate may be peak-aged or over-aged/ mixed condition but it shows over-aged condition for 1000 and 3000h.
CHAPTER 6: FUTURE WORK

1. Conductivity/Resistivity measurement of cold rolled + annealed samples of IN718 and Waspaloy should be conducted and compared with those of microstructure to establish correlation between Conductivity/Resistivity and microstructural change.

2. Detailed microstructure characterization of deformation structures in tensile tested samples of IN740 should be conducted in order to establish the effects of microstructure on the deformation and incorporate these into models for predicting the mechanical properties and strengthening behavior.
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