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PHYSICAL METALLURGY OF NICKEL-BASE SUPERALLOYS



DEFENSE METALS INFORMATION CENTER
Battelle Memorial Institute
Columbus 1, Ohio

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DMIC Report 153

May 5, 1961

PHYSICAL METALLURGY OF
NICKEL-BASE SUPERALLOYS

by

C. H. Lund

to

OFFICE OF THE DIRECTOR OF DEFENSE
RESEARCH AND ENGINEERING

DEFENSE METALS INFORMATION CENTER
Battelle Memorial Institute
Columbus 1, Ohio

FOREWORD

This report was prepared with the purpose of bridging the gap between the theoretical and the practical aspects of nickel-base superalloys. The viewpoint taken is that of a research metallurgist engaged in alloy development, and the aim is to provide a metallurgical understanding and an evolutionary history of nickel-base superalloys through the medium of meshing practical observations with theoretical considerations.

Much of the information included was previously presented in DMIC Memorandums 73, 84, and 95.

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PHYSICAL METALLURGY OF NICKEL-BASE SUPERALLOYS

SUMMARY

In this report, nickel-base superalloys are discussed from the standpoint of strengthening mechanisms, heat treatment, and evolutionary history.

In the first section of the report, solid-solution hardening, precipitation hardening, strengthening by boron and zirconium additions, and failure are considered in general theoretical terms. Dislocation movement and coherency effects are discussed and related to elevated-temperature strength properties.

The next section describes the more prominent reactions taking place during the heat treatment of nickel-base superalloys. The gamma prime and eta precipitates are described. Observations are presented on the behavior of complex carbides of the $M_{23}C_6$ and M_6C types, at both inter- and intragranular locations. Microstructural changes that occur during heat treatment are related to contemporary alloys.

The last section briefly outlines the progress and describes the attitude of alloy development by the British, U. S., and Soviet investigators during the years 1946-1960. Chemical compositions and mechanical properties are tabulated for 30 selected British, U. S., and Soviet alloys.

INTRODUCTION

The first of the nickel-base alloys for high-strength, high-temperature applications (the so-called superalloys) was developed some 20 years ago as the result of the discovery that the elevated-temperature mechanical properties of a basic solid solution could be greatly enhanced by forcing the precipitation of a finely dispersed second phase. Since that time, alloy-development studies have produced vast improvements in the capabilities of nickel-base alloys for service at the higher temperatures. The aircraft industry has supplied most of the motivation for these studies. In structural applications, involving such forms as sheet, forgings, and castings, material improvements can be translated into increased operating temperatures and reduced weight. In power-plant applications, such as turbine blading for turbojet and turboprop engines, they can be translated into increased efficiency and power output.

Even though there has been a concentrated research and developmental program on other, more refractory metals, nickel-base alloys must still be considered as paramount for present high-temperature, load-carrying applications. For the most part, the refractory metals are still in the relatively early developmental stages, and indications from the aerospace industry are that it probably will be several years before refractory metals and alloys will be commercially available for general use as primary aircraft structures. (1)* For in-atmosphere flights, nickel-base superalloys have been used for a wide variety of parts, including turbine buckets, nozzle guide vanes, rotor disks, combustion cans and liners, afterburners, noise suppressors, thrust reversers, nuts, pins, bolts, and miscellaneous fasteners. For space flights, these alloys have been used as the primary structural material on the North American X-15 rocket research planes and as shingles on the outer surface of the Project Mercury re-entry capsule. Also, they are currently being designed into the primary structures of the hypersonic-glide re-entry vehicles with which such programs as Dyna-Soar and Space Ferry are concerned.

STRENGTHENING MECHANISMS

The dominating prerequisite of a superalloy, whether nickel-base or otherwise, is that it possess superior strength at elevated temperatures. As a corollary, it must also have satisfactory corrosion resistance, especially in gaseous environments, at the temperatures of interest. In common with numerous other suitable basis metals, nickel by itself

*Figures in parentheses refer to references listed at the end of the report.

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does not possess sufficient high-temperature strength and corrosion resistance to enjoy more than limited usage at elevated temperatures. However, when judicious additions of other elements are made to nickel, and the resulting alloy is properly heat treated, astonishing increases in strength and corrosion resistance are obtainable and a superalloy truly emerges. The mechanisms for achieving strength in nickel-base superalloys are discussed in the following sections.

Solid-Solution Hardening

Solid-solution hardening can be freely defined as the increase in resistance to deformation obtained by dissolving one element in another. This increase in resistance to deformation can be interpreted by means of the dislocation theory. The movement of dislocations, or atomic disregistry, through a crystal lattice is the mechanism that permits plastic deformation in a metal or alloy. Any disturbance within the crystal lattice that hinders the movement of dislocations reduces the rate of plastic deformation, thus strengthening the alloy. The introduction of dissimilar (solute) atoms forces the crystal lattice of the solvent either to expand or to contract. This enforced change in lattice dimension causes a disturbance, which takes the form of a strained condition within the crystallographic structure of the alloy. Small areas of localized strain surround each of the solute atoms. It is these strained areas that retard the movement of dislocations and strengthen the alloy.

Because of this disturbance phenomenon, all terminal solid solutions are stronger than the pure parent metals. The beneficial effects of solid-solution hardening are valid for ternary and more complex solutions as well as for binary solutions. Each element in a complex solid solution contributes to the ultimate strength of the final solution just as it would in a binary alloy with the parent metal. The effects are additive. However, strengthening by the addition of solute elements occurs only so long as a single-phase solid solution is maintained. If the limits of solubility are exceeded, a second phase will be formed and further additions of the elements contributing to the second phase will not stress the crystallographic structure of the parent solid solution, or matrix. Consequently, maximum solid-solution strengthening is obtained when a large number of elements are dissolved to form an extremely complex solid solution. In this way, an optimum number of localized strain areas are developed to oppose the movement of dislocations. Obviously, maximum benefits are obtained when elements are dissolved which have the greatest possible difference in atomic size from that of the matrix. This results in an increase in the size of each of the localized strained areas, thereby increasing opposition to the movement of dislocations. However, it should be mentioned that these conditions are incompatible to a degree, because the rules of solid solubility show that the elements which have the greatest dissimilarity of atomic size and structure from the matrix solid solution also have the lowest solubility.

In his early work on the relationship of stressed areas and dislocation movement, Cottrell found that an explanation of the solid-solution strengthening mechanism could not include the assumptions of (1) the concept of a dislocation line as a rigid linear discontinuity, and (2) a random distribution of solute atoms in the matrix. (2) If both of these assumptions are accepted, any dislocation line would be randomly surrounded by strained areas, caused by solute atoms. The forces exerted by the strained areas on opposite sides of a dislocation would be equal and opposite in nature. Thus, the effect of the strained areas would be nearly cancelled, and the dislocation line would continue its movement through the structure almost as easily as it would in the absence of strained areas.

As documented by Parker and Hazlett(2), further considerations by Mott, Nabarro, and Cottrell showed that the dislocation line could assume a gently curved shape and that the strengthening effect could not be caused by the strain areas generated by individual, randomly dispersed solute atoms. Consequently, the contemporary theories developed by Cottrell, Suzuki, Fisher, and others postulate the existence of localized segregations of solute atoms. The presence of these segregations is necessary to explain strengthening effects obtained by the interaction of dislocation lines and strained areas. The localized segregations accumulate in the forms of clusters, "atmospheres", or as the result of short-range ordering. When a dislocation line intercepts the strained area surrounding the localized segregations of solute atoms, the movement of the dislocation is retarded. By impeding the movement of dislocations, the strength of the alloy is increased, particularly the yield strength and creep properties.

Nearly all of the nickel-base superalloys developed for elevated-temperature service have contained from 5 to 30 per cent chromium. Notable exceptions have been Hastelloy B, Hastelloy D, and K Monel. Some of the benefits gained by the use of chromium are:

- (1) The strength of nickel is increased by solid-solution hardening.
- (2) The oxidation resistance of nickel is improved.
- (3) The recrystallization temperature range is increased.
- (4) The sulfidation resistance of nickel is improved.

All of these beneficial effects are accomplished by the addition of an element which is readily available at a moderate cost. The useful properties gained by the addition of chromium to nickel were first recognized by Marsh in 1906. (3) He discovered that alloys of 15 to 25 per cent chromium had excellent electrical resistance and high-temperature oxidation characteristics. This combination of properties made these alloys ideally suited for electric furnace windings, an application for which they are still used.

Many modern nickel-base superalloys include not only chromium but also molybdenum or tungsten, or both, as additional solid-solution strengtheners. Cobalt also is present in many of the alloys, but it should have a relatively minor effect as a solid-solution strengthener because of the similarity in size of the nickel and cobalt atoms. The beneficial effects imparted by cobalt will be described later in the sections on precipitation hardening and heat treatment.

Precipitation Hardening

Nickel-base superalloys obtain most of their high strength at high temperature through the precipitation-hardening mechanism. Although the phenomenon was observed by Wilm(4) in 1906, while working on aluminum-copper-magnesium alloys, among the first to document the conditions necessary for precipitation hardening were Merica(5) and co-workers in 1919. The basic principle presented by Merica, et al., was that precipitation hardening depended upon a solid solution possessing decreasing solubility of solute in solvent with decreasing temperature. This curving solvus line is essential to precipitation hardening.

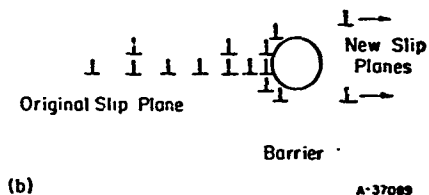
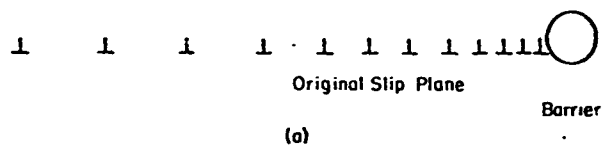
The strengthening due to precipitation hardening depends upon the formation of a dispersed second phase within the solid-solution matrix. Two heat-treating steps are usually necessary to obtain this structure. The first step, called solution treating or solution annealing, involves heating a solid solution above the solvus temperature, holding for a suitable length of time, and then cooling to room temperature at a rate necessary to retain the elevated-temperature structure. This treatment results in a supersaturated solution. The second step, called aging, results in the formation of a dispersed second phase by precipitation from the supersaturated solid solution. Although the aging step can be carried out within a reasonable time at room temperature in some alloy systems, it is usually done at an intermediate temperature. The selected temperature represents a compromise between diffusion rate and degree of supersaturation. At room temperature, the diffusion rates in nickel-base superalloys are so low that no precipitation can occur. Solute diffusion is a phenomenon vital to precipitation because clustering of solute atoms is a prerequisite to precipitation. The solute atoms must migrate, by diffusion, to concentrate or cluster at locally favorable sites and initiate the formation of the second phase. Diffusion rates increase with increasing temperature. Thus, some intermediate temperature above room temperature favors the formation of the second phase. Increasing the temperature, however, usually increases the amount of solute that can be held in solid solution. Because the amount of second phase obtainable is inversely related to the amount of solute which can be held in solid solution, an increase in aging temperature decreases the amount of second phase. As a general rule, increased amounts of second phase result in increased strength properties. Thus, some intermediate temperature above room temperature decreases the amount of

second phase. If a low temperature is selected, the result will be a large eventual volume of second phase, but it will be a long time, perhaps months or years, before anything approaching equilibrium in the precipitation reaction is attained. If a very high temperature is selected, the result will be a small volume of second phase, but this volume will be attained in a short period of time, minutes or hours.

In the case of nickel-base superalloys, the final selection of aging temperature is further complicated by the factors of service temperature and the precipitation of "secondary" second phases, such as carbides. The factor of service temperature is important because it would be unrealistic, from the standpoint of structural stability, to age at a temperature that differs much from the service temperature. This is especially true for long-term applications that are "creep limited". "Secondary" second phases, such as carbides, have a relatively minor effect on strength but can have a marked influence on the ductility at fracture.

The foregoing has been a brief discussion of how the beneficial effects of precipitation hardening were first noticed, and how the necessary conditions for the reaction were established. The reason that the presence of a second phase increases the strength of an alloy remains to be discussed. When the strengthening effects of the second phase were first noticed, the benefits were attributed to the simple act of retarding the sliding action of slip planes over one another by the presence of the other phase. However, as further observations were recorded, it became obvious that this strengthening theory was inadequate. For instance, the theory could not account for the measurable increase in strength before the appearance of an observable second phase.

The formulation and development of the dislocation theory provided a more satisfactory answer. In this interpretation, the main cause of strengthening can be looked at as the result of intersections between the strain areas surrounding a particle of the second phase and the movement of dislocations along a slip line. As a particle of the second phase is formed, it produces elastic strain in its immediate area. The size of the resulting strained area depends upon many factors, but, for the purpose of explaining a strengthening mechanism, the important thing is that it exists. As dislocations move through the crystals along slip lines, they cannot continue along the easiest path of slip when they run headlong into the strain area surrounding a particle of second phase. Because plastic deformation is dependent upon the movement of dislocations, anything that hinders or arrests their movement also restricts plastic deformation, thereby strengthening the alloy. When contact is made between dislocations and a strain field, the dislocations pile up against this barrier. In order to continue, the dislocations must climb around the barrier, a process governed largely by diffusion rates, until the barrier is cleared. They can then continue along the next available plane of easiest slip. (See Figure 1(6).) Such a description provides an explanation of strengthening



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FIGURE 1. MOVEMENT OF DISLOCATIONS PAST A BARRIER⁽⁶⁾

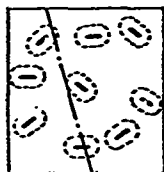
effects before the appearance of an observable second phase, because the lattice is strained as soon as the second phase starts to form.

When a second phase is formed by precipitation from a solid solution (this is to distinguish it from a mechanically produced dispersed phase that is inert to its surroundings), there usually is a period of coherency between the second phase and the matrix solid solution. Coherency is defined as the enforced registry or alignment, in both crystallographic structure and lattice spacing, between matrix solid solution and precipitating second phase.

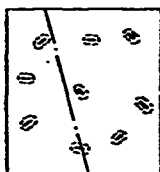
Often the normal structure of the precipitating phase is very similar to that of the matrix. In this case, only a small amount of internal strain results because of the small amount of mismatch. More often, however, there is a difference of either crystallographic structure or lattice spacing that results in a greater degree of internal strain. Thus, coherency is present during the early period of precipitate growth when registry with the matrix is forced. Because of this forced registry, the second phase has no characteristics to distinguish it from the matrix and thus cannot be seen under a microscope.

As the precipitate grows, so does the localized internal strain. In time, these strains build to the point where local shearing takes place and the precipitate becomes an isolated particle of second phase. When shearing occurs, there is a definite release of the internal strain and a decrease in the size of the strain-affected area. Subsequent agglomeration of the second-phase particles further reduces the effectiveness of the strained areas because fewer are present, and dislocations can travel a greater distance between contacts. (See Figure 2(a).)

If the second phase normally has the same crystallographic structure as the matrix, coherency strains are the result of a difference in lattice spacing. The Ni_3Al compound, usually designated gamma prime, in nickel-base superalloys is an example of this kind of precipitate. Both the solid-solution matrix, designated gamma, and the Ni_3Al compound have a face-centered cubic crystal structure. Although the lattice parameter of the matrix changes slightly with small changes in composition, the difference in lattice parameters between matrix and the second phase is very slight, generally not more than 0.5 per cent. However small this difference may seem, it creates considerable internal strain during the period of coherency when the lattice of the precipitate is forced to conform to the lattice of the matrix. The gamma-prime phase is capable of taking a considerable amount of other elements into solution, namely chromium, titanium, and cobalt. Taylor and Floyd, and Nordheim and Grant have shown that up to 65 per cent of the aluminum atoms in Ni_3Al can be replaced by titanium. (7)



a. Coherent Precipitation and Surrounding Volume of Strained Matrix



b. Incoherent Precipitation

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FIGURE 2. SLIP INTERFERENCE BY COHERENT AND INCOHERENT PRECIPITATION OF THE SAME DISPERSION⁽⁸⁾

Note fewer intersections between slip lines and the relatively smaller strained areas surrounding the incoherent precipitate.

Sometimes the normal (or most stable) crystallographic structure of a precipitated second phase will differ from that of the matrix. During the period that such a phase is coherent with the matrix, its crystal structure will conform to that of the matrix even though this is not the structure of lowest energy. Such a structure is known as a transition phase. When the coherent precipitate shears away from the matrix to form a particle of second phase, the crystal structure of these particles usually will be that of lowest energy or normal arrangement. Sometimes the transition phase actually forms particles of second phase with an abnormal crystal structure, i.e., similar to the matrix. These particles are dissolved and reprecipitated in the normal manner as precipitation continues.⁽⁸⁾ The Ni_3Ti compound, usually designated eta, in nickel-base superalloys is an example of such a precipitate. Eta has a hexagonal-close-packed crystal structure (hcp) in contrast to the face-centered cubic structure (fcc) of gamma, the matrix. Although this precipitate is fcc during the period of coherency with the fcc matrix, it can be precipitated as particles of second phase which have either the hcp (normal; lowest energy) or fcc (abnormal) structure. As precipitation continues, however, the fcc form of the eta phase will be dissolved and reprecipitated as the hcp form. Aging at low temperatures allows more of the fcc form of eta to be precipitated than does aging at higher temperatures.

Although both the eta and gamma-prime precipitates are beneficial to strength properties, the gamma prime is considered to be more desirable. One reason for this is that eta has a stricter regime of precipitation than does gamma prime. Eta has almost no solubility for aluminum, nickel, cobalt, or chromium atoms, and adheres closely to the stoichiometric Ni_3Ti composition. As mentioned earlier, gamma prime has greater solubility for foreign atoms, particularly titanium, and is less rigidly confined. Eta also has a hcp structure. This necessitates a crystallographic restacking, from the fcc present during coherency, before the stable hcp phase can be formed. It is hypothesized that a combination of these factors results in:

- (1) A delay in the precipitation of eta
- (2) The sudden release of a relatively greater portion of its strain energy (as compared to gamma prime) once precipitation of the second phase (shearing from the matrix) takes place. See Figure 2.⁽⁸⁾

Another reason eta is considered less desirable than gamma prime is the greater tendency for eta to agglomerate into larger particles once coherency is broken. Eta forms relatively long acicular particles of second phase which contribute less to the strengthening than do the tiny, finely dispersed spheroidal or cubic shaped particles of gamma prime.

Diffusion rates are important during the formation and growth of the second phase. As mentioned earlier, the initial step in precipitation is the clustering of solute atoms, i.e., the aluminum and titanium atoms in nickel-base superalloys. Any elements present in the solid solution that reduce diffusion rates will retard the clustering, and hence the precipitation, of the second phase. The heavy elements with high melting points, e.g., molybdenum and tungsten, have been found to have a beneficial retarding effect.

These same elements are also thought to retard diffusion at the interface of the solid-solution matrix and the coherent second phase. The retardation of diffusion at this interface is important because the growth of the second phase depends upon a transfer of aluminum and titanium atoms from the matrix to the precipitate. Precipitation hardening is most beneficial to the strength properties of an alloy during the period of coherency and soon after coherency is broken. It reasonably follows that molybdenum and tungsten, by slowing down this transfer of aluminum and titanium atoms, extend the period of coherency and thereby reduce the creep rate of the alloy. Once coherency is broken, the isolated particles of second phase start to agglomerate and "overaging" is said to have begun. If carried to the ultimate, all of the second-phase particles would agglomerate into one large mass. The result obviously would be a very weak structure. Fortunately, this requires an extremely long period of time, much beyond the intended service life of the nickel-base superalloys.

The presence of cobalt in the superalloys exerts a beneficial effect by increasing the solubility temperature of aluminum and titanium. Thus, the cobalt-containing alloys can maintain their strength at higher temperatures than can cobalt-free alloys, because of the higher solutioning temperature of the gamma-prime phase.

Strengthening by Boron and Zirconium Additions

Boron and zirconium have been found to increase the stress-rupture life of nickel-base superalloys. Only very small amounts of these elements, 0.0003 to 0.015 per cent boron and 0.01 to 0.04 per cent zirconium, are necessary to impart this strengthening effect. Boron has a greater effect on properties than zirconium when only one of the elements is added, but optimum properties are obtained when both are added. When added in the proper amounts, both elements also improve the hot workability of wrought nickel-base super-alloys. If too much boron is added, complex borides are formed at the grain boundaries. These complex borides usually are hard and brittle, and sometimes have a low melting point, factors which reduce the ductility and hot workability of the alloys.

When present in proper amounts, boron atoms, and probably zirconium as well, are thought to fill in vacancies and lattice imperfections at or near the grain boundaries. Vacancies and lattice imperfections are prevalent in these areas because of misfits in orientation between grains. Boron has an atomic diameter of 1.72 Å while that of zirconium is 3.16 Å. These are odd-size atoms, boron being smaller and zirconium larger than the atomic diameters of the major constituents of nickel-base superalloys. Decker, Rowe, and Freeman have shown that boron and zirconium could easily fill the vacancies and lattice imperfections available in a typical superalloy. (9) This filling or healing of lattice imperfections at the grain boundaries is important because the grain boundaries are excellent sources of the vacancies necessary for dislocation climb. When dislocations bump into a grain-boundary barrier, they must climb around the barrier in much the same manner as they do when meeting a particle of second phase. Dislocation climb is usually thought to be diffusion limited, i.e., the rate of climb is dependent upon the rate of self-diffusion within the alloy system. (6) The presence of lattice vacancies at the grain boundaries makes diffusion easier, and benefits dislocation climb, thus weakening the alloy. As presently understood, the role of boron and zirconium is probably to fill these lattice vacancies.

Bulygin, et al., reported that boron additions to a typical nickel-base superalloy raise the temperature at which fracture changes from trans-crystalline to intercrystalline. (10) In essence this means a raising of the equicohesive point, viz., that temperature at which shear strain is greater at the grain boundary than within the grain. In addition, Decker, et al., (9) have proposed that boron, in filling the lattice imperfections near the grain boundaries, retards carbon segregation and subsequent carbide formation in the grain boundaries. This mechanism is discussed in more detail in the section on "Heat Treatment and Grain Boundary Behavior".

This review of strengthening mechanisms acknowledges is not detailed and comprehensive. Moreover, many of the interactions involved in strengthening have not been sufficiently investigated as yet. Many details are unsettled. For example, although the gamma-prime precipitate is generally designated as $\text{Ni}_3(\text{Al-Ti})$, examination has revealed that other elements are soluble in this basic phase. (11) Solubility is necessary because a greater volume of gamma prime is present than could possibly be formed if the composition were a rigid $\text{Ni}_3(\text{Al-Ti})$. Because of the similarity of atomic structure between matrix and second phase, one would expect the volume of second phase to be about four times the sum of the atomic percentages of aluminum and titanium. This is not the case. The volume of second phase is always more than would be expected, when measured on samples overaged for sufficient time so as to at least approach equilibrium. This phenomenon has been noted in many investigations.

Failure

Failure is briefly considered as a separate topic because nickel-base superalloys sometimes fail before their full strength can be utilized. Factors other than those mentioned in the sections on strengthening mechanisms promote failure before full advantage can be taken of dislocation blocking and pinning. That is, failure may occur while the creep rate is still low.

Failure, at the high temperatures of nickel-base superalloy application, usually is associated with grain-boundary phenomena to which impurities and grain-boundary constituents are intimately connected. The strength (perfection) analyst recognizes the presence and effect of these factors, but prefers merely to acknowledge their existence because they do not make a readily definable contribution to strength. It remains for the failure (imperfection) analysts to do the active work in this field. The practical metallurgist is aware that great strength is embodied in the superalloy composition; his task is to achieve fuller utilization of this vast potential strength.

The presence of low-melting-point "tramp" elements at the grain boundaries is an example of conditions that promote premature failure. In nickel-base superalloys, lead, bismuth, tin, and others are especially bad in this respect. Only very small amounts of these elements, in the order of 0.001 per cent, are required to impair seriously the performance of nickel-base superalloys. Because these tramp elements have low melting points and low solubility in the matrix, they tend to remain in the grain boundaries during solidification. On reheating, they can cause premature failure by either incipient melting or cracking. Vacuum-melting techniques have done much toward eliminating these low-melting-point elements during the melting cycle. At the high temperatures used in melting the superalloys, the low-melting-point elements are vaporized from the bath and deposited on the cool walls of the furnace chamber. A low pressure is necessary for this "elimination by vaporization" to be most effective.

From a failure standpoint, vacuum melting is also beneficial in that it minimizes the number of nonmetallic inclusions present by reducing the gas content in the metal. This is a definite aid in fabrication as well as in service. The absence of stringers or accumulations of nonmetallic inclusions does not increase the strength of the alloy, but it definitely reduces the likelihood of failure.

Most nickel-base superalloys operating in high-temperature, high-stress applications are thought to fail by the joining of localized cracks that initiate at grain boundaries. With increasing time under service conditions, the localized cracks extend, join, and eventually cause fracture.

Decker, Rowe, and Freeman published the results of an extensive investigation on cracking at the grain boundaries.⁽⁹⁾ This was a practical investigation of failure and showed that boron and zirconium additions, in specific amounts, decreased the tendency toward crack formation in the grain boundaries. The onset of cracking was postponed from the primary or early secondary stages of creep until the tertiary stage of creep, thus extending both the rupture life and rupture elongation. Along with, or because of, this extended service time, surface cracking became more prevalent. Many investigators have shown that, in the specific case of aircraft turbine buckets, failure was preceded by cracking along the thin leading and trailing edges of the buckets. However, the mere appearance of these cracks was not a good indicator of expected failure. There is adequate evidence that although cracking was initiated at about the same time in buckets made from air- and vacuum-melted material, failure occurred faster in the air-melted alloys. This would indicate that microscopic cracking at the grain boundaries may play an important role in fracture.

Interactions at the grain boundaries are dependent upon time, temperature, stress, and prior heat treatment. The physical make-up and constituents at the grain boundaries exert a profound effect on the failure of nickel-base superalloys, particularly in long-term applications. When some constituents are present, such as low-melting-point tramp elements, they are permanently located at grain boundaries. These elements must be removed if optimum strength properties are to be obtained. Other constituents, such as carbides and gamma prime, respond to thermal treatment and can be present in several forms. These constituents must be present in the most advantageous form if optimum strength properties are to be obtained. Superalloys are weakened considerably in high-temperature applications if there are gamma-prime depleted areas at the grain boundaries. Some carbides must be present at the grain boundaries to insure sufficient ductility so as to avoid premature, brittle fracture.

HEAT TREATMENT AND GRAIN-BOUNDARY BEHAVIOR

The importance of heat treatment of nickel-base superalloys cannot be overemphasized. Because of the complex composition of these materials, a wide variance in properties can be achieved in any given alloy by use of different heat treatments. The optimum heat-treatment cycle for a given alloy will depend upon the properties desired. Most commercial alloys are offered with a recommended heat treatment that provides an optimum

compromise of properties. For a definite set of operating conditions, another heat treatment may be specified, tailored to give the best combination of properties for the specific conditions involved. Thus, once the response to heat treatment is established for a given alloy, heat treatments can be specified to best meet the particular operational demands of temperature and stress state. For example, the producers of René 41 recommend one heat treatment when tensile strength is critical and another one when creep is most important. See Table 1.(12,13) Other examples, showing even finer subdivision of requirements, are found in the Nimonic alloys. With these alloys, the producers recommend variations of heat treatment that will give greater or less extension at failure in creep (Table 1). It must be emphasized, however, that the response of these alloys depends upon their chemical composition and that a given heat treatment cannot be shifted from one alloy to another. The interaction between the ingredients and the heat-treating conditions is critical.

The ingredients of importance are the following:

- (1) Matrix solid solution (gamma)
- (2) Nickel-aluminum-titanium intermetallic compounds (gamma prime, eta)
- (3) Carbides (M_6C^* , $M_{23}C_6$, M_7C_3 , TiC)
- (4) Nitrides and cyanonitrides (TiN, TiCN)

When the alloys are in the solution-treated and quenched condition, the matrix contains nearly all of the ingredients in a single-phase solid solution. Only the nitrides (TiN has some solubility for columbium and carbon) are stable enough to remain as discrete particles at temperatures up to the melting point of the alloy. TiC is a compound with solubility for columbium (when present) and nitrogen. It is important to remember that the matrix serves as a reservoir for the elements necessary to form second phases, and that the matrix constantly interacts with the second phases once they are formed. In addition, during the solution portion of the heat-treatment cycle, the matrix not only acts as a reservoir, but with increasing time at temperature, the atoms seek the positions of lowest energy, a process sometimes called conditioning.

The temperature at which all of the aluminum and titanium are taken into solid solution within a given time is called the solvus temperature. Certain elements, when present in the matrix, increase the solvus temperature. This allows the gamma-prime and eta phases to remain as discrete particles of second phase at a higher temperature, thus raising the useful temperature range of the alloy. Cobalt has been found to be beneficial in raising the solvus temperature of nickel-base superalloys.(12) Cobalt also increases the solubility of carbides in the matrix.(12)

*The "M" represents the metallic radical, usually chromium, molybdenum, and possibly titanium.

TABLE 1. RECOMMENDED HEAT TREATMENTS FOR SOME
NICKEL-BASE SUPERALLOYS

<u>Rene 41⁽¹³⁾</u>		
Treatment A	1950 F - 4 hr - air cool + 1400 F - 16 hr air cool	Provides optimum short-time tensile properties
Treatment B	2150 F - 2 hr - air cool + 1650 F - 4 hr - air cool	Provides optimum stress-rupture properties
<u>Nimonic 80A⁽¹²⁾</u>		
Treatment A	1975 F - 8 hr - air cool + 1290 F - 16 hr - air cool	Standard heat treatment - best compromise of long- and short-time properties
Treatment B	1975 F - 8 hr - transfer direct to furnace at 1560 F - hold for 24 hr, furnace cool + 1290 F - 16 hr - air cool	Provides greater creep extension at fracture
<u>Nimonic 90⁽¹²⁾</u>		
Treatment A	1975 F - 8 hr - air cool + 1290 F - 16 hr - air cool	Standard heat treatment - best compromise of long- and short-time properties
Treatment B	1975 F - 8 hr - transfer direct to furnace at 1650 F - hold for 24 hr, furnace cool + 1290 F - 16 hr - air cool	Provides greater creep extension at fracture

If the high-temperature structure, that of a single-phase solid solution, is rapidly quenched and reheated, the first change in hardness is due to submicroscopic segregation of chromium atoms. This chromium clustering, which is believed to be short-range ordering, results in a maximum on a plot of hardness versus temperature. Such a peak is only evident on heating, however. On cooling, the effects of chromium clustering are masked by the more influential precipitation of gamma-prime phase. The temperature at which the peak hardness occurs during heating varies with composition. For nickel-base superalloys, the peak generally is near 1000 F. Some investigators have referred to this phenomenon as a "K-state".

Further heating of the quenched structure results in the precipitation of a second phase: gamma prime or eta, or both. The temperature at which this precipitation can be detected optically depends upon the time a sample is held at temperature. However, some changes in crystal structure can be detected sooner by other methods, such as electrical-resistivity and internal-friction measurements. As indicated earlier, the sequence of steps in the precipitation reaction is clustering, nucleation, coherency, and growth. The purpose of the aging treatment is to produce precipitate particles of a specified size and distribution. Sponseller and Flinn⁽¹⁴⁾ have shown that an initial particle size (prior to creep testing) of 1000 to 1500 Å gave the lowest second-stage creep rate for an alloy of 75Ni-15Cr-10Al. They showed further that these results are in good agreement with the formulas of Weertman for the rate of creep at high and low stresses in dispersion-hardened alloys.⁽¹⁵⁾

Different solution and aging treatments are used on the same alloy, depending on whether it is going to be heat treated to obtain optimum stress-rupture or tensile properties. This is due to the difference in fracture mechanisms involved. Maximum stress-rupture strength is usually obtained with a relatively large grain size, whereas maximum tensile strength is obtained at relatively fine grain sizes. Also, stress-rupture strength mechanisms are diffusion controlled, whereas short-time tensile properties are not. The relatively short time until fracture in tensile tests does not permit a great deal of reorganization of gamma prime and carbides, a diffusion-controlled process. On the other hand, Decker, et al.,⁽⁹⁾ have shown that considerable reorganization of gamma-prime and carbide particles occurs during long-time creep tests. Of particular importance was the depletion of gamma-prime particles which they observed at and near the grain boundaries transverse to the applied stress. Stress definitely was required to produce depletion of the gamma prime.⁽⁹⁾ Microcracks formed in these depleted areas, joined, and caused failure. The main point here is that the behavior of the alloy varies with the time-temperature-stress situation. The aging process, begun in heat treatment, may or may not continue in service. Furthermore, stress strongly influences aging behavior. An important function of heat treating is to put

the gamma-prime particles in the most favorable condition for accepting further aging under stress.

In general, higher solution and aging temperatures are used to obtain optimum stress-rupture properties, and lower temperatures are used for optimum tensile properties. See Table 1. The higher solution temperatures take more of the aluminum and titanium into solution and provide a larger grain size. The higher aging temperatures provide gamma prime of medium size before stress is applied. The lower aging temperatures provide a fine grain size and a finely dispersed gamma prime of small particle size. These are the most advantageous for tensile strength and also give higher hardness.

For most applications, a combination of strength and ductility is necessary. Carbides are very influential on the ductility of nickel-base superalloys. Much has been written on the role and behavior of carbides in superalloys, and many heat treatments have been devised to position them correctly. However, there is some disagreement as to what constitutes the correct positioning. Betteridge documents strong evidence that precipitation of the $M_{23}C_6$ -type carbide in the grain boundaries is necessary to achieve optimum stress-rupture properties.⁽¹²⁾ According to his data, this structure affords greater elongation at fracture and thus increases the strength. Without these carbides, a brittle fracture occurs before full utilization of ultimate strength. By precipitating more carbide at the grain boundaries, it is possible to attain greater elongation before fracture in the Nimonic alloys. This is illustrated in Table 1.^(12,13) Heat treatments "B" for Nimonic 80A and Nimonic 90 are designed to provide increased elongation at fracture during stress rupture by precipitating increased amounts of carbide at the grain boundaries. The increased carbide precipitation is the result of the intermediate step in the heat treatment. It is interesting to note that the presence of cobalt in Nimonic 90 (15 to 21 per cent) raises the temperature of this intermediate step. Additions of cobalt to nickel-base superalloys increase the solubility of carbides in the matrix. As a result, either higher precipitation temperatures or increased carbon contents, or both, are necessary to provide adequate precipitation of carbides at the grain boundaries. Thus, cobalt increases the solvus temperatures of both gamma prime and carbides in nickel-base superalloys. By increasing the solvus temperature of gamma prime, additions of cobalt increase the useful temperature range of the alloys. By increasing the solubility of carbides, cobalt additions require the use of higher carbide precipitating temperatures.

Most investigators in the United States do not look kindly upon $M_{23}C_6$ carbides in the grain boundaries. The major concern, however, is not with stress-rupture properties but with brittleness in shorter time applications and in welding. Tensile elongation is lowered. Structures welded under a high degree of restraint have cracked upon subsequent thermal treatment. The tensile stresses set up by heating under restraint, combined with the

usual stresses introduced by thermal gradients and the effect of aging during further heat treatment, cause fracture at the brittle $M_{23}C_6$ carbide network in the grain boundaries. The complexly alloyed nickel-base superalloys have relatively low thermal conduction. Thus, steep thermal gradients are produced whenever anything except the thinnest section sizes are heated. If the alloy does not have sufficient ductility to relieve these stresses, the alloy will rupture internally. This is especially true when other stresses, e.g., aging, machining, grinding, etc., are superimposed upon the thermal stresses.

The disagreement on the relative worth of intergranular $M_{23}C_6$ carbides is most pronounced when data on their effect on stress-rupture properties are studied. Some data show that intergranular carbides are not only beneficial but necessary to achieve optimum stress-rupture life.⁽¹²⁾ Formation of $M_{23}C_6$ carbides at the grain boundaries results in a chromium-depleted zone adjacent to the carbides (the "M" radical in $M_{23}C_6$ is predominantly chromium). There also is less gamma prime in this depleted zone. Thus, this zone is weaker but more ductile than the areas adjacent to it. Stresses that concentrate at the grain boundaries must be relieved by either localized plastic deformation or cracking. By providing an "avenue of high ductility", the stresses can be relieved by localized yielding rather than by cracking.⁽¹²⁾

Other data show that microcracking in these same depleted zones is the very cause of failure.⁽⁹⁾ An examination of the structures resulting from gamma-prime and carbide precipitation while under stress showed that creep caused agglomeration of $M_{23}C_6$ -type carbides at the grain boundaries normal to the applied stress. The areas immediately surrounding these carbide agglomerations became depleted of gamma prime, thus weakening them. Microcracks formed at the interface of the $M_{23}C_6$ particles and the gamma-prime depleted zones.⁽⁹⁾ A joining of a succession of these microcracks caused premature, brittle failure.

Weaver⁽¹⁷⁾ found that the formation of microcracks was affected by microstructure, composition, and stress. Stress-rupture life and total elongation at fracture were linearly related to the number of microcracks. Brittle fractures (short stress-rupture lives) in stress rupture showed few, but lengthy, microcracks, indicating that crack propagation was rapid. Heavy carbide precipitation, in globular form, at the grain boundaries promoted nucleation of microcracks and also helped restrict them within the intercarbide distances.

The recent trend toward adding greater amounts of molybdenum or tungsten, or both, to superalloys has further complicated the understanding and control of carbides. These elements facilitate the formation of another carbide M_6C . This carbide forms preferentially to the $M_{23}C_6$ type and is the more stable of the two at higher temperatures. Increasing amounts of the M_6C carbide are formed with increasing amounts of the heavy elements

(molybdenum and tungsten). This is particularly true when these elements are present in amounts of 6 per cent or more.

The thermal behavior of the two types of carbides in René 41, a nickel-base superalloy containing 10 per cent molybdenum, was illustrated by Weisenberg and Morris. Figure 3, reproduced from their work(13) illustrates the greater stability of the M_6C -type carbide at higher temperatures. It also helps to explain the two heat treatments recommended for the alloy. The 1950 F solution temperature recommended for short-time tensile properties allows the dissolution of the $M_{23}C_6$ carbides and the gamma prime, but the M_6C carbides are unaffected. The presence of the M_6C carbides restricts grain growth and a fine grain size is retained. The relatively low aging temperature recommended, 1400 F, results in a finely dispersed gamma prime of small particle size. The combination of fine grain size and finely dispersed small particles of gamma prime gives optimum short-time tensile properties.

For optimum stress-rupture properties, the higher solution temperature, 2150 F, is recommended. At this temperature, gamma prime and both carbides are taken into solution. This permits some grain growth which is believed to be beneficial to high-temperature stress-rupture properties. The relatively high aging temperature recommended, 1650 F, results in a coarser gamma-prime particle size, also believed to be beneficial to stress-rupture properties. Taking all of the carbides into solution and reprecipitating them at 1650 F also results in the formation of $M_{23}C_6$ -type carbides at the grain boundaries. As mentioned earlier, Betteridge has shown this to be the most advantageous as-heat-treated condition (prior to creep under stress) for optimum stress-rupture properties.

The unique property of the nickel-base superalloys that makes them so useful in varying temperature applications is that these materials are not permanently softened by overheating. The hardness and strength can be recovered to a great extent by reaging at the desired temperature without the use of a re-solution treatment.

EVOLUTION OF SUPERALLOYS

The evolution of modern nickel-base superalloys has coincided with the evolution of the gas-turbine engine for aircraft. The aircraft industry has supplied much of the motivation for the development of superalloys for high-strength, high-temperature applications.

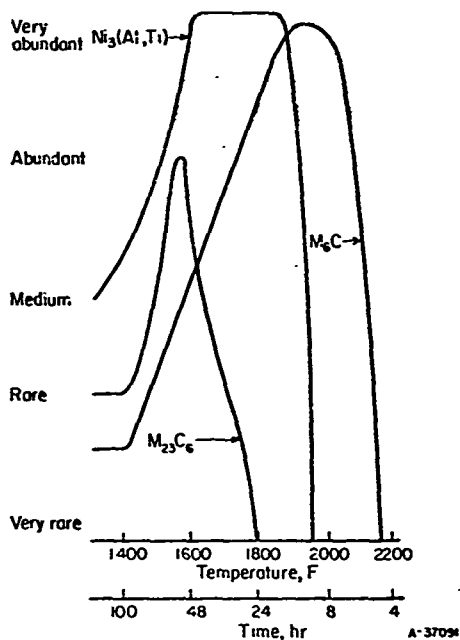


FIGURE 3. ISOTHERMAL PHASE REACTIONS IN RENÉ 41⁽¹³⁾

Bar-stock specimens were initially treated at 2200 F and water quenched. After aging at the times and temperatures indicated, phases were extracted and examined by X-ray diffraction.

Whittle developed the first workable model of the aircraft gas-turbine engine in 1939 and 1940. The operating principles of that engine, and of all those that have followed, included:

- (1) Intake of air
- (2) Compression of air
- (3) Mixing of air with fuel
- (4) Combustion of fuel-air mixture to provide expanding, hot gases
- (5) Channeling of the hot gases through an exhaust tube to provide forward thrust.

The more recent models also include an afterburning (or reburning) stage, which is an addition to the five basic steps outlined above. The afterburner provides increased thrust by burning additional fuel in the hot gases of Step 5.

To make the engine self-sustaining, a turbine is inserted between the combustion and exhaust stages. The function of the turbine is to drive the air compressor. The turbine consists of a rotating wheel (rotor), rotor blades (buckets), and stationary blades (nozzle guide vanes) which direct the flow of gases to the buckets. The rotor is connected to the compressor by a shaft. As the hot gases leave the combustion area, they strike the buckets, cause rotation, and drive the compressor, an action similar to that of a windmill. In a turbojet engine, the buckets absorb only enough energy from the flow of hot gases to drive the compressor, the balance being allowed to exit through the exhaust tube and produce thrust. In a turboprop engine, more energy is absorbed by the buckets and a lesser amount is exited for thrust. This greater amount of absorbed energy is used to drive propellers as well as the compressor.

Whittle's prototype engine illustrated that the efficiency of the gas-turbine engine for aircraft would depend mainly upon the temperature of the gases after combustion. However, an increase in the gas temperature also meant an increase in the temperature of the turbine blades and turbine rotor. The buckets and rotors must withstand not only thermally induced stresses (hot gases and thermal gradients) but also mechanical stresses induced by the rotation. Nozzle guide vanes do not rotate and are subjected primarily to thermal stresses.

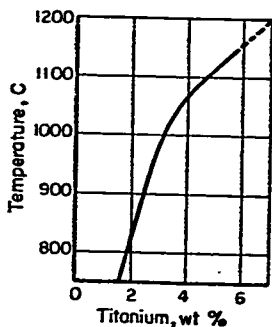
Modified stainless steels were the best materials available when the first engines were produced, and, although these engines were successful, it was obvious that stronger materials were necessary before major advances could be made in efficiency and power by raising the temperature of the hot gases. Raising the temperature of the gases increases the temperature in the combustion and exhaust stages as well as in the turbine

sections. Nickel-base superalloys have been widely used in engines for such parts as buckets, nozzle guide vanes, rotors, combustion liners, afterburners, noise suppressors, thrust reversers, nuts, bolts, pins, and miscellaneous fasteners. They also have been used as primary structural material in some of the newer aircraft designs.

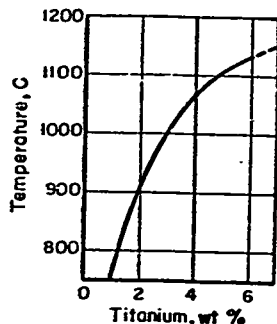
Workers at the Mond Nickel Company, in Great Britain, developed the first series of age-hardenable nickel-base superalloys to meet high-temperature, high-strength applications just described. The first alloy, developed in 1941, (12) was called Nimonic 80. Nimonic 80 was an outgrowth of the discovery that the addition of a specified amount of titanium to Nimonic 75 resulted in the precipitation of an observable intermetallic compound. The behavior of the compound could be controlled by heat treatment. Further work on Nimonic 80 showed that additions of aluminum increased the creep resistance of the alloy. This led to the development of Nimonic 80A, available in 1944. At about the same time, the International Nickel Company was developing the first age-hardenable alloys in the United States, Inconel X and Inconel W. These were evolved in a manner similar to Nimonic 80, by the addition of aluminum and titanium to the Inconel base composition. All of these early alloys were essentially nickel-chromium solid solutions plus approximately 2.0-2.5 per cent titanium and 0.5-1.5 per cent aluminum.

In 1945, Mond announced Nimonic 90. In this alloy, 15-21 per cent of nickel was replaced with cobalt. As mentioned earlier, cobalt increases the solubility temperature of the gamma prime compound. Thus, a comparison of the solubility curves for Nimonic 80A and Nimonic 90 at the same titanium level (2.4 per cent) and the same titanium/aluminum ratio (2:1) shows that the solubility temperature has been raised 145 F, from 1615 F (880 C) in Nimonic 80A to 1760 F (960 C) in Nimonic 90. (12) (See Figure 4.) By raising the solubility temperature 145 F, the effective working temperature is raised a corresponding amount. From these curves it was apparent that further increases in the service temperature could be obtained by adding greater amounts of titanium and aluminum. However, the attractiveness of greater titanium and aluminum additions was more theoretical than practical. By increasing the strength and the upper temperature at which the strength was retained, formability problems were greatly increased. Fabrication operations were marginal and yields were low. However, improved methods of hot working were developed, and in 1951, a modification of Nimonic 90 having increased titanium and aluminum levels was announced. This was called Nimonic 95.

Although work by Gresham, Dunlap, and Wheeler in Great Britain during 1948-1950 showed the beneficial effects of molybdenum additions (18), it was not until 1955 that a molybdenum-containing British nickel-base superalloy made its appearance. This alloy, called Nimonic 100, was also hardened by aluminum and titanium additions. Thus, the status of superalloy development in Great Britain as of 1951 is illustrated by Nimonic 95,



a. Solubility of Titanium and Aluminum in 80-20 Nickel-Chromium (Ti/Al Ratio 2/1)



b. Solubility of Titanium and Aluminum in 60/20/20, Ni/Co/Cr Alloy (Ti/Al Ratio 2/1)

A-37353

FIGURE 4. SOLVUS CURVES FOR (a) NIMONIC 80A-TYPE COMPOSITION (NO COBALT), AND (b) NIMONIC 90-TYPE COMPOSITION (20 PER CENT COBALT)⁽¹²⁾

a nickel-chromium-cobalt alloy containing approximately 3.0 per cent titanium and 2.0 per cent aluminum.

The status of superalloy development in the United States as of 1951 can be illustrated by the Waspaloy, GMR-235 and Inconel 700 alloys. Work by Bieber (International Nickel Company), Theilemann (Pratt and Whitney Aircraft), and others resulted in alloys of the Nimonic 95 type, but containing molybdenum additions as well. One further significant change was evident in the Inconel 700 and GMR-235 compositions. Although the total aluminum and titanium content was similar to that in Nimonic 95, the titanium/aluminum ratio had been reversed from 3:2 to 2:3.

The status of superalloy development in the Soviet Union in 1951 was mainly a posture of catching up with the West. By 1951 they had adopted the British Nimonic 75 and Nimonic 80A compositions and renamed them EI435 and EI437, respectively.

During the 1951-1955 interval, the British developed Nimonic 100. The significant changes in this alloy were the addition of 5 per cent molybdenum and a definite reversal in the aluminum and titanium contents. Nimonic 100 contains approximately 5.25 per cent aluminum and 1.5 titanium, an aluminum/titanium ratio of about 4:1.

During this same time period, United States investigators developed a large number of alloys including Udimet 500, M-252, and Hastelloy R-235. Nearly all of these alloys were of the same general composition. M-252 was somewhat unusual by virtue of its high molybdenum content, 10 per cent. The minor constituents, e.g., boron, zirconium, manganese, silicon, etc., were studied with respect to their influence on properties. Processing variables probably received more attention than anything else during 1951 to 1955. Much was learned about the hot-working characteristics of nickel-base superalloys. The newer alloys were becoming so strong at high temperatures that it was necessary to have more knowledge of processing variables in order even to produce parts from some of the alloys, let alone to increase the percentage of yield. The benefits of vacuum melting were also beginning to be recognized during this period, and a good deal of basic work, mostly in relatively small furnaces, was done on establishing melting procedures.

Workers in the Soviet Union during 1951 to 1955 developed a boron-containing modification of EI437, called EI437B, and also did a great deal of experimental work on additions of the heavy elements, molybdenum and tungsten. According to Khimushin(19), EI617, a superalloy containing 7 per cent tungsten and 3 per cent molybdenum, became available in 1954. Alloys developed by the Soviets have differed from those of the Western

nations in three ways: (1) the use of considerable tungsten, (2) the complete lack of cobalt in nickel-base alloys, and (3) lower aluminum and titanium levels. The Soviets also use small amounts of vanadium (0.3-0.5 per cent) which they claim increases the formability of wrought alloys. It has no effect on the strength properties.⁽¹⁹⁾

During the last 5 years, 1955 to 1960, the British have developed two wrought alloys, Nimonic 105 and 115. Nimonic 105 is basically Nimonic 100 with a 3.5 to 4.0 per cent increase in chromium content to improve the high-temperature corrosion performance, particularly in the presence of sulfur-bearing compounds.⁽¹²⁾ Nimonic 115 is the latest in the Nimonic series. From the standpoint of availability, Nimonic 115 currently is in the transition stage between developmental and commercial. The alloy reportedly has excellent high-temperature properties, although no data have been released for publication. In addition, several casting alloys were developed by the British during the 1955 to 1960 period. Although each of the Nimonic's 75, 80, and 90 have had a modification tailored for casting purposes (named Nimocast 75, 80, and 90, respectively), superalloys specifically designed for castings, i.e., to be used in the as-cast condition, are a relatively new innovation for the British. Four such alloys are being perfected at present, i.e., Nimocast 258, M4VC, M6VC, and M8VC.⁽²³⁾ Nimocast 258 is available in either the air- or vacuum-cast form. M4VC, M6VC, and M8VC are vacuum cast, and are presently available only in experimental quantities.

During 1955 to 1960, many alloys were developed in the United States, e.g., Udimet 700, Unitemp 1753, René 41, Astroloy, Inconel 713 C, Inconel 717, Nicrotung, DCM, TRW-1800, and others. Some of these are wrought alloys, or at least workable, while others are used as-cast. The United States has been the leader in the development of superalloys to be used in the as-cast condition. Perhaps the major reason for this is the greater interest in vacuum melting in the United States. The use of large-capacity vacuum furnaces permits increased quality control and reliability of precision-cast parts on a volume basis.

Although foreign and domestic engine manufacturers have used castings for stationary turbine blades (nozzle guide vanes), rotating turbine blades (buckets) have been predominantly wrought. The thought that castings generally are less reliable than wrought products of comparable composition has tended to exclude the former from turbine-bucket applications. Because turbine buckets are subjected to both mechanical and thermal stresses, whereas the nozzle guide vanes are exposed to only the thermal, performance requirements are more stringent for buckets.

The early United States interest in casting-type superalloys has brought forth a number of superior high-temperature casting alloys. Table 3 and Figure 6 show that the cast alloys, as a class, have the higher

stress-rupture properties at temperatures above 1500 F. The principal reason for this is that greater amounts of those elements which promote solid solution and precipitation strengthening are permitted in cast alloys. Plastic deformation is not necessary in the fabrication of parts; therefore, hot formability is not a problem.

Two alloys containing tungsten were developed in the United States during 1955 to 1960. One was a wrought alloy, Unitemp 1753, and the other was for castings, Nicrotang. Both alloys contain approximately 8 per cent tungsten. Late in the 1955 to 1960 period, the International Nickel Company developed a precipitation-hardenable alloy which relies, for its strength, on a nickel-columbium intermetallic compound rather than on gamma prime or eta. This alloy is known as Inconel 718. Early data reveal that the alloy contains approximately 5.0 per cent columbium and only small amounts of titanium and aluminum. (20) Although the strengthening second phase has not been positively identified, it is suspected to be the Ni_3Cb compound or a solid-solution based on this compound. The strength properties of Inconel 718 are most attractive at 1400 F and below. Thus, the temperature range of useful strength properties of the alloy is on the lower fringe of the temperature range usually associated with nickel-base superalloys. However, the alloy is significant because the hardening compound is based on columbium rather than on the usual aluminum or titanium or both. The sluggishness of the precipitation of the nickel-columbium compound alleviates many of the welding, pickling, and annealing problems usually associated with precipitation-hardenable nickel-base superalloys.

The Soviets, during the 1955 to 1960 period, continued their development of tungsten-containing superalloys. EI826 was developed in 1956 and was the result of further work on the EI617 composition. (19) The EI826 alloy, in fact, is sometimes referred to as EI617AB. During this period the Soviets also became interested in casting alloys. Although no new compositions were developed specifically for casting, a modified version of EI 617, designated ZhS3, was designed to be cast. Recent Soviet literature has emphasized another casting alloy, ZhS6. Although the composition of this alloy is unavailable, the pattern of Soviet alloy development suggests that ZhS6 probably is a modification of EI826, possibly containing a higher level of aluminum. The Soviet alloys EI652 and VZh 98 were developed as high-temperature sheet materials. Both alloys have relatively high chromium contents, 27 per cent. By virtue of the 3 per cent aluminum addition, EI652 should be precipitation hardenable. VZh 98, on the other hand, is solid-solution hardened by the addition of 14 per cent tungsten, further proof of the Soviet interest in tungsten for use in high-temperature alloys.

Tables 2 and 3 list the chemical compositions and typical elevated-temperature strengths of nickel-base superalloys that are representative of the many in current use in a variety of applications. To include the entire list of known nickel-base superalloys would make the tabulation unwieldy. Instead, alloys were chosen which reflect types of application and represent evolutionary sequences. The list includes selections from British, Soviet, and United States alloys.

Table 2 shows the organized, economical manner in which the British have undertaken alloy-development programs. Each alloy represents a logical step or sequence in temperature of application. The apparent neatness of the British program probably is a reflection of the fact that one organization, Mond Nickel Co. Ltd. and its affiliates, has assumed the major portion of the initiative in alloy development and marketing of nickel-base superalloys. Development programs in the Soviet Union got off to a later start. The systematic evolution and lack of duplication of effort in the Soviet program suggest that alloy development may have been regulated by the state. The competitive economic system of the United States has resulted in the development of a large number of superalloys, many of which are similar in composition. This duplication of effort has not been wasteful, however, because it has resulted in the quickest actual utilization of the strongest alloys. The recent trend in the United States toward the increased use of the practice of licensing the production of trademarked alloys has reduced the duplication of effort.

Table 3 summarizes available typical tensile-strength and stress-rupture data for the alloys listed in Table 2. Figure 5 is a plot of the typical tensile-strength data from room temperature to 1800 F for these alloys, and Figure 6 is a plot of the stress-rupture properties of selected alloys at 900 C (1650 F). This latter temperature was chosen for two reasons: (1) it is characteristic of the bucket temperature in many present-day turbines for jet aircraft, and (2) it is the highest temperature for which data are available on most of the foreign alloys. The use of typical values when comparing strengths is sometimes misleading, and the data presented should not be interpreted as guaranteed minimums. But, in the absence of guaranteed minimum values for most of the alloys, it was deemed necessary to present the data in this form.

It should also be mentioned that in the case of René 41 and Unitemp 1753, optimum properties were used in Table 3 and Figures 5 and 6. As described earlier in the section on Heat Treatment and Grain-Boundary Behavior, the strength properties of nickel-base superalloys are greatly affected by heat treatment. Thus, where tensile strengths are reported here for René 41 and Unitemp 1753, they represent the values obtainable when these alloys are heat treated for maximum tensile strength. Where stress-rupture values are reported, they represent values obtainable when

TABLE 2. NOMINAL COMPOSITIONS OF REPRESENTATIVE NICKEL-BASE SUPERALLOYS

[illegible]

11 The first-reviewed form is considered the most pertinent of those available.

(d) The intermetallic compounds of boron and zirconium, these elements are not included in the "selling elements."

(5) Estimated

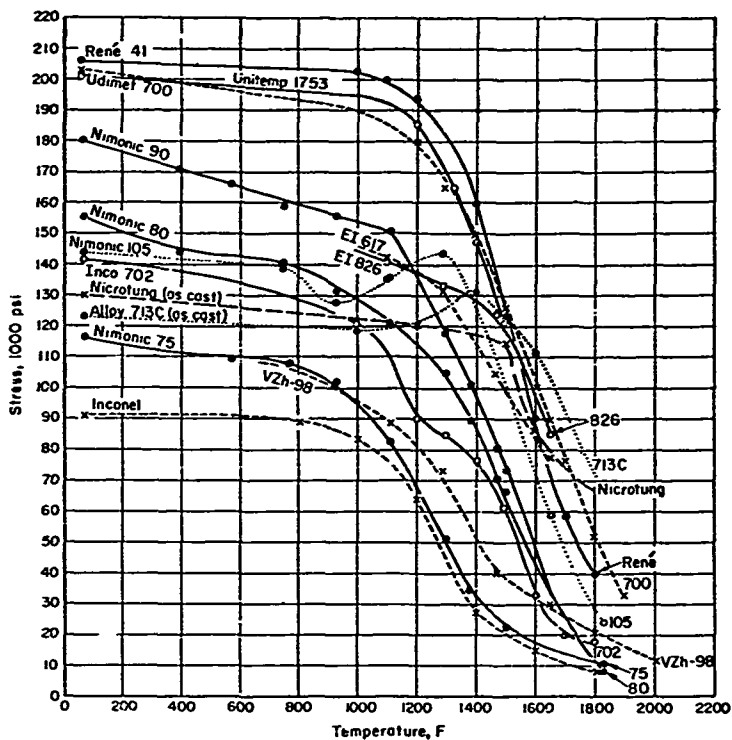


FIGURE 5. TYPICAL ULTIMATE TENSILE STRENGTHS OF SOME NICKEL-BASE SUPERALLOYS

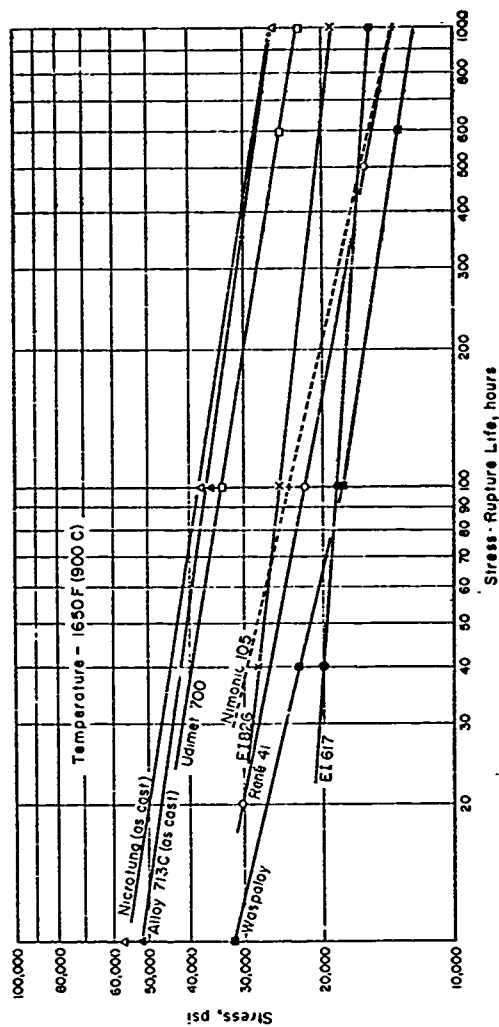


FIGURE 6. TYPICAL STRESS-RUPTURE PROPERTIES OF SOME NICKEL-BASE SUPERALLOYS AT 1650 °F (900 °C)

René 41 and Unitemp 1753 are heat treated for maximum stress-rupture strength. One should not expect to obtain both optimum tensile strength and optimum stress-rupture strength in the same part.

Figure 6 illustrates the superior stress-rupture strengths of the cast alloys at 1650 F, and the apparent greater stability, with time at temperature, of the Soviet tungsten-containing alloys. The latter is illustrated in Figure 6 by the comparison of the slope of the curves for EI617 and EI826 with those of René 41, Nimonic 105, and Waspaloy. Although the rupture stress of the earlier Soviet alloy EI617 is about the same as that of the earlier United States Waspaloy at 100 hours, there is considerable spread at 500 and at 1000 hours. The same holds true for the later Soviet alloy EI826 in comparison with the later United States and British compositions of René 41 and Nimonic 105.

The aircraft gas-turbine engine has been in existence for 20 years. Design developments within this time period have pushed the operating temperatures steadily upward. In the early stages of development, the temperatures of turbine buckets were in the neighborhood of 1500 F; today they are 1800 F. At 1500 F, alloys of relatively simple composition could adequately handle the stresses and corrosion conditions within the engine. However, the operating conditions have become more severe as the power requirements have increased. Increased speeds result not only in higher temperatures within the engine, but also in higher temperatures for the skin of the vehicle as the result of aerodynamic heating. Because of the latter circumstance, nickel-base superalloys have been used as the primary structural materials for a few of the later high-speed vehicles such as the North American X-15 rocket research plane. The higher metal temperatures encountered as power requirements are increased have focused the research metallurgist's attention on four major items:

- (1) Increasing, or at least maintaining, the effectiveness of the precipitation-hardening mechanism at ever higher temperatures
- (2) Increasing the contribution of solid-solution hardening to the strength properties
- (3) Increasing recognition of the high-temperature corrosion problem.
- (4) Increasing our understanding of the role of carbides.

Item (1) has been approached by the addition of cobalt and by the addition of greater amounts of aluminum and titanium (Figures 4a and 4b). The solvus temperature increases with increasing amounts of these elements. The addition of the heavy elements molybdenum and tungsten tend to slow down diffusion rates and hence the rate of precipitation. This should prolong the time that a precipitate is effective at a given temperature.

Item (2) becomes of more importance as the operating temperature approaches the solvus temperature of the solute in the alloy because a lesser volume of second phase will be available for strengthening. More aluminum and titanium will be in solid solution and less will be combined with nickel in the form of strengthening intermetallic compound. For this reason, solid-solution strengthening becomes more important as operating temperatures approach the solvus temperature.

Item (3) also becomes of increasing importance as operating temperatures increase. Nickel-base superalloys have inherently good oxidation resistance from the standpoint of general attack as measured by weight gain or loss. As the service temperature approaches 1800 F, however, intergranular oxidation becomes a serious problem, particularly in thin sections such as sheet and the trailing edges of turbine buckets. Figure 7 shows the depth of intergranular oxidation penetration of a typical alloy (René 41) as a function of temperature. In applications where thin section sizes are required, intergranular penetrations of 1 mil (.001 inch) are significant. Figure 7 shows that at a temperature of 1800 F, a penetration of 1 mil in depth is obtained in approximately 5 to 6 hours. Maiamand and Vidal have shown that preferential oxidation of titanium, aluminum, and chromium was the principal cause of intergranular attack.⁽¹²⁾ These authors made spectrographic determinations of the chromium, aluminum, and titanium contents at various depths beneath the surface of a typical nickel-base superalloy (Nimonic 80A) that had been exposed for 300 hours at one of various temperatures in air. Their data showed that chromium, aluminum, and titanium were depleted from the area under the surface. Some investigators feel that aluminum is preferable to titanium in the newer superalloys designed to meet the ever-increasing operational temperatures. Increased aluminum content not only decreases the tendency to form eta phase, which overages more rapidly than does gamma prime, but it also is thought to render the alloy less susceptible to intergranular oxidation. At elevated temperatures, both aluminum and titanium contribute to the uniform, over-all oxidation on the surface of an alloy. The rate of intergranular oxidation is thought to be dependent upon the diffusion of oxygen through the surface oxide to the oxidation-susceptible grain boundaries. The denser oxide formed by aluminum probably aids in retarding the diffusion of oxygen through the surface oxide, thereby inhibiting intergranular penetration.

Item (4) is of special importance with the increased use of superalloys in sheet form. Most investigators believe that the location and form of the carbides have a great effect on ductility. The "least undesirable" form is globular $M_{23}C_6$ at the grain boundaries. In this form, the carbide acts less as a "crack raiser" and tends to limit the cracks to within the intercarbide area.

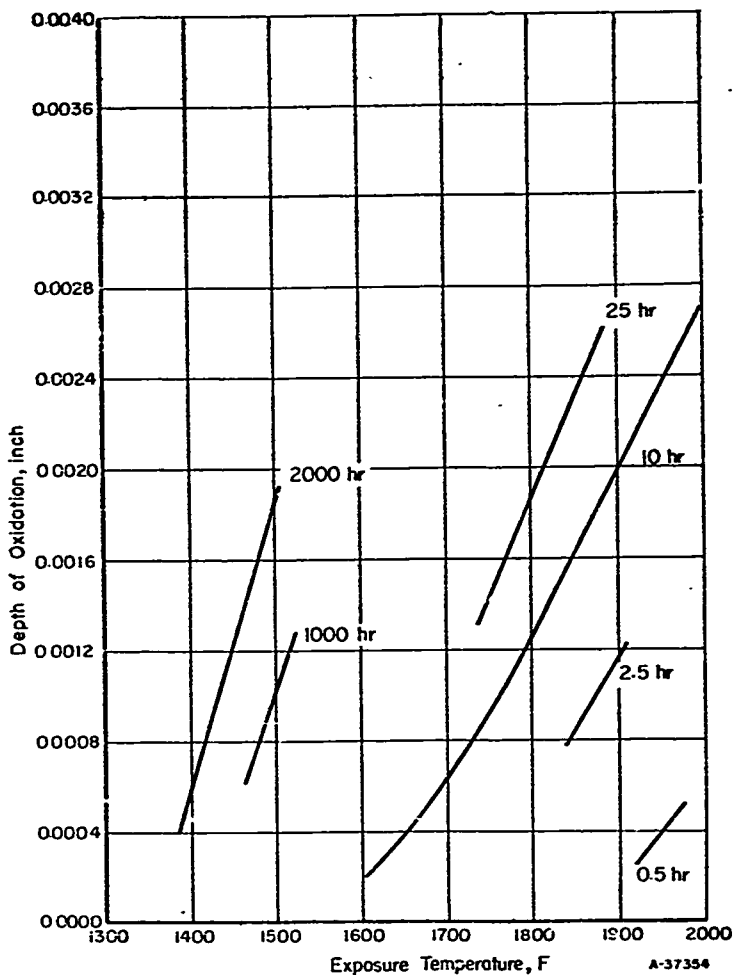


FIGURE 7. DEPTH OF INTERGRANULAR OXIDATION VERSUS EXPOSURE TEMPERATURE FOR RENE' 41(21)

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