STRUCTURAL METALS TO 1800°F

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A NEW LOOK AT SUPERALLOYS

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The past history of superalloy development indicates that perhaps the title would be more appropriate as "Another Look at Superalloys." Each time it was felt that everything worthwhile had been accomplished, major new improvements came about to rejuvenate interest in superalloys. In 1945 maximum operating temperature at high engineering stress levels was 1550°F, in 1950 the limit rose to 1700°F, today 1800°F, and the maximum limit has not yet been reached. Credit must be given to industrial organizations for most of the latest improvements in superalloys, since almost all Government supported research was dropped about 1954. Most of the improvements in composition and mechanical properties have been achieved by improved melting, casting, and fabrication techniques.

The very definition of "Superalloy", "an alloy developed to withstand relatively high stresses at high temperatures, a condition wherein good oxidation resistance is frequently required", reveals their applicability in the space program. These alloys were initially developed for aircraft gas turbine components operating in the range 1200-1300°F, and today the evolution of superalloys permits satisfactory service with blade temperatures at 1750°F. Many of the alloys have been adapted for production in sheet form for structural and skin applications. Wrought superalloys have also been developed specifically for sheet-metal applications; a prime example familiar to most of us is the use of Inconel X as the skin material for the X-15 rocket plane.

The superalloys are divided into three groups: (1) Nickel-base; (2) Cobalt-base; and (3) Iron-nickel-chromium-base alloys. They are particularly applicable in the temperature range 1200-1800°F, where they excel in strength-to-weight ratio and oxidation resistance, both characteristics considered to be of prime importance for space application. These alloys have become available in the wide variety of forms necessary to fulfill the needs of structural components for missiles and space vehicles for relatively short time application, and ramjet engine components such as turbine wheels and blades or fuel elements for reactors. The latter uses place emphasis on long time properties such as good creep, stress-rupture, and oxidation resistance for continuous or intermittent operation at high temperatures and relatively high stresses. Figure 1 demonstrates quite vividly the role of superalloys in the temperature range 1200-1800°F. The superalloy band includes wrought and cast alloys, with the wrought alloys (top part of the band) having higher strengths initially but not enjoying as much stability at elevated temperatures. Wrought alloy strength begins to drop around 1200°F, whereas cast alloy strength (lower part of the band) is maintained close to 1400°F.

Nickel-Base Alloys

Nickel-base alloys have evolved from the simple Nichrome V (80 Ni-20 Cr) alloys to the complex precipitation-hardening present day alloys. Table 1 illustrates the changes in chemical composition achieved through alloy development and these can be correlated with improvements in mechanical properties.

The Nimonic Alloys have been developed by the British in response to increased needs for gas turbine engines. "Nimonic 80" was developed in 1940 and "Nimonic 105" in 1958. "Nimonic 80" introduced precipitation-hardening nickel-base superalloys. Research and development in the United States paralleled the British work on superalloys with "Inconel X", basically the same as "Nimonic 80", and the complex superalloys such as Waspaloy, Unitemp 1753, the Udimets, Astroloy, and the high strength casting alloys such as Haynes Alloy 713C, Nicrotung, and IN-100 have been the most recently developed.

The changes in chemical composition during the development of these alloys involved the addition of cobalt, aluminum and titanium, and shifting the ratio of these two, refractory elements (Mo, W, Ta, etc.), and minor additions of boron and zirconium.

The nickel-base superalloys are strengthened by three different mechanisms: (1) solid solution hardening; (2) precipitation hardening; and (3) the effects of boron and zirconium additions. Chromium contributes to the excellent oxidation resistance of these alloys, raises the recrystallization temperature, and strengthens nickel by solid solution hardening. Aluminum and titanium strengthen the matrix material by precipitation of the gamma prime (γ'), $Ni_3(Al, Ti)$ complex. Increasing the aluminum also gives the added benefits of decreasing the density and contributing to the excellent oxidation resistance. Cobalt increases the solvus temperature, thereby permitting retention of the precipitate out of solution to a higher temperature, and also increases the solubility of carbides in the matrix. Refractory metal additions contribute to solid solution strengthening and carbide formation, and thereby improve the creep-rupture properties of superalloys. The addition of limited amounts of boron and zirconium improve the stress-rupture properties of these alloys. Excessive amounts of these elements have proven to be detrimental in that a brittle network forms in the grain boundaries. Boron is more effective than zirconium and a combination of both has proven to be most beneficial. These alloying elements retard the effects of carbide and γ' precipitate agglomeration at the grain boundaries from late in the initial stage of creep until the start of the third stage. Boron and zirconium atoms are believed to occupy voids in the grain boundaries and impede dislocation movement. The overall effect is an increase in rupture life and ductility.

Figure 2 illustrates the effect of temperature on the short time tensile strength of wrought and cast nickel-base superalloys. The curves support the previous discussion of the strength/weight plot with respect to strength-temperature stability of cast vs. wrought products. The mechanical properties can be correlated with the above discussion on composition. The highest strength wrought alloys from room temperature to approximately 1100°F are Udimet 700, Unitemp 1753, Astroloy, Udimet 500, and Rene 41 in approximate order. At about 1200°F the order changes slightly showing Astroloy and Udimet 500 to be slightly superior and at elevated temperatures (1800°F) the strengths of all alloys have dropped approximately 100,000 psi with Astroloy displaying some superiority over Udimet 700. Inconel X has been included to show the improvement that has been achieved in recently developed alloys.

Much the same condition exists in the cast alloys. The most recently developed IN-100 and NASA TN-D-260 demonstrate superior strength properties to Inco 717C, Nicrotung, Inconel 713C, and Udimet 700 at elevated temperatures (1800°F). The drop in strength of the cast alloys does not occur until approximately 1400°F as compared to 1200°F for the wrought alloys. Cast alloys also remain slightly stronger between 1600-1800°F.

A review of table 1 reveals that the higher strength wrought alloys contain 15-20 Cr, 6-20 Co, 2.5-8 (Al + Ti) in varying ratios, one or more refractory elements, .003-.1 B, .02-.1 Zr, and 1-11 Fe. The complexity of these alloys introduces several of the strengthening mechanisms discussed previously and is responsible for their high temperature strength. On the basis of chemical composition, it sometimes becomes difficult to distinguish between cast and wrought alloys; however, the cast alloys are lower in chromium, generally considerably lower in cobalt, and higher in aluminum + titanium with the ratio in favor of aluminum. They also generally contain more than one refractory alloying addition (Mo, W, Cb, V, Ta). These alloys are usually used as cast, whereas a variety of heat-treating cycles have been established to impart the specific characteristics desired for service of the wrought alloys.

Cobalt-Base Alloys

Unfortunately, cobalt-base superalloys have not been developed at a rate comparable to the nickel-base alloys. A partial explanation for this may be the lack of Government support and the limited availability of research and development funds in industry. The cobalt-base superalloys developed initially for application as precision cast gas turbine blades have demonstrated excellent creep and rupture properties and good oxidation resistance up to 2000°F. Consequently, these alloys have found application in high-temperature gas engine wheels and blades, turbo superchargers, and power plant equipment.

Cobalt-base alloys contain an appreciable amount of chromium (18-20 Cr) and carbon (0.2-0.6 C), and derive their strength chiefly through the precipitation of chromium-carbide. However, the presence of refractory metals such as tungsten, columbium, and molybdenum also contribute to their strength through solid solution hardening. Nickel is employed primarily to stabilize the high temperature crystallographic structure (FCC) form of cobalt. Table 2 gives the composition of some more recently developed cobalt-base superalloys and HS-25 (L-605) and Stellite 31, two earlier alloys. The addition of boron has been found to improve the elevated temperature mechanical properties of the cast alloys, and titanium additions to wrought alloys have improved high temperature strength.

Figure 3 illustrates the effect of temperature on the strength of cobalt-base superalloys. A similar correlation can be made between composition and mechanical properties as was done previously for the nickel-base alloys. The same holds true for comparison of wrought and cast alloys, as can be expected. Although it is readily apparent that the cobalt-base alloys are lower in strength than the nickel-base superalloys, one must also be cognizant of the fact that, particularly in the cast alloys, the cobalt-base alloys are more stable in the temperature range 1400-1800°F. It is this characteristic of the cobalt-base alloys which makes them more useful at temperatures in excess of 1600°F for longer time applications.

Iron-(Ni-Cr and Cr-Ni)Base Alloys

This class of alloys has suffered most in the superalloy development program. Possibly the lack of interest is due to less glamorous strength properties than those of nickel or cobalt-base alloys, but for applications requiring thermal shock resistance and corrosion resistance to oxidizing and reducing atmospheres they are outstanding.

The chemical composition of these alloys will determine their metallurgical structure and properties. The strongest and toughest alloys are austenitic with various chromium-nickel combinations dictated by service specifications such as high-temperature strength required, resistance to carburization and corrosion, minimum ductility acceptable, tolerances, and cost. The basic strengthening mechanism is carbide precipitation, and the alloys are responsive to heat treatment. Properties such as corrosion resistance, creep strength, and ductility can be controlled by composition (carbon content) and heat treatment to redistribute or alter the shape of the carbides.

Figure 4 shows the effect of temperature on some typical alloys in the iron-nickel-chromium-base group. D-979 contains 45 Ni and can be considered a nickel-base alloy with an appreciable (30 percent) iron content plus molybdenum, tungsten, aluminum, and titanium. The RA-330 and Incoloy T (modification of "Incoloy", Fe-Ni-Cr Alloy) are annealed sheet, and the N-155 (AMS-5376), and iron-nickel-chromium alloy with 20 cobalt, is in the as cast condition. Note that at elevated temperatures the strength of this group of alloys is considerably below that of the two classes of alloys discussed previously.

Stress-Rupture Properties

The discussion of the three alloy systems comprising the superalloys has practically been limited thus far to short time tensile strength at various temperatures and briefly on corrosion resistance. For continuous or intermittent operation for extended periods of time at elevated temperatures, however, creep and stress-rupture properties become the controlling factors.

In the case of nickel-base alloys, the complex compositions permit a wide variance in properties by use of different heat treatments. Optimum heat-treating cycles have been established for a given alloy, dependent upon the specific properties desired. One heat treatment can be specified for maximum short time tensile strength, while another produces optimum creep properties. In the case of the Nimonic alloys, heat treatments have been specified to produce maximum ductility at creep failure. Each heat-treating cycle is dependent upon composition and cannot be used interchangeably between various alloys. There are four basic ingredients in nickel-base superalloys that are affected or interact with the critical heat-treating conditions:

1. Solid solution matrix (gamma)

2. Ni_3 (Al, Ti) gamma prime and Ni_3Ti beta precipitate

3. Carbides (M_6C, $M_{23}C_6$, M_7C_3, TiC)

4. Nitrides (TiCN, CbN)

The basic heat-treating cycle consists of solution treatment followed by aging. From the standpoint of structural stability, the choice of aging temperature is of the utmost importance, and service temperature is the prime consideration.

Figure 5 compares the stress-rupture life of several superalloys in the wrought and cast condition. As has been mentioned previously, although nickel-base alloys are considerably stronger, the cobalt-base alloys such as X40 (HS-31) are more stable at elevated temperatures. This can be expected due to the strengthening mechanism and the aging phenomena involved; the sluggish reaction of the precipitation of carbides as compared to the precipitation of gamma prime, $Ni_3(Al, Ti)$ in the nickel-base alloy systems. Rupture data for the new NASA developed casting nickel-base alloy is not included but is expected to exceed IN-100 slightly. The alloy is not being produced commercially at this time. In the wrought alloys Nimonic 115, one of the latest nickel-base alloys developed by Mond in England, is equal to Udimet 700 in stress-rupture properties.

Thus far, we have discussed briefly the state of the art of superalloys, and merely touched upon applications, problem areas, and anticipated areas of research and development. It is a foredrawn conclusion that the contemplated applications and problem areas will dictate the research and development needs, and map out the course to be taken.

For several years, the continued development of wrought-age-hardenable superalloys has supplied the designer with jet engine materials capable of meeting the requirements at high service temperatures. Recently, however, the designers have become hampered by materials limitations and further improvement is deemed mandatory. Application of these materials has expanded to structural components in airframes and skins and fuel elements, in addition to engine components. Increased acceleration, space environments, and re-entry phenomena have increased the performance requirements of these materials. Materials limitations therefore spearheaded the search for new and improved alloys, but research and development was curtailed somewhat in range due to lack of Government support and limited research funds of private organizations. Nevertheless, great strides have been made, particularly in cast age-hardenable nickel-base superalloys. Alloys inherently stronger in creep-rupture and elevated temperature strength than the corresponding wrought composition have been developed. The pros and cons of wrought versus cast alloys will not be discussed other than to mention that it is generally felt that wrought alloys have increased reliability, shock resistance, and ductility.

One of the major requirements in superalloy development is to develop materials capable of covering the temperature gap between 1800°F and 2000°F. Most superalloys have satisfactory oxidation resistance up to 2000°F and can be coated to increase corrosion resistance to temperatures up to 2300°F. The drastic drop in tensile strength and creep resistance limits their use, however, above 1750-1800°F for wrought alloys and 1800-1900°F for cast alloys. The Air Force is sponsoring a program directed toward developing an alloy or technique capable of producing a wrought material (sheet) with a minimum short time tensile strength of 50,000 psi at 1900°F. For short time applications, strengths in the 45,000-50,000 psi range at 2000°F are considered adequate. A more detailed listing of requirements follows:

AREA I Cheaper and easier to make superalloys for high stress, high temperature applications (2000°F)

- (a) Improved thermal shock resistance of nickel-base alloys.
- (b) Improved strength cobalt and iron-nickel-chrome-base alloys.
- (c) Wrought alloys with strengths and stress-rupture properties equal to cast alloys.
- (d) Casting alloys with a high level of reproducibility which do not require vacuum melting and casting techniques.

AREA II Development of alloys which are strengthened by mechanisms not dependent upon carbide or gamma prime precipitation in order to achieve greater strength-temperature stability.

(a) Investigation of dispersion hardened alloys.

AREA III Investigation of fabrication techniques and processes, and physical and mechanical property evaluation, to establish:

(a) An overall cost reduction in superalloy utilization.

(b) Optimum utilization of maximum mechanical properties by improved joining techniques.

(c) Comprehensive design criteria for the newest and best superalloys available.

Effort expended in Areas I and II should be directed toward reducing the composition complexity and precise controls exercised to achieve minimum specifications for currently available superalloys. The slack in development of cobalt and iron-base alloys should be picked up, and introduction of new alloy systems other than the three basic superalloy systems discussed should be contemplated. In this area, vanadium alloys show promise on a strength-to-weight basis, but impose the problem of catastrophic oxidation at elevated temperatures. Vanadium has the lowest density of the ten highest melting point refractory metals, exhibits good weldability and formability (ductility). The strength-to-weight ratio of V-5Ti-20Cb exceeds that of F-48 (columbium alloy) and Mo-0.5Ti-0.07Zr up to 2200°F and 2000°F, respectively. An attempt to develop chromium alloys for structural purposes is not feasible because of the poor ductility. Ductilizing chromium by a liquid phase sintering process should improve fabricability and joining characteristics, but most of the strength is sacrificed due to the low melting point matrix material. Use of chromium seems destined to remain as an alloy addition or as a coating for oxidation resistance.

In the area of dispersion hardening, there is a basic need to increase the state of technology on dispersion strengthened systems. Along these lines, the Air Force is sponsoring a basic program to investigate the strengthening mechanisms in such systems. An attempt will be made to uncover the basic differences in precipitation-hardening and dispersion-hardening systems, and to investigate the factors influencing the characteristics of dispersion strengthened alloy systems. This information, coupled with the ever increasing technology on dispersion hardening, will enable researchers to conscientiously formulate programs which will eliminate much of the trial and error method adopted since the development of SAP aluminum alloys. Powder metallurgy techniques previously considered somewhat unfruitful, to the point of hopelessness, with respect to the superalloy systems, should be revived. Recent unpublished information indicates the possibility of a nickel-chromium matrix strengthened by thoria to have high strength and stress-rupture properties comparable to some of the best superalloys.

Area III of the proposed requirements is of the utmost importance to the designer. Many of the recently developed alloys have not been fully evaluated from a structural point of view. Several of the commercially available high-strength superalloys cannot be used at their full potential except in the form of precision castings. Development of fabrication and processing techniques will increase their use for structural applications.

The composition complexity of these alloys and the strengthening mechanism involved introduce numerous problems in welding and brazing. Another factor is the limited workability of some of the highest strength wrought alloys such as Udimet 700 which can only be formed into the simplest configurations. Comprehensive investigations such as have been carried out on Rene 41, welding, brazing, and fabrication parameters, are needed.

Specific Air Force requirements emphasize the need for wrought alloys to produce sheet material. The direct aim is to achieve the greatest strength-to-weight ratio possible for the obvious reason of increasing performance characteristics of long range missiles and space vehicles. Weight savings mean longer ranges, faster speeds, and increased economy for every pound of fuel consumed. The use of thin gauge sheet materials will probably require composite structures such as honeycomb to supply the stiffness necessary to prevent buckling at very high stresses. In the temperature range 1400-2000°F, the superalloys have practically no competition as yet, but the possibility of developing titanium alloys in this range exists.

Another area of importance is oxidation resistance. It will be noted that strides have been made in increasing superalloy strength, but composition changes in the most recently developed alloys reveal a marked decrease in chromium content. In some cases, the chromium content has been reduced as much as 50 percent. The result has been a decrease in oxidation resistance. The slight increase in aluminum content reduces the density in some instances, but does not provide a sufficient increase in oxidation resistance to compensate for the decrease due to the reduction of chromium. Although coatings for superalloys may not present an exceedingly difficult challenge, nevertheless, at service temperatures of 1800-2000°F the need does exist. The requirement may be stated specifically, "to increase the oxidation and corrosion resistance at temperatures in excess of 1800°F." Coatings or composition changes may provide the solution.

The versatility of superalloys has contributed greatly to the defense effort. Prime examples are the use of Inconel X in the X-15, as mentioned previously; Rene 41, Waspaloy, Udimet 700, Inconel W, and A-286 in the B-70; and Rene 41 and Inco 702 in the Dyna-Soar re-entry vehicle project. These materials are being utilized in the form of turbine wheels and blades, trusses, tubing and fittings, airframe skin panels, reinforcing structures in elevated temperature areas, to mention a few applications.

Finally, industry cannot be expected to support research and development in these alloy systems completely on its own. This would inevitably provide short range research involving minimum risk and generally producing lesser benefits. Industry cannot afford to venture much capital into such long range plans as are mandatory from the Government's point of view. Such programs should be formulated on a cost-share basis for alloy development and longer range composition, structure, and property studies. One cannot fail to see the benefits which can be derived through such effort, whether viewing the situation through the eyes of the Department of Defense or through the eyes of industry.

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TABLE I

NOMINAL COMPOSITION OF VARIOUS NICKEL-BASE SUPERALLOYS

ALLOY	Cr	Co	Al	Ti	C	Fe	Mo	W	B	Zr	OTHER
Nimonic 80	18 - 21	2.0*	0.5 - 1.8	1.8 - 2.7	0.1*	5	—	—	—	—	—
Nimonic 90	18 - 21	15 - 21	0.8 - 2	1.8 - 3	0.1*	5	—	—	—	—	—
Nimonic 100	10 - 12	18 - 22	4 - 6	1 - 2	0.3*	2	4.5 - 5.5	—	—	—	—
Nimonic 105	13 - 16	18 - 22	4.2 - 4.8	0.9 - 1.5	0.2*	1	4.5 - 5.5	—	—	—	—
WROUGHT											
Inconel "X"	14 - 17	1.0*	.04 - 1	2.2 - 2.8	.08*	—	5 - 9	—	—	—	Cb, Cu, S
M - 252	18 - 20	9 - 11	.75 - 1.25	2.25 - 2.75	.1 - .2	5	—	—	.001 - .01	—	Cu, S
Waspalloy	18 - 21	12 - 15	1.0 - 1.5	2.6 - 3.25	0.1 *	2*	3.5 - 5.0	—	.003 - .01	.05 - .12	—
Rene 41	18 - 20	11	1.5	3.1	.09	—	10	—	—	—	—
Udimet 500	15 - 20	13 - 20	2.5 - 3.25	2.5 - 3.25	.15*	4*	3 - 5	—	.01*	—	S
Udimet 700	13 - 17	17 - 20	3.75 - 4.75	3 - 4	.15*	1*	4.5 - 5.75	—	0.1 *	—	—
Unitemp 1753	15.5 - 17.5	6.5 - 8.5	1.75 - 2.25	2.9 - 3.4	.2 - .28	7 - 11	1 - 2	7.5 - 9.5	.002 - .01	.02 - .1	S
Astroloy	15	15	4.2	4.0	.10	—	5.8	—	.025	—	—
CAST											
Haynes "713C"	11 - 14	1.0*	5.5 - 6.5	0.35 - 0.9	.08 - .2	2.5*	3.5 - 5.5	—	.005 - .02	.05 - .2	Cb, S
Inco "717C"	10 - 11	7 - 9	7.25 - 8.0	0.25 - 1.25	0.2 *	2.0*	3.5 - 5.0	—	.005 - .02	.05 - .2	Cb, S
Nicrotung	12	10	4.0	4.0	0.10	—	—	8	0.05	.05	—
IN - 100	8 - 11	13 - 17	5 - 6	4.5 - 5.5	0.15 - 0.20	1.0*	2 - 4	—	.008 - .02	.03 - .09	.7 - 1.2 V
NASA TN-D-260	6	—	6	—	0.15	—	4	4	—	1	2.5 V, 8 Ta

* Maximum

Nimonic series are English wrought alloys.

TABLE 2

NOMINAL COMPOSITION OF VARIOUS COBALT - BASE SUPERALLOYS

ALLOY	Cr	Ni	C	Mo	W	Ta	Ti	B	Co	OTHER
WROUGHT										
J - 1570	20	28	0.20	—	6	—	4	—	Bal.	—
J - 1650	19	27	0.20	—	12	2	3.8	.02	Bal.	—
CAST										
HS - 25(L-605)	20	10	0.15	—	15	—	—	—	—	2Fe, 1.5Mn, .5Si
Stellite 31	25.5	10.5	0.50	—	7.5	—	—	—	—	2Fe*, 1Mn*, 1Si*
Haynes Alloy No. 151	20	—	0.55	—	12.5	—	—	—	Bal.	—
WI - 52	21	1	0.45	—	11	4**	—	—	Bal.	2Fe, .5Si*, .5Mn*
S816	20	20	0.40	4	4	—	—	—	—	4Fe, 4Cb, 1.2Mn, .4Si
Mod. S816 + B	20	5	0.4	4	4	4**	—	1	Bal.	2Fe*, .4Si, 1Mn

* Maximum

** Ta + Cb

Balance of all alloys is nickel.

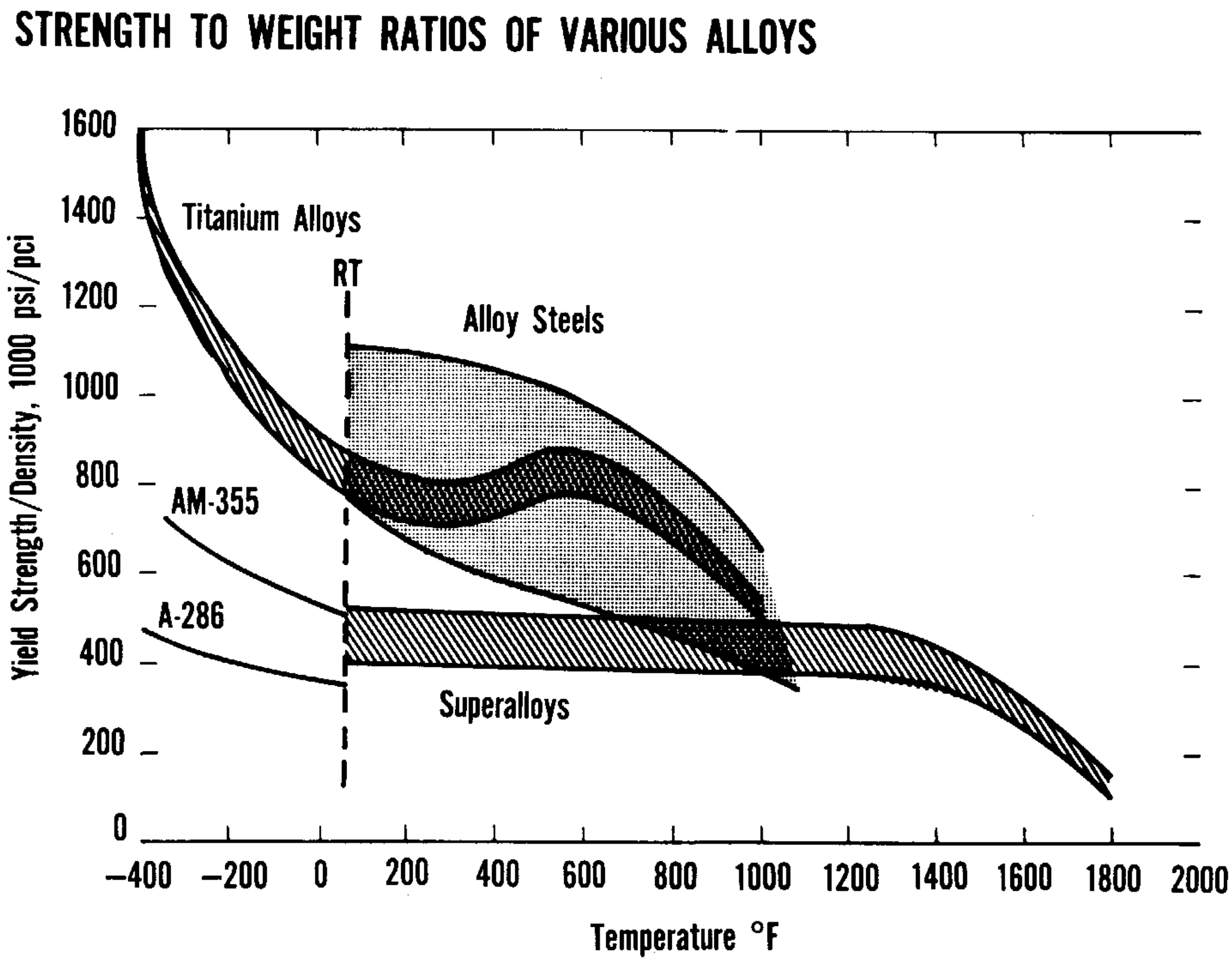

Figure 1. Strength to Weight Ratios of Various Alloys

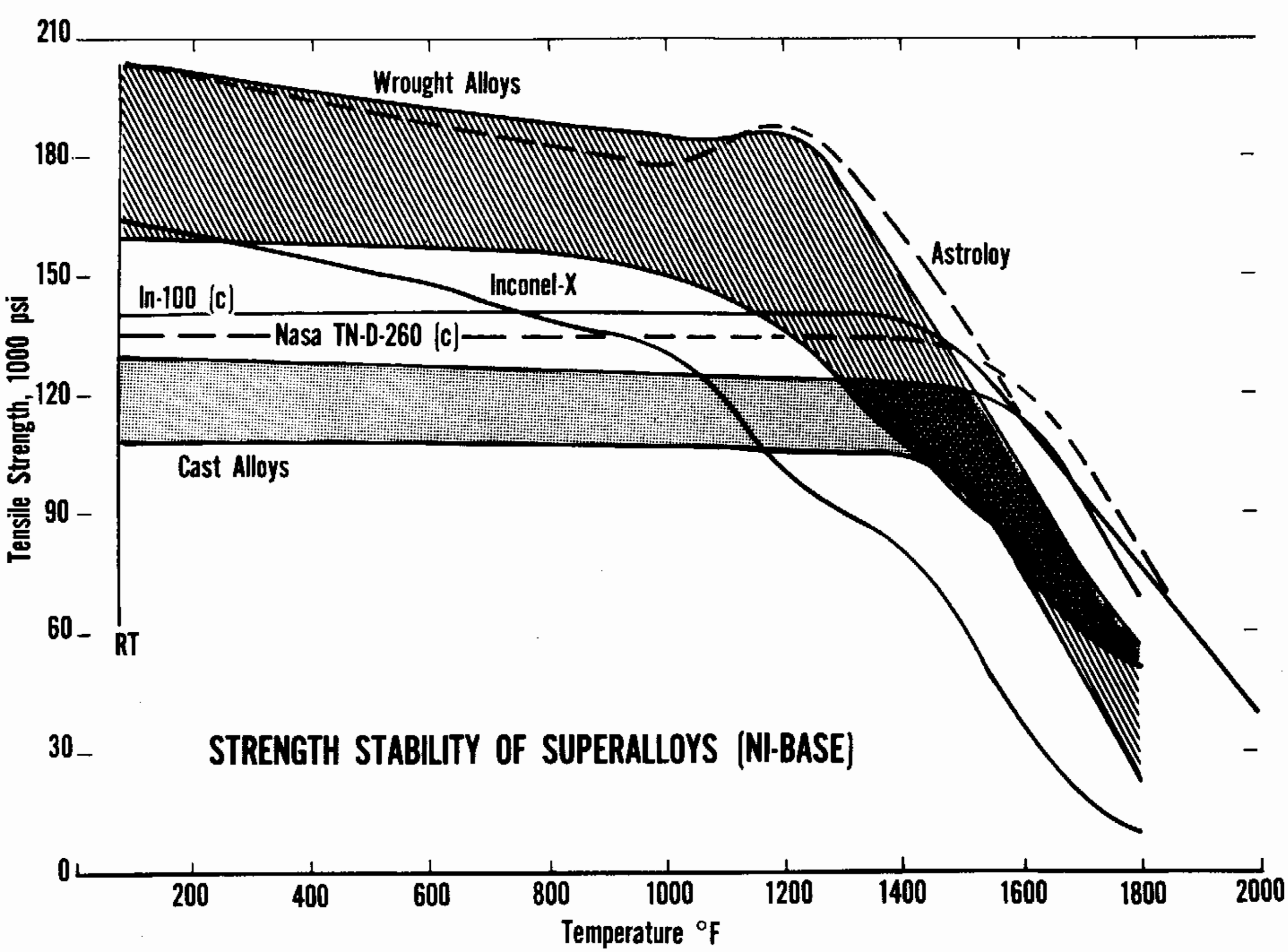

Figure 2. Strength Stability of Superalloys (Ni-Base)

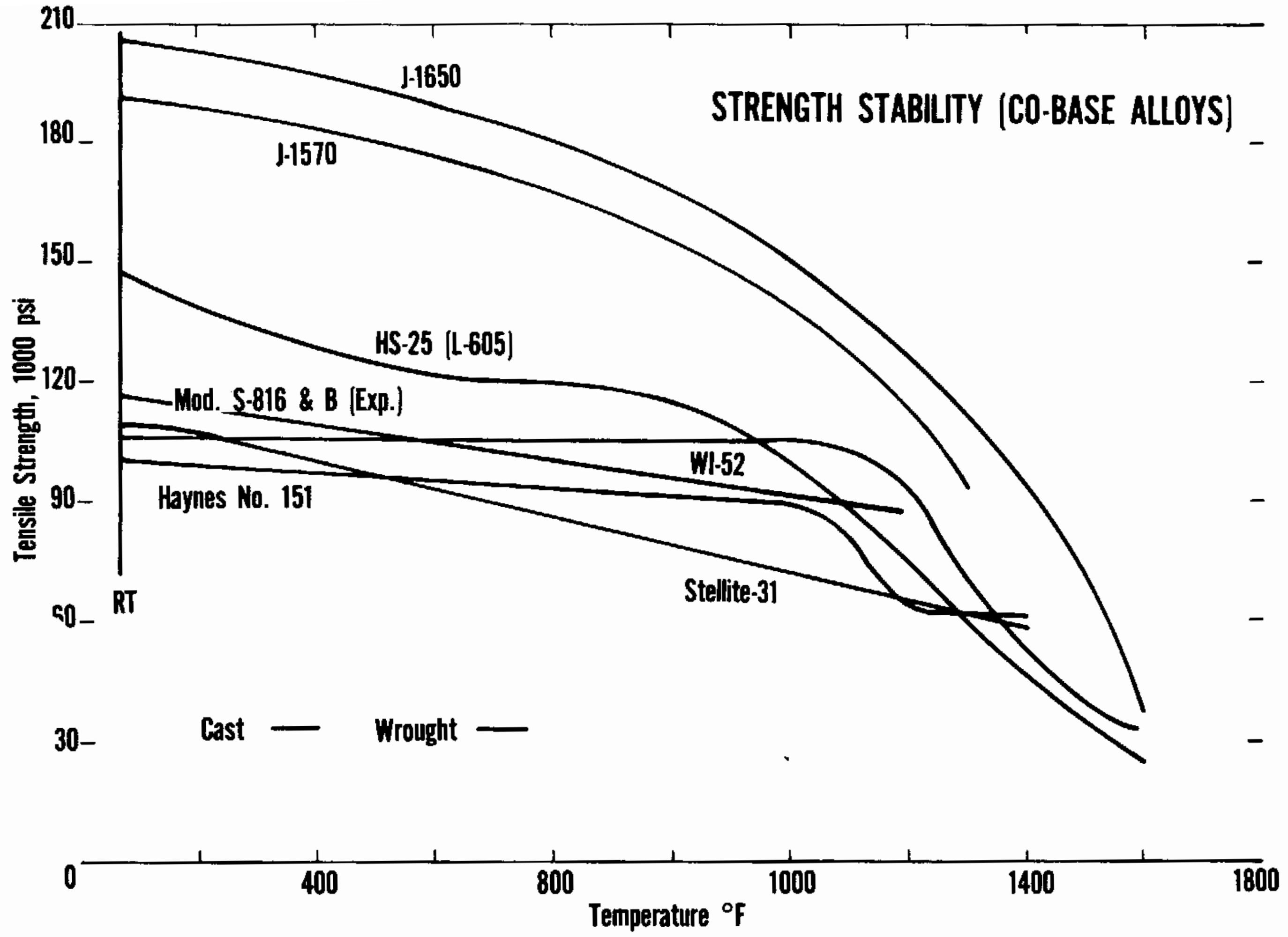

Figure 3. Strength Stability (Co-Base Alloys)

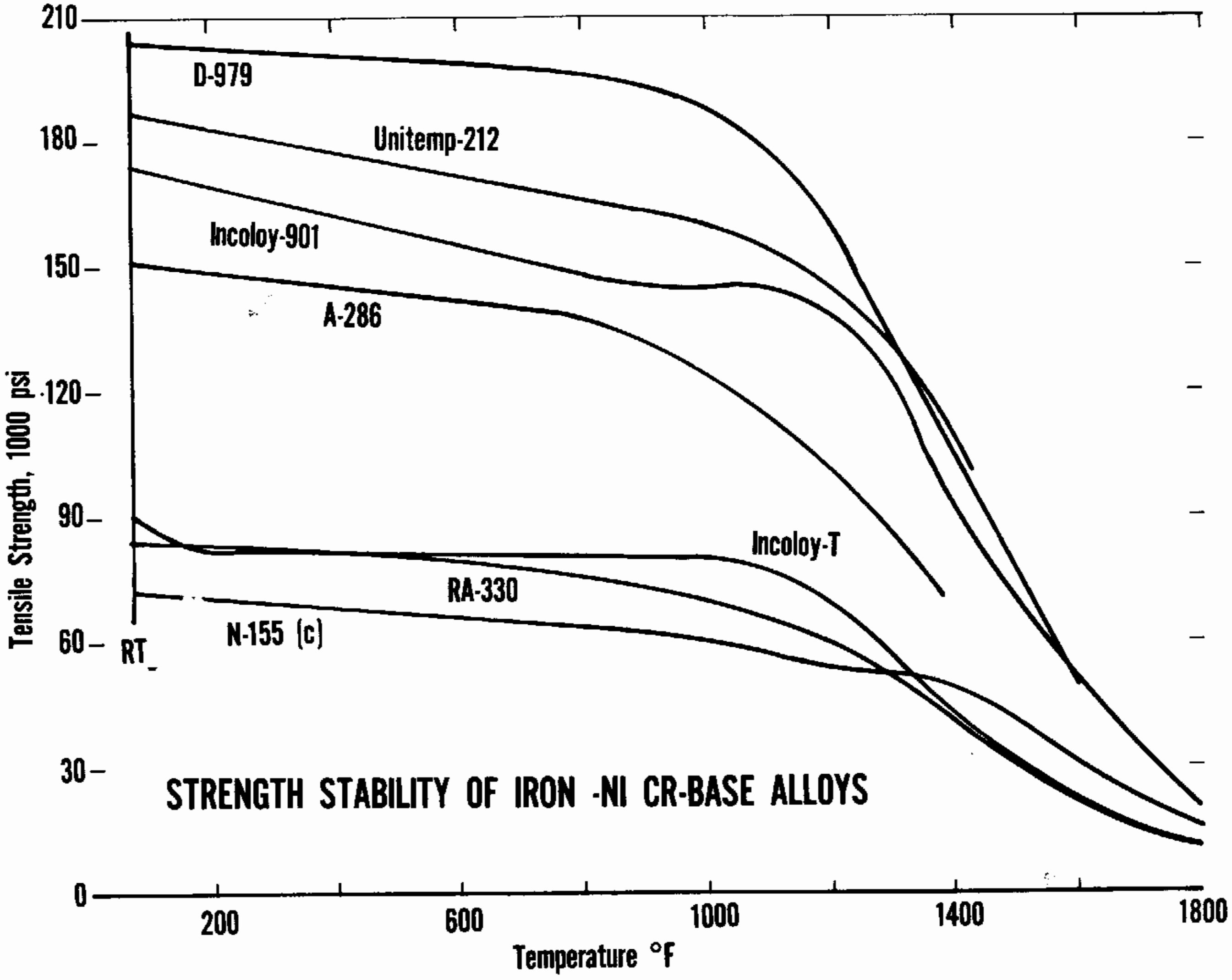

Figure 4. Strength Stability of Iron-Ni-Cr-Base Alloys

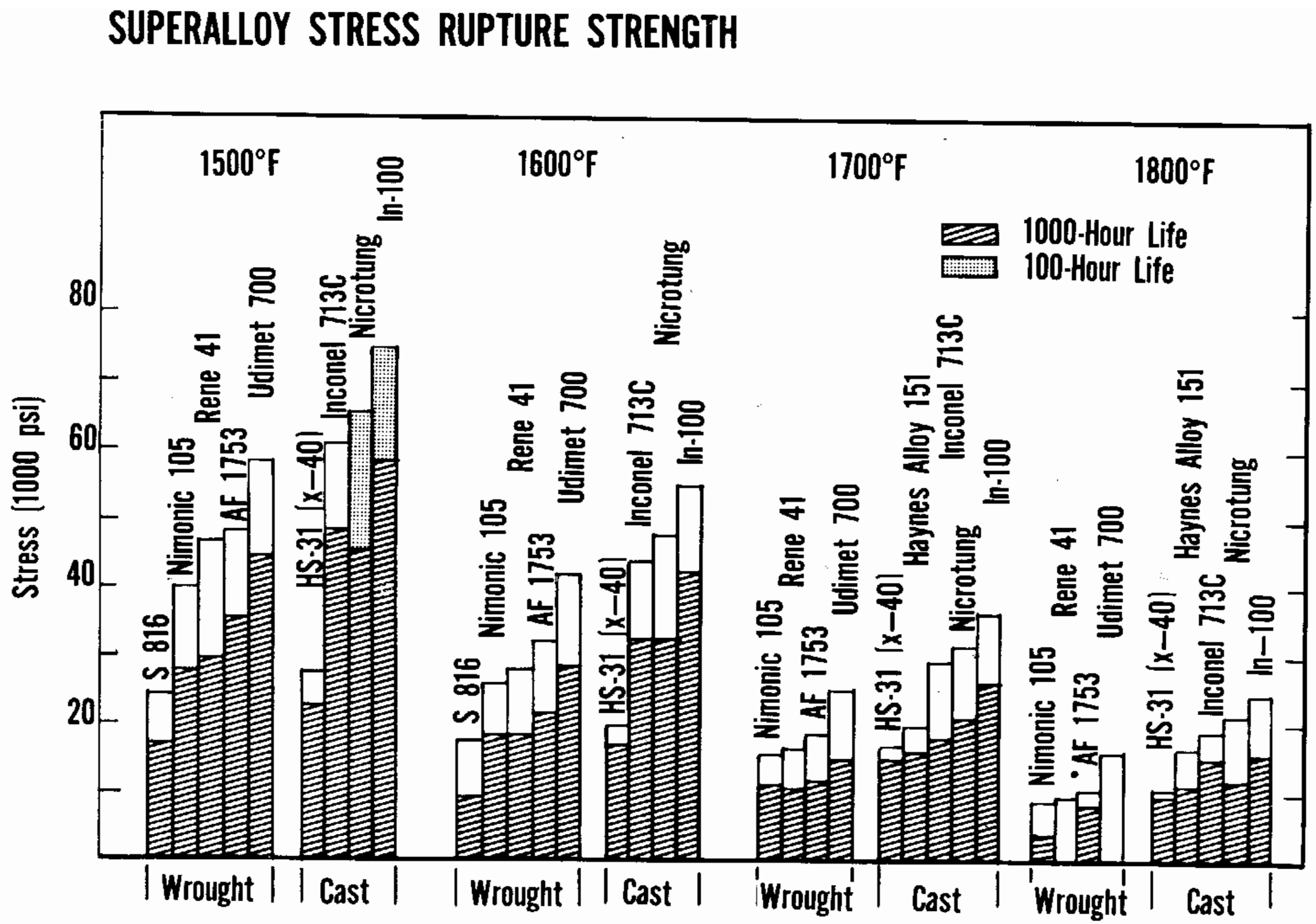

Figure 5. Superalloy Stress Rupture Strength