A SILICON-CONTAINING, LOW-EXPANSION

ALLOY WITH IMPROVED PROPERTIES

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Summary

INCOLOY* alloy 909 was developed from research which produced INCOLOY alloy 903 and INCOLOY alloy 907. Each development in this series of age-hardenable iron-nickel-cobalt low-expansion alloys brought progressive improvements in high-temperature properties. The most recent material, alloy 909, has excellent 538°C notch strength (resistance to the environmental failure mechanism) along with higher tensile properties resulting from a decreased dependence on overaging.

This paper compares the tensile, stress-rupture, low-cycle-fatigue, and crack-growth characteristics of the silicon-containing alloy with those of its predecessors. INCOLOY alloy 909 can achieve a yield strength of 1034 MPa with 538°C/100 h notch bar strength over 895 MPa. The combination of notch rupture properties and short-time tensile strength is the best available in useful low-expansion superalloys. Additionally, the properties are achieved with commercially attractive heat-treatment cycles. The substantial changes in microstructures brought about with silicon are discussed and related to the improved properties. The interaction of test environment, properties, and microstructure are also reviewed.

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Introduction

Superalloys with low, controlled thermal expansion are increasingly in demand for applications in aerospace and land-based gas-turbine engines requiring narrow clearances and close tolerances at high temperatures. The use of such alloys as INCOLOY alloys 903 and 907 for compressor casings, turbine seals, and other engine hardware has resulted in substantially improved fuel efficiency and has simplified major design problems. As shown in Figure 1, these alloys have coefficients of expansion of about half those of conventional superalloys. This paper describes a new alloy, INCOLOY alloy 909, that has properties superior to those of previously available low-expansion superalloys.

Figure 1. Thermal expansion of low COE alloys and conventional superalloys.

Background

Development of the low-expansion superalloys has centered around obtaining a good combination of short-term tensile strength and elevated-temperature notch rupture strength. Good tensile properties are easily attained, but good notch strength has proven to be an elusive property. The necessary omission of chromium to achieve these unique thermal-expansion properties reduces oxidation resistance and also leads to a marked sensitivity to the phenomenon here called Stress Accelerated Grain Boundary Oxygen embrittlement, abbreviated as SAGBO. Embrittlement occurs when the material is subjected to a combination of high temperature (particularly in the area around 540°C), stress, and the presence of oxygen in the environment (1,2). All three factors are routinely encountered in gas-turbine applications, making resistance to SAGBO highly desirable in materials of construction. Because of the SAGBO sensitivity of the low-expansion superalloys, notch rupture strength is often severely reduced. Several unusual, and not always successful, methods including shot peening and coatings have been used to combat the embrittlement problem.

Figure 2 charts the odyssey of low-expansion superalloy development with a plot of 100-h notch rupture strength ($K_t = 2.0$) at 538°C vs. room-temperature yield strength. INCOLOY alloy 903, the first and still by far the most used of low-expansion superalloys, has excellent longitudinal notch rupture and tensile properties when given proper thermomechanical processing (TMP). The processing, which requires massive warm-working followed by "annealing" at 843°C before age hardening, places a considerable hardship on forgers. The resultant microstructure, Figure 3, shows that alloy 903 is highly anisotropic after TMP. When properly thermomechanically processed, alloy 903 has transverse notch strength of less than one-half the longitudinal notch strength (331 MPa vs. 862 MPa).

Figure 2. Room-temperature yield strength and 538°C/100 h notch ($K_t=2.0$) rupture strength of low-expansion superalloys.

Figure 3. Microstructure of alloy 903 after thermomechanical processing and heat treatment. 100X.

But failure to use correct TMP, or applying recrystallizing anneals, leaves alloy 903 with poor notch strength regardless of orientation. In spite of these difficulties, INCOLOY alloy 903 has worked well in several applications. However, the above problems severely limit the alloy's usefulness.

The development of INCOLOY alloy 907 brought an improved combination of notch rupture and tensile strength along with a reduced dependence on TMP. Improvement was achieved by restricting aluminum to very low levels and by increasing titanium and columbium contents. However, to increase the 100-h notch strength to at least 862 MPa, it is necessary to over-age the alloy with uneconomical, long-time age-hardening cycles that result in a substantial loss in tensile strength (Figure 2). The heat treatment also produces a remarkably complex microstructure with abundant intergranular and intragranular needle precipitates (Figure 4).

Development of INCOLOY alloy 909

The recently developed, INCOLOY* alloy 909 is the first low-expansion superalloy to have an excellent combination of tensile properties, notch rupture strength, and low thermal expansion using conventional thermomechanical processing (TMP). The path to INCOLOY alloy 909 was a long and arduous one, but culminated in an unexpected solution: the addition of about 0.4% silicon to the composition of INCOLOY alloy 907 (see Reference 1). The composition of INCOLOY alloy 909 is listed in Table 1 along with the compositions of alloys 903 and 907.

Testing was conducted on laboratory-scale, vacuum-induction-melted heats containing up to 0.5% silicon and having the compositions listed in Table 2. The material was forged and hot rolled to 14.3-mm-diameter rounds. Specimens were heat treated as noted in Table 3. Testing was conducted in air.

Silicon had little or no effect on tensile strength, as shown in Table 4. However, silicon had a dramatic effect on smooth-bar and double-shank notch-bar ($K_t = 2.0$) rupture life at 538°C and 827 MPa.

Table 1 - Nominal Chemical Compositions, wt. %

Element	Alloy 909	Alloy 903	Alloy 907
Nickel	38.2	38.0	38.4
Cobalt	13.0	15.0	13.0
Iron	42	42	42
Columbium	4.7	3.0	4.7
Titanium	1.5	1.4	1.5
Aluminum	0.03	0.9	0.03
Silicon	0.4	0.1	0.1

Table 2 - Chemical Compositions, wt. %, of Material Tested

Heat ^a	C	Fe	Si	Ni	Cr	Al	Ti	Co	Cb+Ta	B
1	0.03	Bal	0.10	38.1	0.04	0.05	1.53	13.9	4.82	<0.001
2	0.02	Bal	0.20	38.1	0.03	0.05	1.53	13.9	4.81	<0.001
3	0.03	Bal	0.35	38.1	0.06	0.07	1.48	14.0	4.81	<0.001
4	0.01	Bal	0.50	38.0	0.01	0.10	1.53	13.8	4.91	<0.001
5	0.01	Bal	0.39	38.5	0.12	0.05	1.59	13.4	4.80	0.001

^aLaboratory-melted heats except for Heat 5, which was of commercial size.

Figure 4. Microstructure of over-aged alloy 907. 300X.

Table 3 - Heat-Treating Schedules for Low-Expansion Alloys

Alloy	Heat Treatment
903	816°C/1 H WQ + 718°C/8 H FC to 621°C/8 H AC
907	982°C/1 H AC + 774°C/12 H FC to 621°C/8 H AC
909	982°C/1 H AC + 718°C/8 H FC to 621°C/8 H AC

Table 4 - Effect of Silicon on Room-Temperature Tensile Properties^a

Heat	Silicon Content, wt. %	Yield Strength (0.2% Offset), MPa	Tensile Strength, MPa	Elongation, %	Reduction of Area, %
1	0.10	1102	1371	15	33
2	0.20	1074	1378	14	32
3	0.35	1042	1345	16	35
4	0.50	1064	1350	16	37

^aLongitudinal tests on specimens of 14.3-mm-diameter hot-rolled rods.

Figure 5. Effect of silicon content on smooth-bar rupture life at 538°C/827 MPa of alloy 909. Material annealed 982°C/1 h, AC, and aged 718°C/8 h, FC 56°C/h, 621°C/8 h, AC.

Figure 6. Effect of silicon content on notch ($K_t=2.0$) rupture life at 538°C/827 MPa of alloy 909. Specimen from Heat 4 fractured in shank (86% of notch stress) at 1069 MPa (stress increased after 120 h).

Figure 5 shows that smooth-bar rupture life increased two orders of magnitude, apparently peaking with silicon content between 0.3% and 0.5%. Notch rupture life, shown in Figure 6, similarly increased with Heat 4 actually being notch ductile after stress was increased to 1069 MPa to obtain fracture in a reasonable time.

Subsequent studies repeated the same patterns even in transverse orientations and confirmed that silicon contents between 0.3% and 0.6% developed the optimum combination of thermal expansion, tensile properties, and smooth- and notch-bar rupture properties. INCOLOY alloy 909 was thus born, and a commercial heat was melted with a silicon content aimed at 0.4%.

Characterization of INCOLOY alloy 909

In an effort to learn more about the interesting silicon effect and to characterize the new alloy, a major test program was initiated. The program included creep and low-cycle-fatigue crack growth in both air and helium, notched low-cycle fatigue, R. R. Moore high-cycle fatigue, and in-depth metallographic studies as well as the usual tensile and rupture tests.

Material was obtained from commercially produced 25.4 mm X 101.6 mm hot rolled flat from Heat 5 in Table 2. This heat was produced as a 508-mm-diameter ingot by vacuum-induction melting and vacuum-arc remelting. The ingot was cogged to 203.2 mm X 304.8 mm before it was hot rolled to flats. Etch slices revealed no macro-segregation. All test specimens were annealed and age hardened as before, and test orientation was long transverse unless otherwise stated. Results of the test programs were compared with those for INCOLOY alloy 903 also processed to a hot rolled flat but with the TMP and subsequent heat treatment (Table 2) used for that alloy.

Tensile and Rupture Properties

Room-temperature tensile properties in Table 5 show that although yield strength is about 103 MPa less than that of INCOLOY alloy 903*, it is still more than adequate for the application of these alloys. Smooth and notch ($K_t = 2.0$) rupture strengths at 538°C and 896 and 1000 MPa, listed in Table 6, show that INCOLOY alloy 909 offers a threefold increase in transverse rupture strength vs. INCOLOY alloy 903 as well as about a 10 to 15% increase in longitudinal rupture strength.

Fatigue

Notched ($K_t = 2.0$) low-cycle-fatigue tests were conducted in air at 538°C at 20 cpm under load control. The results shown in Figure 7 illustrate the vastly improved notched LCF strength of INCOLOY alloy 909. At 10^4 cycles, notched LCF strength of INCOLOY alloy 909 is about 70% greater than that of INCOLOY alloy 903 with TMP, and at 10^5 cycles it is about 65% greater.

*NOTE: The heavy thermomechanical processing given INCOLOY alloy 903 and low annealing temperature used with that alloy contribute substantially to this differential in room temperature strength.

Table 5 - Room-Temperature Tensile Properties of Hot-Rolled Flat From Heat 5^a

Yield Strength (0.2% Offset), MPa	1020
Tensile Strength, MPa	1313
Elongation, %	16
Reduction of Area, %	32

^aLong transverse tests on material heat treated as in Table 3.

Table 6 - Long Transverse Rupture Properties at 538°C of Hot-Rolled Flat From Heat 5^a

Stress, MPa	Smooth-Bar Life, h	Elongation, %	Reduction of Area, %	Notch-Bar ($K_t=2$) Life, h
896	1000.3 ^b	-	-	1153 ^b
1000	199.8	4	9.5	97

^aMaterial heat treated as in Table 3.

^bTest discontinued at time shown.

Table 7 - Room-Temperature Rotating-Beam Fatigue Strength^a

Alloy	Cycles	Fatigue Strength, MPa
Alloy 903	10 ⁶	469
	10 ⁸	365
Alloy 909 ^b	10 ⁶	545
	10 ⁸	455

^aR. R. Moore tests on specimens from hot-rolled flats heat treated as in Table 3.

^bHeat 5.

Figure 7. Low-cycle fatigue strength of alloys 903 and 909.

Rotating-beam, R. R. Moore high-cycle-fatigue test results conducted at room temperature in air are listed in Table 7. Again, INCOLOY alloy 909 has greater fatigue strength than INCOLOY alloy 903 (with about a 16% increase at 10⁶ cycles and about a 25% increase at 10⁸ cycles).

Crack-Growth Behavior

INCOLOY alloy 909's resistance to SAGBO embrittlement is graphically demonstrated by its creep crack-growth and fatigue crack-growth properties in both air and helium, particularly when contrasted with INCOLOY alloy 903 having TMP.

Testing Methods. Compact tensile-specimen blanks were cut from hot-rolled flat stock with the notches oriented parallel to the rolling direction. The blanks were heat treated as shown in Table 2 and machined into 9.2-mm-thick samples of the same dimensions used in an earlier crack-growth study (3). The specimens were fatigue precracked at room temperature at low stress-intensity ranges using more than 50,000 cycles to grow the precracks.

Fatigue tests were run at 538°C in air and in 99.995% helium. The compliance technique (described in Reference 3) was used to measure the crack lengths. Tests were run in load control with sine-wave loading, an R ratio of 0.1, and frequencies of 2 or 20 cycles per minute in air and 2 cycles per minute in helium.

Creep crack-growth rates were determined at 538°C in air. Crack lengths were determined by measurements of the electrical potential vs time produced by a constant 4 A direct current. In both the fatigue and creep

tests, three specimens were commonly employed for each type of test condition. The fracture surfaces of a number of the samples were examined by scanning electron microscopy.

Results. The results of these tests in Figures 8 through 10 clearly show the superior crack-growth resistance of INCOLOY alloy 909 over INCOLOY alloy 903. The differences between alloys were greatest in the slower tests in air; i.e., in the creep and 2 cpm fatigue tests (Figures 8 and 9). When tested in helium at 2 cpm, the two alloys showed the least difference (Figure 9). Thus, the poorer behavior of INCOLOY alloy 903 appears to be due to the greater degradation in properties caused by the air environment, a manifestation of the SAGBO effect.

Figure 8. Creep crack-growth results for alloys 903 and 909.

Figure 9. Fatigue crack-growth results for alloys 903 and 909 in air and helium.

Crack growth under creep loading appeared to be entirely intergranular in both alloys. Under cyclic loading, the fracture paths in alloy 909 were primarily intergranular at low stress-intensity ranges, but tended to become transgranular at higher ΔK levels. Higher cyclic frequency and a helium environment promoted transgranular cracking. This same general pattern was also found with INCOLOY alloy 903, except that intergranular cracking tended to persist to higher ΔK values.

Microstructure

The microstructure of INCOLOY alloy 909 bears little resemblance to that of its predecessors. Much is still unknown about the microstructure and the phases formed in it, allowing only a summary of results obtained to date.

Pitfalls are encountered when one merely etches the alloy. Figure 11 shows the microstructure of a sample after swab etching in a sulfide stain mixture. Figure 12 shows the identical area after removal of the stain etch (the same structure would appear after an electrolytic etch with phosphoric acid and water). The material had been hot rolled about 40% in reduction of area at 982°C and then annealed and age hardened as described in Table 2, which represents a fairly typical thermomechanical processing sequence for this product.

What appears to be the grain structure in Figure 12 is really a semi-continuous discrete precipitate outlining both prior and existing grain boundaries. The "true" grain structure, observed in Figure 11, clearly shows the fine, recrystallized grain with the precipitate appearing as light dots.

Figure 13 shows a TEM view of the microstructure produced by the standard heat treatment. Both intragranular and grain-boundary particles of micro-meter dimensions are evident in the structure, as well as finer precipitates within the grains. Examinations of extracted particles by X-ray diffraction indicates the larger particles are a mixture of carbides, Fe_2Si silicides, and possibly an Fe_2Cb laves phase.

At higher magnification, Figure 14, the finer structure within the grains is evident. The long acicular particles are probably either $\text{Ni}_3(\text{Ti}, \text{Cb})$ epsilon phase found in INCOLOY alloy 907, Ni_3Cb delta (orthorhombic) phase, or Ni_3Ti eta phase. The cuboidal particles are probably $\text{Ni}_3(\text{Al}, \text{Ti}, \text{Cb})$ gamma prime or ν'' (BCT). There also appears to be a very much smaller precipitate lying between the cuboidal particles. These small particles are thought to be ν' (or ν'') precipitated during the final aging at 621°C.

Of particular interest is the role of silicon in the structure. The results thus far suggest that silicon forms rather coarse Fe_2Si silicide particles in the grain boundaries. Just why such particles should be so helpful to the alloy's SAGBO resistance and notch rupture strength is far from obvious. Whether the Fe_2Si particles have a direct effect on the alloy's properties, or if there is some additional effect by elemental silicon acting to prevent premature cracking at grain boundaries is a matter of speculation. One can postulate that either silicon or the silicide may be acting as gettering agents, trapping oxygen as it diffuses through the grain boundaries. Silicon or the silicides might also slow oxygen diffusion. Further research to answer these questions is under way.

Figure 10. Fatigue crack-growth results at 20 cpm for alloys 903 and 909.

Figure 11. Microstructure of alloy 909 after swab etching with a sulfide stain. 500X.

Figure 12. Microstructure of alloy 909 after removal of stain etch shown in Figure 11. 500X.

Figure 13. Microstructure (TEM) of alloy 909 with the standard heat treatment.

Figure 14. Microstructure (TEM) shown in Figure 13 at higher magnification. The finer structure within the grains (see text) is evident.

Summary

INCOLOY alloy 909 is the first low-expansion superalloy combining good resistance to Stress Accelerated Grain Boundary Oxygen embrittlement with good tensile properties but without restrictive TMP and resulting directionality. These properties are achieved by adding 0.3% to 0.6% silicon to a low-aluminum, 38% nickel, 13% cobalt, 1.5% titanium, 4.7% columbium, balance iron composition. These significant improvements rising from such a small silicon addition were quite unexpected:

1. Both creep and fatigue crack-growth properties are dramatically improved over INCOLOY alloy 903 with TMP.
2. Room-temperature high-cycle-fatigue and elevated-temperature, notched low-cycle-fatigue strengths are greatly improved.
3. The notch rupture properties ($K_t = 2.0$) at 538°C and short-term tensile-strength combination is the best attainable in useful low-expansion superalloys.
4. Commercially attractive heat-treatment cycles, including short-time treatments, are used to achieve properties instead of uneconomical and strength-reducing long-time overaging heat treatments.
5. Thermal-expansion properties are essentially unaltered.

The mechanism by which silicon exerts such a profound influence on properties, microstructure, and thermal-processing characteristics is not clear. The apparent indication of Fe_2Si silicides does not offer immediate answers and in fact seems to raise more questions. Work is in progress to unveil the mechanism(s) providing SAGBO embrittlement resistance in INCOLOY alloy 909.

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