MECHANICAL PROPERTIES OF A NEW HIGHER-TEMPERATURE

MULTIPHASE® SUPERALLOY

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Summary

A new superalloy in the MULTIPHASE® family of alloys has been developed with greater temperature resistance than Waspaloy. The new alloy has 260 ksi or higher room-temperature ultimate tensile strength. However, the new alloy extends the usefulness of MULTIPHASE® alloys from the former high temperature limitation of 593° C (1100° F) up to 730° C (1350° F). The effects of thermal mechanical processing on room-temperature as well as elevatedtemperature tensile properties were determined. Furthermore, the effects processing variables on the stress of cold work and aging temperature rupture properties were determined over the range of 593°C (1100°F) to 730°C (1350°F). The effects of overaging were examined both as a result of heat treatment and exposure to temperature under load. Attention was given to impact properties before and after service exposure to determine the effect of in-service embrittlement. Stress for 100-hour and 1000-hour rupture lives, established for several temperatures, exhibit superior performance for the new alloy when compared to Waspaloy. Additional work is in progress to establish the immunity of the new alloy to stress corrosion cracking and hydrogen embrittlement.

Introduction

Background

The first commercial alloy in the MULTIPHASE (MP) system was MP35N, developed by G. D. Smith of the DuPont Company. The MULTIPHASE system is different from most nickel-based and cobalt-based alloys, many of which derive their strength from the precipitation of gamma-prime or from solidsolution effects. The MP system has a complex structure which gives it great strength, ductility and fracture toughness. Firstly, it relies upon the partial transformation by cold work of the face-centered-cubic (fcc), high temperature form of cobalt to a network along the (111) planes of plates of the low-temperature, hexagonal-close-packed (hcp) form. 2 This structure appears in the Widmannstatten pattern in a micrograph. Because the c:a ratio of the hcp is higher than is required for normal close-packing, the plates are put in compression, and this causes them to twin. The basal planes of the hcp plates are parallel with the (111) planes of the fcc, but those of the twinned portions are normal to this direction. Such a structure forms a formidable series of barriers to the movement of dislocations to provide high strength; but the further transformation to hcp under stress also gives unusually good ductility. This is due to a form of the transformation-induced plasticity (TRIP) mechanism described by Zackay, et al. 3 A further increase in strength is provided by precipitation along the fcc/hcp boundaries during aging. 4 MP35N, having a composition of 35% cobalt, 35% nickel, 20% chromium and 10% molybdenum, also exhibits extremely high corrosion resistance along with its high strength and ductility. negative aspects of the alloy are that it has a relatively low service temperature limit of 427°C (800°F) the mix cost is high, and it requires a considerable amount of cold work for hardening.

A development program was undertaken to overcome these negative aspects; this resulted in MULTIPHASE Alloy MP159 which has a maximum service temperature of 593°C (1100°F). The ideas behind the development of this alloy were to use the known precipitation-hardening elements-aluminum, columbium and titanium to improve high-temperature performance and to use iron to reduce the mix cost as much as possible. A large number of independent variables with their probable interactions and many dependent properties with their inevitable trade-offs, were involved. A fractional-factorial experiment was used and generated an extensive data base. An interesting sidelight of this work was the finding that the critical Nv number marking the boundary of the onset of the embrittling sigma phase is very dependent upon the iron concentration; the number decreases from about 2.77 at 0% iron to about 2.60 at 20% and increases to about 2.88 at 40% iron. However, while this alloy successfully met the user requirements at the time, advancing designs called for a still-higher service temperature.

Alloy Development

A new alloy with comparable strength and ductility, capable of operating at (704°C) 1300°F was needed. This temperature is above the fcc/hcp transus of MP159 and would anneal out the complex structure upon which the alloy depends for its strength. Steps to raise the transus included a drastic reduction in iron and modest increases in chromium and molybdenum. The data base from the earlier experiment showed the columbium and titanium were most effective in improving stress-rupture strength with the least loss in ductility, although both of these elements lower the transus. Aluminum was eliminated because, although it was very effective in improving stress-rupture life, it caused a severe loss in ductility. It also

reduced room- and elevtated-temperature tensile strength. Its removal could also be expected to raise the transus by a very small amount. In order to obtain quantitative data upon which to base an alloy composition, a half replicate design was used: titanium levels were set at 3% and 4%, columbium at 0.6% and 1.2%, iron at 0% and 7%. The remainder of the design was close to the MP159 chemistry except that aluminum was not included. As a result of this experiment, the following alloy composition was decided upon: Cb, 1.10%; Ti, 3.8%; Ni, 25.7%; Cr, 19.5%; Mo, 7.5%; Fe, 1.0%; B, 0.01%; C, 0.01%; Co, bal. This alloy, when cold worked and aged at the appropriate levels, met the higher temperature requirements as shown below.

Experimental Procedure

Alloy Processing and Specimens

A five-hundred pound heat of the new, optimized alloy was VIM-VAR melted, cast, extruded, hot rolled and then cold drawn. At appropriate stages in the processing, suitable homogenizing and annealing cycles were used. Two final cold-work levels, 36% and 48%, were used in this evaluation. The upper cold-work level was selected because it was known from prior work on MP159 that it would result in room-temperature tensile strengths in excess of 260 ksi. The lower level was projected based on preliminary results that indicated a lesser amount of cold work would produce adequate strength.

Bars for testing were supplied for two basic sizes that could be used to manufacture bolts, studs, or specimens from either 5/16" or 3/8" diameter. Blanks were cut into 1-3/4", 2", and 3" lengths for various size studs and specimens. The larger of the two sizes, 3/8", was used to make standard .252" diameter tensile specimens.

Blanks were aged at 593° C, 650° C, and 704° C (1100° F, 1200° F and 1300° F) for four hours then air cooled. After aging, blanks were ground to size to remove all traces of oxide prior to thread rolling. The three-inch long 3/8" diameter studs were machined into .252" diameter tensile specimens.

Tensile Testing

Tensile tests were made on studs and specimens at room and at elevated temperatures of 593°C, 650°C, and 732°C (1100°F, 1200°F and 1350°F). The elevated-temperature tensile testing was done in a water-cooled, quartz-element furnace. Specimens were soaked at temperature for one-half hour before they were pulled. Stresses, reported in ksi, are based on the actual fracture load, in pounds, divided by the cross-sectional area calculated from the basic pitch diameter for studs, and from the actual gage diameter for specimens. When studs were tested, three threads were exposed. A 266.9 kN (60,000 lbs.) hydraulic loading tensile machine was used for all of these tests. A rigid set-up is used in the tensile machine that has prealigned links and threaded bushings to minimize the effect of bending.

Stress Rupture Testing

Stress-rupture tests were run using dead-weight loading machines where an elevator applied the load without shock loading the specimen. Alignment of the machines is corrected by sections in the specimen load train that are thin webs at 90° to each other which will cancel bending moments. The elapsed time was measured to the nearest 0.1 hour. Other details of the general practice, such as attachment of thermocouples and the machine and instrument calibration, followed those procedures outlined in MIL-STD-1312.

Results and Discussion

Tensile Properties

At the 48% cold-worked level, the room-temperature tensile strength was approximately 300 ksi level, but the ductility was marginal as evidenced by the seven to nine percent elongation and the narrow spread between the yield strength and ultimate strength, indicating minimal uniform elongation. However, the high reduction of area (25% to 40%) indicates significant necking before fracture. Average tensile properties for specimens tested at room and elevated temperatures are shown for various aging treatments in Figure 1.

At elevated temperatures, the 593° C (1100° F) properties with 48% coldwork and 593° C (1100° F) age exhibit very poor ductility and indicates a region of cold-work and aging treatment that must be avoided. Increasing the aging temperature and/or reducing the degree of cold-work alleviates this embrittled condition.

The undesirable loss of ductility for the 593°C (1100°F) age mentioned above is not observed for tests made at 650°C (1200°F) and at 732°C (1350°F). Since it is anticipated that this material would be used at temperatures greater than 593°C (1100°F), it is not likely that anyone would choose to use the 593°C (1100°C) aging treatment. The higher the aging temperature the less the degree of embrittlement at 593°C (1100°F).

It should be noted that, all strength properties are superior to Waspaloy; the higher aging temperatures gave equivalent or superior %RA values.

The tensile properties for the 36% cold-worked material are shown in Figure 2. The 36% cold-worked material had somewhat lower tensile strength than the 48% cold-worked material, but it was higher than Waspaloy.

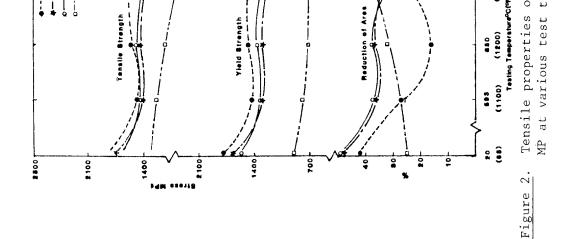
Decreasing the cold work from 48% to 36% shifted the minimum in ductility from 593° C (1100° F) age and 593° C (1100° F) test temperature to the 593° C (1100° F) age and the 650° C (1200° F) test temperature. Furthermore, the degree of embrittlement was much less for the lower amount of cold work ($\sim 16\%$ RA vs. $\sim 1.0\%$ RA). (Compare Figures 1 and 2).

The maximum variation between stud and specimen UTS values was approximately 10%. The usual variation was only a few percent without either being consistently greater than the other.

Creep-Rupture Properties

The creep-rupture testing was concentrated on parts that were aged at 704°C (1300°F) and 650°C (1200°F) owing to the greater ductility and good strength. Initial loading was at 724 MPa (105 ksi) and 732°C (1350°F) and 1034 MPa (150 ksi) and 650°C (1200°F). The Larson-Miller parameter was established for these two points, and estimates of the stresses to achieve 100-hour lives were determined using linear regression analysis. The actual Larson-Miller parameter vs. stress curves are shown in Figures 3 and 4.

The majority of the creep-rupture testing was done on 3/8 studs; therefore, these data were used for the 100- and 1000- hour life projections at various test temperatures after the several aging treatments. The resulting stresses are tabulated in Tables I and II.



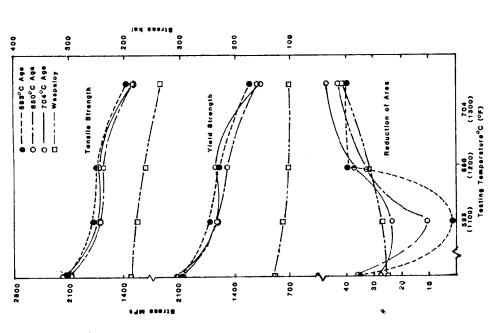
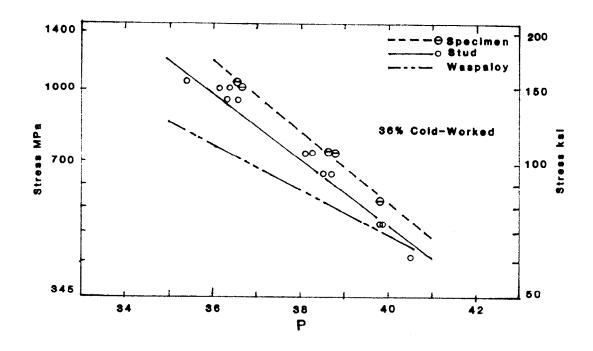


Figure 1. Tensile properties of 48% cold-worked MP at various test temperatures.



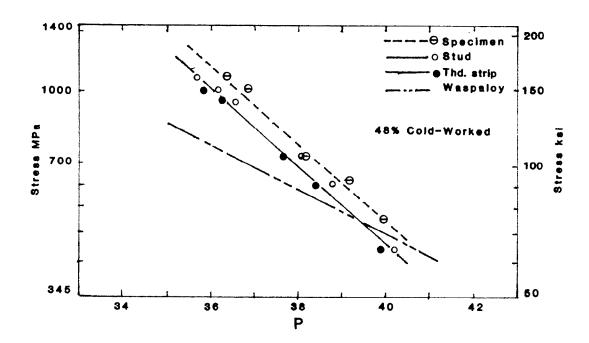
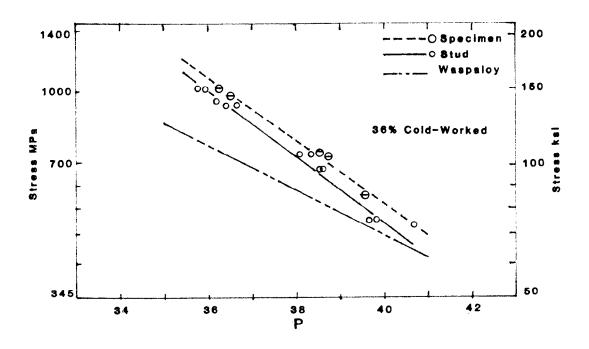


Figure 3. Larson-Miller curves for MP aged at 650°C (1200°F).



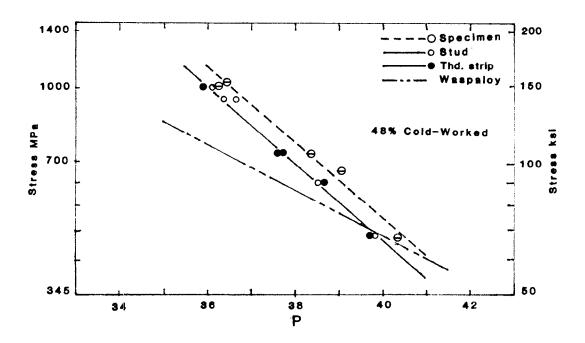


Figure 4. Larson-Miller curves for MP aged at 704°C (1300°F).

The square brackets in these tables indicate the percentage increase in stress of the MULTIPHASE (MP) alloy over Waspaloy. The MP has as much as a 17% higher strength over Waspaloy at 704°C (1300°F) for 100-hour rupture life

TABLE I

Projected Stress in MPa (ksi) [% increase over Waspaloy] to Obtain 100-Hour Rupture Life at Various Temperatures

Test	Aging Cycle, OC (OF)			
Temperature	4 Hours at	4 Hours at	4 Hours at	Waspaloy
OC (OF)	593(1100)	650(1200)	704(1300)	
		48% Cold-Worked		
593(1100)	1,310.0(190.0)[58.3]	1,291.4(187.3)[56.1]	1,305.9(189.4)[57.8]	827.4(120.0)
650(1200)	956.3(138.7)[32.1]	1,954.9(138.5)[31.9]	977.0(141.7)[35.0]	723.9(105.0)
704(1300)		619.2(89.8)[8.9]	648.1(94.0 [13.9]	568.8(82.5)
732(1350)	425.4(61.7) [-13.7]	450.9(65.4)[-8.5]	484.0(70.2 [-1.8]	493.0(71.5)
		36% Cold-Worked		
593(1100)	1,299.3(181.2)[51.0]	1,221.1(177.1)[47.5]	1,276.9(185.2)[54.3]	827.4(120.0)
650(1200)	957.7(138.9)[32.3]	1,152.8(167.2)[59.2]	971.5(140.9)[34.2]	723.9(105.0)
704(1300)		662.6(96.1)[16.5]	665.3(96.5)[17.0]	568.8(82.5)
732(1350)	665.3(61.7)[-13.7]	522.6(75.8)[6.0]	513.0(74.4)[4.1]	493.0(71.5)

TABLE II

Projected Stress in MPa (ksi) [% increase over Waspaloy] to Obtain 1000-Hour Rupture Life at Various Temperatures

Test				
Temperature	4 Hours at	4 Hours at	4 Hours at	Waspaloy
°C (°F)	593(1100)	650(1200)	704(1300)	
		48% Cold-Worked		
593(1100)	1,059.0(153.6)[43.7]	1,052.8(152.7)[42.8]	1,072.8(155.6)[45.6]	737.0(106.9)
650(1200)	689.5(100.0)[14.6]	701.2(101.7)[16.5]	729.5(105.8)[21.2]	601.9(87.3)
704(1300)		350.3(50.8)[-25.0]	385.4(55.9)[-17.4]	466.8(67.7)
732(1350)	133.8(19.4) [-66.4]	140.0(20.3)[-64.9]	213.7(31.0) [-46.4]	398.5(57.8)
		36% Cold-Worked		
593(1100)	1,042.5(151.2)[42.5]	1,023.2(148.4)[38.8]	1,060.4(153.8)[43.9]	737.0(106.9)
650(1200)	737.0(106.9)[22.4]	730.8(106.0)[21.4]	740.5(107.4)[23.0]	601.9(87.3)
704(1300)	432.3(61.7)[-7.4]	439.2(63.7)[-5.9]	421.3(61.1)[-9.8]	466.8(67.7)
732(1350)	279.2(40.5)[-29.9]	293.0(42.5)[-26.5]	261.3(37.9)[-34.4]	398.5(57.8)

In order to determine the stability of the MULTIPHASE alloy, tension impact tests were made on the as-aged material and after aged material was exposed to 732°C (1350°F) at a stress of approximately 345 MPa (50 ksi) from 100 to 1000 hours. A significant decrease in energy to failure was observed after exposure (eg. from 150 to 200 ft.-lbs. down to 10 to 14 ft.lbs.). However, inadvertently the threads were rolled on the specimens after aging. The threads should have been formed before aging and embrittlement might have been minimized. The embrittling effect of rolling threads after aging but prior to exposure is illustrated by the tensile data shown in Table III. Additional work is in progress in this area of the embrittlement study.

TABLE III

Effect of Stressed Exposure at 732°C (1350°F) on the Room Temperature Tensile Properties of 48% Cold-Worked MULTIPHASE (MP) Aged at 704°C (1300°F) for 4 Hours When Threads Were Rolled After Aging But Before Exposure

Property	No Exposure	97.5 ksi for 10 hours	65.7 ksi for 100 Hours
UTS, ksi	312.6	280.2	Broke in threads
.2% Y.S. ksi	304.3	276.1	Broke in threads
E%	7.0	5.5	Broke in threads
RA%	27.4	14.5	Broke in threads

A fractional-factorial technique was used in the mechanical property evaluation and from this the following may be concluded:

- Cold work had a very significant effect on both stud and specimen tensile strength, that is, increasing cold work increases the tensile strength.
- Increasing aging temperature increased the stud tensile strength at room temperature but had no significant effect at higher temperatures. The effect on specimens was not significant at any temperature.
- 3. There was a negative effect of size at the 593°C (1100°F) aging temperature on room-temperature tensile strength but no significant effect of size at the higher aging or test temperatures.
- 4. Cold-work had a negative effect on stress-rupture life. This showed as a decreasing stress to produce 100- and 1000-hour lives as the percent of cold-work as increased.
- 5. The effect of aging temperature on stress-rupture life was mixed; there was a positive effect when comparing 593°C (1100°F) and 650°C (1200°F) but a negative effect when comparing 650°C (1200°F) and 704°C (1300°F). This occurred at both 100-hour and 1000-hour life stress, suggesting that the optimum aging temperature may be between 650°C and 704°C (1200°F and 1300°F).
- 6. Up to 650°C (1200°F), there was some evidence, when comparing the tensile strengths of studs and specimens, that the notches (threads) detracted from the tensile strength. This effect was about 5% of the average tensile strength.
- 7. Cold-work increases the yield strength at all temperatures of aging and testing.
- 8. Comparing 593°C (1100°F) and 650°C (1200°F) aging treatments, an increase in aging temperature tended to decrease the yield strength but comparison of the 650°C (1200°F) and 704°C (1300°F) aging gave an improvement in yield strength.

- 9. As would be expected, cold work had a negative effect on elongation and reduction of area, and as the test temperature increased, this effect was diminished.
- 10. No firm conclusion could be made about the effect of aging on elongation, and more testing would be required in this area.
- 11. The effect of aging temperature on reduction of area was greater between 593°C (1100°F) and 650°C (1200°F) than between 650°C (1200°F) and 704°C (1300°F) and diminished as the test temperature increased.

It should be emphasized that this alloy development program is continuing and the above data indicate the potential of alloys in the MULTIPHASE family to compete with Waspaloy up to 704°C (1300°F). Neither alloy composition nor thermal-mechanical processing have been optimized and additional improvements in the performance of MULTIPHASE alloys are anticipated.

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