DMIC review OF RECENT DEVELOPMENTS

Nickel- and Cobalt-Base Alloys

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ALLOY DEVELOPMENT AND EVALUATION

Contour Rolling of René 95 Disks

An investigation to develop manufacturing techniques for contour cross rolling René 95 alloy disks was recently initiated at General Electric.(1) *In Phase I of this program, a process will be* developed to produce disks from both René 95 wrought and powder materials. In Phase II, subsize disks will be formed from the type of material that performs best in Phase I. Full-scale disks will be produced and evaluated in Phase III from the same material selected for Phase II.

At this point in the program, two heats of René 95 alloy have been melted and cast and processed into wrought and powder forms for subsequent use in forming the subscale disks.

Development of Processing Methods For Superalloy Sheet

An investigation to develop a reproducible process for the production of high-quality sheet of nickel-base alloy, Unitemp AF2-1DA, is continuing at Universal-Cyclops Speciality Steel Division.(2) Vacuum arc remelted (VAR) and extruded material has been hot and cold rolled to 0.030-inch sheet with reportedly little difficulty. Vacuum arc remelted and press forged material also has been hot and cold rolled to 0.030-inch sheet with reportedly little difficulty. Efforts to produce sound electroslag refined (ESR) ingots have not been successful because of cracking at the center of the ingots which may result in the discontinuance of this phase of the program.

Mechanical property tests, including tensile and stress-rupture tests, have been conducted on the materials at various thicknesses. The tensile and stress-rupture properties of 0.030-inch sheet are given in Tables 1 through 4.

OXIDATION, HOT CORROSION, AND PROTECTION

Aluminide Slurry Coatings

A program to develop and evaluate slurry-applied nickel and cobalt aluminide coatings for improved hot-corrosion resistance of nickel-base superalloys for gas-turbine blades and vanes is being carried out at Solar.(3) The goal in this program is to develop a coating that will possess at least twice the life of commercially available coatings on superalloys such as B-1900, Alloy 713C, and NASA VIA.

Two different application techniques--sinter and aluminize and fusion aluminide--will be investigated. In the sinter and aluminize process, the slurry consisting of the nickel- or cobalt-base modifier and an ethyl cellulose-xylene vehicle is sprayed on specimens. The coating then is vacuum sintered, followed by spraying with an aluminum slurry. Finally, the coatings are fused in an inert atmosphere and diffused to form the aluminide.

The fusion aluminide process consists of spraying the aluminum plus nickel- or cobalt-base modifier on specimens, fusing in an inert atmosphere at 1400 to 1600 F and then diffusing at 2000 F until most of the aluminum is converted to NiAl or CoAl.

Solar expects to obtain coatings more reliable than present commercial coatings because no inert materials will be added to the slurries and none of the materials will be reused.

Results to date indicate that modifiers of nickel, 85Ni-15Cr, and 85Ni-15Co, can be successfully applied to cast Alloy 713C specimens by vacuum sintering at 2050 and 2150 F. Solar found, however, that limitation of the sintering temperature to less than 2100 F was necessary in order to maintain compositional control by reducing losses of elements by vaporization.

The sulfidation resistance of aluminized specimens at 1750 F was improved by the addition of the chromium modifier to the coating.

Oxidation Resistant Claddings on IN-100

The effect of exposure time, cycle frequency, and temperature on the cyclic oxidation behavior of three foil claddings on IN-100 alloy was investigated at Lewis Research Center.(4) The three claddings consisting of Ni-20Cr-4Al-1.2Si, Fe-25Cr-4Al-1Y, and Ni-30Cr-1.4Si in the form of foils ranging from 0.051 to 0.254 mm in thickness were diffusion bonded to IN-100. Specimens of the clad alloy were exposed to temperatures of 1040 and 1090 C (1905 and 1995 F) for times to 400 hours using 1- and 20-hour cycles.

Based on gravimetric, metallographic, and surface recession analyses, the Ni-20Cr-4Al-1.2Si

TABLE 1. TENSILE PROPERTIES OF 0.030 INCH UNITEMP AF2-1DA SHEET[2]

(VAR-Extruded)

Heat Treatment	Test Temperature, F	Ultimate Strength, ksi	0.2% Yield Strength, ksi	Elongation, percent
		Hot Rolled		
A-1	Room	178.1	171.1	1.0
B	Room	199.2	177.1	3.2
A-1	1400	136.6	136.6	0.7
B	1400	138.4	134.0	2.6
A-1	1800	48.8	36.0	9.0
B	1800	11.0	4.3	158.0
		Cold Rolled		
A-1	Room	159.1	159.1	0.3
B	Room	187.1	151.8	6.5
A-1	1400	138.7	135.9	0.8
B	1400	153.1	136.8	4.9
A-1	1800	48.7	34.5	5.7
B	1800	46.1	28.3	8.8

Heat Treatment A-1: 2125 F/2 hours/RAC
1950 F/2 hours/RAC
1400 F/16 hours/AC

Heat Treatment B: 1650 F/24 hours/to
2000 F/1 hour/to
1000 F/1 hour/WQ
1400 F/16 hours/AC

TABLE 2. TENSILE PROPERTIES OF 0.030 INCH UNITEMP AF2-1DA SHEET[2]

(VAR-Press Forged)

Heat Treatment	Test Temperature, F	Ultimate Strength, ksi	0.2% Yield Strength, ksi	Elongation, percent
		Hot Rolled		
A-1	Room	179.[illegible]	165.3	1.1
B	Room	207.7	185.2	5.8
A-1	1400	145.7	144.5	1.1
B	1400	150.0	131.8	7.6
A-1	1800	49.8	36.5	8.2
B	1800	16.8	3.2	147.4
		Cold Rolled		
A-1	Room	171.4	154.1	2.1
B	Room	186.4	155.0	5.7
A-1	1400	138.1	133.9	1.0
B	1400	150.3	132.9	3.6
A-1	1800	49.7	35.3	8.4
B	1800	49.9	30.9	9.0

Heat Treatment A-1: 2125 F/2 hours/RAC
1950 F/2 hours/RAC
1400 F/16 hours/AC

Heat Treatment B: 1650 F/24 hours/to
2000 F/1 hour/to
1000 F/1 hour/WQ
1400 F/16 hours/AC

alloy afforded the most protection, while the Ni-30Cr-1.4Si alloy offered the least protection at both test temperatures. Furthermore, the oxidation behavior of the former cladding was rather insensitive to cycle frequency, cladding thickness, and exposure time; its performance was comparable to that of a commercial aluminide coating on IN-100, as shown in Figure 1. On the other hand, the lives of the other two claddings were extended by increasing the cladding thickness. In order to obtain a life of 400 hours at 1040 C (1905 F), a 0.127-mm thickness of these two claddings was required.

The oxidation resistance of all three claddings was degraded to various degrees by interdiffusion of the cladding and the base metal. This effect, however, was small for the 0.127-mm-thick Ni-20Cr-4Al-1.2Si alloy cladding at 1040 C for exposures to at least 200 hours.

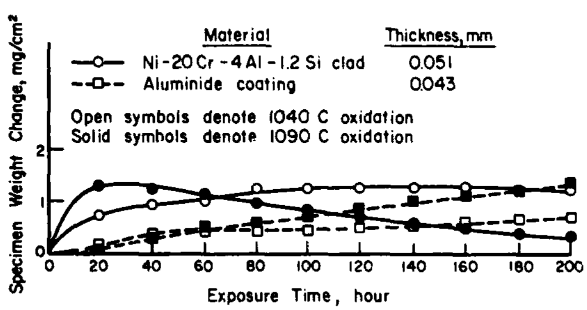

FIGURE 1. COMPARISON OF CYCLIC OXIDATION RESISTANCE OF Ni-20Cr-4Al-1.2Si CLAD IN-100 AND COMMERCIAL ALUMINIDE-COATED IN-100 AT 1040 AND 1090 C.[4]

(Twenty-hour cyclic exposures)

Effects of Electric Fields on Oxidation and Hot Corrosion

A basic study to determine the effect of electric fields on the oxidation/hot corrosion of metals and alloys has just begun at Arthur D. Little.[5] In previous studies at Arthur D. Little (Contract N00019-70-C-0249), the imposition of a negative voltage (-2 to -300 V) was shown to accelerate corrosion of nickel and Ni-20Cr alloy with and without the presence of sulfur, while the application of a positive voltage was shown to decrease the corrosion reaction.

The first experiments in the current program were designed to determine the effects of time and temperature on the depth of corrosion when nickel specimens are polarized negatively. The

TABLE 3. STRESS RUPTURE PROPERTIES OF 0.030 INCH UNITEMP AF2-1DA SHEET(2)

(VAR-Extruded)

Heat Treat-ment	Test Tempera-ture, F	Rupture Stress, ksi	Rupture Life, hours	Elonga-tion, percent
		Hot Rolled		
A-1	1400	85.0	0.5	1.1
B	1400	85.0	2.6	7.8
A-1	1800	20.0	3.1	14.1
B	1800	20.0	0.1	147.7
		Cold Rolled		
A-1	1400	85.0	0.2	0.9
B	1400	85.0	7.7	1.0
A-1	1800	20.0	6.1	9.1
B	1800	20.0	1.1	10.6

Heat Treatment A-1: 2125 F/2 hours/RAC
1950 F/2 hours/RAC
1400 F/16 hours/AC

Heat Treatment B: 1650 F/24 hours/to
2000 F/1 hour/to
1000 F/1 hour/WQ
1400 F/16 hours/AC

TABLE 4. STRESS RUPTURE PROPERTIES OF 0.030 INCH UNITEMP AF2-1DA SHEET(2)

(VAR-Press Forged)

Heat Treat-ment	Test Tempera-ture, F	Rupture Stress, ksi	Rupture Life, hours	Elonga-tion, percent
		Hot Rolled		
A-1	1400	85.0	0.8	(a)
B	1400	85.0	4.4	20.3
A-1	1800	20.0	15.3	8.8
B	1800	20.0	0.1	240.5
		Cold Rolled		
A-1	1400	85.0	2.9	1.6
B	1400	85.0	14.4	1.9
A-1	1800	20.0	8.1	7.5
B	1800	20.0	1.4	5.7

Heat Treatment A-1: 2125 F/2 hours/RAC
1950 F/2 hours/RAC
1400 F/16 hours/AC

Heat Treatment B: 1650 F/24 hours/to
2000 F/1 hour/to
1000 F/1 hour/WQ
1400 F/16 hours/AC

(a) Sample broke outside gage length.

results to date indicate that with Na_2CO_3, K_2SO_4, or Na_2CO_3-SO_2 present in a propane/air flame, the corrosion depth and intergranular penetration of nickel is increased by the application of a negative potential to the metal. Moreover, the polarization was found to have very much less of an effect on oxidation/corrosion in the lower temperature flames (about 1000 C), compared with the effect in higher temperature flames (about 1200 C).

Oxidation of Nickel-Aluminum Alloys

The effect of chromium, silicon, and titanium on the isothermal oxidation rate and cyclic spall resistance of cast single-phase alloys in the nickel-aluminum system was determined in a study at Lewis Research Center.[6] Chromium, silicon, and titanium additions of 1, 3 and 10 atomic percent were substituted for the aluminum in Ni-10Al(γ), Ni-25Al(γ'), and Ni-50Al(β). These alloys along with the binary alloys and unalloyed nickel were isothermally and cyclically oxidized in still air at 1100 C (2010 F) for times to 100 hours.

The results of this study indicated that none of the Ni-10Al alloys had better oxidation resistance than nickel. Also, only the alloy containing 1 percent silicon was more oxidation resistant than Ni-10Al. The 10 percent chromium or titanium alloys were the least oxidation resistant of these alloys.

The Ni-25Al alloys containing 0, 1, and 3 percent chromium, silicon, or titanium were more oxidation resistant than nickel. The γ' alloys with 1 and 3 percent silicon were more resistant to oxidation than Ni-25Al alloy and as oxidation resistant as the best β alloys. The γ' alloys with 10 percent chromium, silicon, or titanium showed the poorest oxidation resistance of the γ' alloys.

All of the β alloys exhibited better oxidation resistance than nickel. The alloy containing 1 percent silicon was more oxidation resistant than Ni-50Al alloy, but the silicon-containing alloy was not single phase. The β alloys containing 10 percent chromium, silicon, or titanium showed poorer oxidation resistance than the other β alloys.

Oxidation and Hot Corrosion of Dispersed-Phase Alloys

The influence of dispersed rare-earth oxides on the high-temperature oxidation and hot corrosion of nickel- and cobalt-base superalloys is being investigated at Battelle/Columbus.[7] Alloys being studied in this program include:

Ni-20 wt% Cr-3 vol% Y_2O_3
Ni-20 wt% Cr-3 vol% CeO_2
Co-25 wt% Cr-3 vol% Y_2O_3
Co-25 wt% Cr-6 wt% Al-3 vol% Y_2O_3
Co-20 wt% Cr-6 wt% Al-3 vol% Y_2O_3.

CeO_2 additions to the cobalt-base alloys in place of the Y_2O_3 may also be studied. The alloys are being prepared by milling in a Szegvari Attritor, cold pressing and sintering, canning and hot rolling.

Oxidation tests have been carried out on several alloys at temperatures from 1000 to 1200 C (1830 to 2190 F) in slowly flowing oxygen at a pressure of 100 torr. The results of these tests are given in Table 5.

CRACKING IN SUPERALLOYS

Edge-Notch Sensitivity of Waspaloy

A study of the time-dependant edge-notch sensitivity of Waspaloy alloy sheet at temperatures between 900 and 1400 F was conducted at the University of Michigan.[8] Tensile and creep-rupture properties including the notch sensitivity were evaluated on sheet material in thicknesses ranging from 0.013 to 0.075 inch.

The sheet-thickness was shown to have no influence on the results. Time-dependent notch sensitivity was observed at temperatures from 900 to 1300 F and was dependent on the heat treatment. Overaging was found to eliminate the time-dependent notch sensitivity. Optimum combination of smooth and notched strengths for sheet solution treated at 1975 F were achieved by aging at 1550 F for 24 hours.

The nature of the interaction of dislocations with the γ' precipitates was examined in the program. The dislocation-motion mechanism was found to vary with the heat treatment and test conditions. Gamma-prime particles smaller than a critical size were sheared by the dislocations, while particles larger than a critical size were by-passed by dislocations. According to the investigators, the former mechanism promoted the deformation-time characteristics that gave rise to the time-dependent notch sensitivity.

TABLE 5. OXIDATION DATA ON NICKEL- AND COBALT-BASE ALLOYS WITH AND WITHOUT ADDITIONS OF RARE-EARTH OXIDES[7]

Oxidation Time, hours	Weight gain, mg/cm^2 Chart	Weight gain, mg/cm^2 Weighed After Cooling
	Ni-20 wt% Cr-3 vol% Y_2O_3	
	1100 C (2010 F)	
51	0.45	0.26
50	--	0.20
50	0.88	0.70
50	0.81	0.91 (annealed)
24	--	0.31
	0.41	0.38 (annealed)
27	0.30	0.36
25	0.72	0.69
6	0.26	0.26
6	0.60	0.67
1	0.17	0.16
	1200 C (2190 F)	
54	0.56	-1.43
47	1.04	0.32 (annealed)
51	0.57	-0.40 (annealed)
	Ni-20 wt% Cr-3 vol% CeO_2	
	1100 C (2010 F)	
53	1.19	0.95
25	0.19	0.02
	TDNiCr	
	1100 C (2010 F)	
50	0.19	0.19

Oxidation Time, hours	Weight gain, mg/cm^2 Chart	Weight gain, mg/cm^2 Weighed After Cooling
	Co-21 wt% Cr-3 vol% Y_2O_3	
	1000 C (1830 F)	
50	0.23	-0.11
25	--	0.29
5	--	0.29
	1100 C (2010 F)	
64	0.35	0.48
50	--	-0.18
	0.63(?)	-0.90
	0.43	-0.45
25	--	-0.08
27	0.46	0.44
5	--	0.13
	1200 C (2190 F)	
50	0.40	-0.65
70	-0.26	-1.49
	Co-28Cr-4.5Al-2.8Y_2O_3	
	1100 C (2010 F)	
50	2.85	1.22
25	1.99	1.23
25	2.10	0.97
25	1.81	0.79
	Co-25Cr-5Al	
	1100 C (2010 F)	
52	1.54	-2.3

NEW PUBLICATION

Vacuum Melting and Casting of Superalloys

A comprehensive report on the vacuum melting and casting of superalloys has been prepared at Battelle/Columbus for NASA.[9] This report discusses important factors to consider in melting and casting, melting processes, and vacuum casting. The report is available for $3.00 from the National Technical Information Service, Springfield, Virginia 22151.

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(8) Wilson, D. J., and Freeman, J. W., "Sensitivity of the Creep-Rupture Properties of Waspaloy Sheet to Sharp-Edged Notches in the Temperature Range of 1000 to 1400 F", Report NASA CR-1849, University of Michigan, Ann Arbor, Mich. (June 1971).

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