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review OF RECENT DEVELOPMENTS

Nickel-and Cobalt-Base Alloys

H. J. Wagner • May 15, 1968

PHYSICAL METALLURGY

Thermomechanical Treatment of Compressor Disk Alloy

Preliminary results have been reported on the effects of various thermomechanical treatments applied to Alloy AF-3, General Electric's experimental alloy for hot turbine-engine compressor disks.(1) Composition of Alloy AF-3 is Ni-0.15C-3.5Al-2.5Ti-15Cr-8Co-3.5Cb-3.5W-3.5Mo-0.05Zr-0.01B. The starting material for the study was 1/4 to 3/8-inch-diameter rods prepared by hot rolling of extruded stock. After hot rolling, the material was solution treated at 2000 F for 1 hour and then intermediate-aged at temperatures from 1450 to 1850 F, the objective of which was to promote more ductile grain boundaries by precipitating discrete carbide particles. Subsequently, eight different combinations of deformation and aging treatments were applied.

On the basis of 1000 F creep and tensile properties, the optimum treatment was concluded to be as follows: intermediate age at 1650 F/24hr/AC, resolution at 2000 F/1 hr/AC, and age at 1100 F/64 hr + 1350 F/64 hr. At 1000 F, the tensile strength was about 240 ksi, notch-tensile strength 285 ksi, yield strength (0.2 percent offset) 175 ksi, reduction of area 16 percent, and elongation 15 percent. The time to produce 0.2 percent creep at 1000 F was 1500 hr at a stress of 150 ksi. Very little change in microstructure was observed after 1500 hr at 1200 or 1400 F, but an acicular phase, tentatively identified as sigma, appeared after aging at 1600 F. While 1600 F is expected to be far beyond the normal operating range for the alloy, the occurance of sigma at this temperature was taken to indicate the need for long-time microstructural-stability studies at the lower temperatures.

Effect of Composition of Microconstituents

The influence of chromium or gamma-prime formation in nickel-base alloys is being studied as part of TRW's program on microstructural stability of superalloys.(2) Six alloys based on TRW 1900 were investigated; these represented two combinations of aluminum-plus-titanium weight percentages (1Ti + 6.3 Al and 2Ti + 7Al) and three chromium levels (10, 5, and 0 percent). The remainder of the alloy was 10 Co, 9 W, 1.5 Cb, 0.03 B, 0.1 Zr, balance Ni.

Chromium was found to have two major effects on gamma-prime formation. Increasing the chromium levels appeared to (1) shift the gamma/gamma-prime eutectic reaction toward higher aluminum-plus-titanium levels and (2) to decrease the size (coarseness) of the gamma-prime precipitate. In both effects, more variation occurred between 0 and 5 percent chromium than between 5 and 10 percent.

Other observations were that increasing the chromium content of TRW 1900 appeared to increase the proneness to sigma formation and to lower the gamma-prime solution temperature, but increasing the chromium content also increased the stress-rupture life at 1400 and 1800 F.

PROCESS DEVELOPMENT

Melting and Forging of AF2-1DA Alloy

A 3000 lb production heat of Unitemp AF 2-1DA alloy has been melted (vacuum induction followed by vacuum consumable-arc remelting) and cast into 13-inch-diameter ingots, and press forged to a 6-inch octagon.(3) Considerable difficulty was encountered in forging the cast alloy because of its poor ductility. At 2100 F, the cast material was reported to have no ductility, and even within the specified working range, 2050 to 1850 F, the reduction of area was not over 18.9 percent at a strain rate of 20 in./in./sec. The forged octagon, however, was far more ductile than the cast ingot, showing a 50-degree F increase in workability range, and showing a reduction of area between 20 and 58 percent at 20 in./in./sec. in the range of 1850 to 2100 F.

Microstructural examination and analysis of extracted phases showed that a grain boundary network of gamma-prime was present in the cast ingot, but these were randomly distributed throughout the wrought structure. Neither sigma nor mu phase were found after thermal cycling under conditions designed to produce these phases.

Directional Solidification of TAZ-8B Alloy

Directional solidification of NASA's TAZ-8B alloy (NI-6Cr-5Co-6Al-8Ta-4W-4Mo-1.5Cb-1Zr-0.125C-0.004B) markedly increases its tensile strength, elongation, and creep-rupture strenth.(4) The alloy was cooled in a heated mold having controlled temperature zones, in order to promote the oriented grain structure. Figures 1 and 2 show the mechanical properties in comparison with random polycrystalline TAZ-8B alloy.

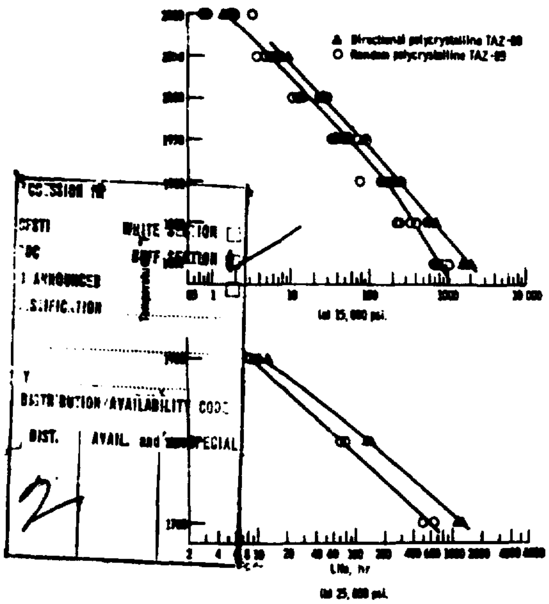

FIGURE 1. COMPARISON OF STRESS-RUPTURE PROPERTIES OF RANDOM AND DIRECTIONAL POLYCRYSTALLINE TAZ-8B ALLOY(4)

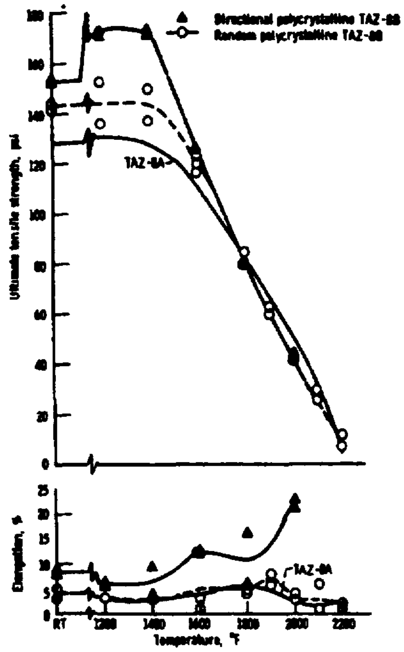

FIGURE 2. TENSILE PROPERTIES OF TAZ-8 ALLOY SERIES(4)

Manufacturing Methods for Producing Alloy L-605 Hardware

At Marquardt, Alloy L-605 has been used in the cold-worked condition for numerous aerospace applications, such as combustion chambers and tail pipes.(5) These combustion-chamber components were designed to utilize the high strength-to-weight ratios that can be obtained by cold shear spinning. However, in some cases gross cracking has occurred either during shear spinning or during a final expanding operation. Control of the microstructure and hardness was found to be particularly important for the shear spinning of Alloy L-605 forgings. Investigation of the effects of variations in iron, silicon, and manganese contents on hot and cold fabricability revealed no clearly defined effects of iron or manganese, but showed that silicon should be held to a maximum of 0.25 percent. Vacuum-induction melted plus vacuum-arc remelted material seemed to be slightly more fabricable than air-melted plus vacuum-arc-remelted material.

The optimum annealing parameters were found to vary from heat to heat of the same composition and with prior fabrication history; however, Marquardt recommended that annealing time and temperature be selected so as to minimize hardness, grain-growth, and grain-boundary precipitates. The forging temperature for shear spinning preforms should be high enough to minimize grain-boundry carbides and low enough for grain size control. The optimum temperature appeared to be approximately 2150 F.

The room-temperature strength properties of 20 percent cold-worked alloy could be increased significantly with a minimum decrease in ductility and notch sensitivity by an aging treatment of 700 F for 10 hours.

Tubing and Bar Process Development of Nickel-Chromium-Thoria Alloys

DuPont's final report on the development of manufacturing processes for TD Nickel-Chromium describes the procedures for obtaining 1/8-in. O.D. x 0.012-in wall tubing and 1/4-in.-diameter bar.(6)

The preferred process for tubing was to extrude 8-in.-diameter compacts to 4-in. billets for reextrusion to tube blanks. Extrusion conditions of 1-1/2-in. O.D. x 3/16-in. wall blanks were developed for both 1800 F and 2200 F extrusion temperatures, but better fabricability in subsequent processing was observed with the 2200 F extrusions.

Of several alternatives investigated for subsequent reduction to tubing, the best was to "tube reduce" to about 7/8-in. O.D. then cold draw with 25 percent area reduction per pass with intermediate anneals at 2200 F. The finished 1/4-in. tubing had ultimate tensile strengths of 6000 to 7000 psi at 2000 F.

Attempts to warm draw, as a means of increasing high-temperature strength, were of limited success, but further work would be necessary to bring this development to the degree reached by the cold-drawing route.

For producing rod experimentally, extrusion, swaging, rolling, and rotary forging were used effectively. Rod 1-in. in diameter had an ultimate strength of 15,000 to 20,000 psi and elongations of 5 to 18 percent at 2000 F. A limited amount of 1/4-in. rod was produced.

APPLICATIONS

Lightweight Heat Exchanger Materials

TD Nickel, L-605 and Hastelloy X alloys were evaluated as tubing materials in a tube-and-shell type heat exchanger for hydrogen-air service with a maximum air inlet temperature of 2540 F and a maximum metal temperature of 2140 F.(7) The heat exchanger was divided into three zones, with increasingly higher maximum metal temperatures, and accordingly made of different materials:

High-temperature zone: 2140 F - TD Nickel
Intermediate temperature zone: 1840 F - Alloy L-605
Low-temperature zone: 1540 F - Hastelloy X

The heat exchangers utilized 1/8-inch O.D. thin-walled (10 mils or less) tubing. Various coating systems applied to TD Nickel and Alloy L-605 were also evaluated.

The use of TD Nickel in the high-temperature zone was limited to a maximum metal temperature of 2000 F since no satisfactory coating systems were found. For a 9-mil-wall thickness, the maximum metal temperature recommended for uncoated, brazed TD Nickel tubing was 1950 F.

For L-605 alloy tubing having an 8-mil thick wall, the maximum metal temperature was 1840 F when the Misco MDC-1A coating was applied for oxidation protection. Alloy L-605 was generally found to be an attractive heat exchanger material having higher ductility than TD Nickel. Moreover, brazing and welding of the L-605 alloy were more readily accomplished than for TD Nickel. The advantage of TD Nickel over Alloy L-605 appeared to be marginal.

Hastelloy X tubing with a 3-mil wall was found to meet the service requirements of the low-temperature zone.

Cast Forging Dies

IN 100 and MAR-M200 have been selected from six candidate nickel and cobalt alloys for further investigation as precision-cast hot-forging dies for titanium alloys.(8) Over the temperature range of interest, these two alloys had higher compressive yield strengths and higher hot hardness than Alloy 713 C, Udimet 700, F-484, and X-40.

For the molding material, IIT Research Institute chose a zircon-base ceramic facing shell backed with sodium silicate bonded magnesite. This molding material was selected because of its high thermal diffusivity.

Materials For Transpiration-Cooled Turbine Blades

TD Nickel-Chromium*, DH242**, and Hastelloy X*** have been selected for investigation as 5-mil wire for eventual application in porous, transpiration-cooled gas-turbine blades.(9) Also recommended for further investigation and development was General Electric's 1541 alloy (Fe-15Cr-4Al-0.7 Y). These alloys were selected on the basis of cyclic oxidation tests at temperatures up to 2200 F for 600 hr, mechanical-property retention, and fabrication considerations.

Included in the oxidation evaluation were measurements of weight gain, weight of spalled oxide, and depth of oxidation penetration. TD Nickel-Chromium rated high in each of these categories. Alloy DH242 was also good in these respects, especially at 2100 and 2200 F, but it was surpassed by the iron-chromium-aluminum alloys.

Rentention of strength and ductility after the cyclic temperature exposure was found to be good for each of the four alloys.

Fabrication into fine wire was considered a difficulty only for TD Nickel-Chromium; GE1541 alloy is not yet widely available as wire. DH242 and Hastelloy X wire, on the other hand, are easily obtainable. Sintering, which will be refined to form a rigid structure from wire, is expected to be difficult for the GE1541 alloy because of its yttrium content, and possibly sintering will be difficult also with Hastelloy X. Electron-beam welding of all alloys except TD Nickel-Chromium was found to be good.

In summary, the alloys selected for further investigation as wire were the result of the judgments and evaluation of several areas spanning a wide range of conditions and performance characteristics.

* Trademark of Fansteel Metallurgical Corporation.
** Trademark of Driver-Harris Company. Nominal compisition is Ni-20Cr-1Cb.
*** Trademark of Union Carbide Corporation.

OXIDATION AND HOT CORROSION

Effect of Rare Earths

Preliminary observations of the effect of rare-earth additions on the oxidation resistance of nickel-base superalloys René 100 and AF 2-1DA have led to the conclusion that the behavior should not be generalized.(10) Addition of some elements, at the proper concentration, can improve oxidation resistance and/or hot-corrosion resistance at certain temperatures. The improved surface stability was found to be at the expense of mechanical properties, however.

As an example, static and cyclic oxidation tests of René 100 modifications at 1600 and 1800 F showed that certain additions of yttrium, gadolinium, manganese, yttrium plus thorium plus manganese, and lanthanum plus manganese improved the behavior of the base alloy by factors of 2 to 20. Lanthanum and cerium, which were least effective in oxidation, improved the hot-corrosion resistance of René 100 by factors of 10 to 30, although gadolinium was the most effective in improving both oxidation resistance and hot-corrosion resistance.

Tensile ductility and stress rupture were drastically reduced by the reactive metal additions, but heat treatment was suggested as a means of remedying the adverse effects.

Relationship to Composition

Allison Division of General Motors found that the volume loss in hot-corrosion tests of ten cast nickel-base superalloys decreased with increasing chromium and aluminum contents, and increased with increasing amounts of tungsten and molybdenum.[11] The variables were related by the equation:

$$\log_{10} VL = 5.85238 \times 10^{-9} T^3 - 1.33860 \times 10^{-5} T^2 + 6.32837 \times 10^{-2} W + 8.63834 \times 10^{-2} Mo - 6.77702 \times 10^{-2} Cr - 8.982 \times 10^{-2} Al + 11.2807,$$

where T is the maximum temperature in cycling test, VL is the volume loss, and W, Mo, Cr, and Al represent the weight percentages of these elements.

The alloys tested were 713C, 713C modified by increasing the chromium content by 2 percent, 713C modified by increasing the chromium as before and adding 0.38 percent yttrium, IN 100, GMR 235, PDRL 163, IN 728 NX, MAR-M421 (with chromium 1 percent lower than specification), MAR-M246, and Inco 717. Nominal compositions of the alloys are shown in Table 1 (boron, zirconium, and carbon omitted).

In statistically designed experiments, eight tests each cycling 500 times in a hot-corrosion rig to maximum temperatures of 1700 to 1900 F were run on each alloy.

TABLE 1. NOMINAL COMPOSITION OF NICKEL-BASE SUPERALLOYS[11]

Alloy	Cr	Ti	Al	Cb	Mo	W	Other
713C	14	0.85	6.0	2.3	4.5	-	-
713C (Mod Cr)	16	0.85	6.0	2.3	4.5	-	-
713C (Mod Cr Y)	16	0.85	6.0	2.3	4.5	-	-
IN 100	10.3	4.8	5.5	-	3.0	-	0.85V, 14 Co
GMR 235	15.5	2.0	3.0	-	5.3	-	10 Fe
PDRL 163	17	-	6.3	1.0	1.5	2	2 Ta
IN 728 NX	17	-	6.3	1.0	1.5	2	10 Co, 2 Ta
MAR-M421	15.5	1.8	4.3	1.8	1.8	3.5	10 Co
MAR-M246	9	1.5	5.5	-	2.5	10	10 Co, 1.5 Ta
Inco 717	11	1.0	7.7	2.4	3.5	-	9.5 Co

According to the report, PDRL 163 and IN 728 NX showed significantly better resistance to hot corrosion than all other alloys at cyclic temperatures through 1850 F. The 713C modifications were significantly better than standard 713C only at 1800 and 1850 F maximum cycle temperatures.

SYMPOSIUM NOTICE

The 71st Annual Meeting of the American Society for Testing and Materials (ASTM) will be held at San Fransico during June 23-28, 1968. There are many interesting symposia planned for the meeting. Two such symposia that should be of interest to readers of the Review of Recent Developments are:

Biaxial Behavior of Alloys sponsored by the ASTM-ASME-MPC Joint Committee on The Effect of Temperature on the Properties of Metals (two sessions, June 24).

Interfaces in Composites, sponsored by ASTM Committee D-30 on High-Modulus Fibers and Thin Composites (two sessions, June 28).

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DMIC Reviews of Recent Developments present brief summaries of information which has become available to DMIC in the preceding period (usually 3 months), in each of several categories. DMIC does not intend that these reviews be made a part of the permanent technical literature. Copies of referenced reports are not available from DMIC; most can be obtained from the Defense Documentation Center, Cameron Station, Alexandria, Virginia 22314.

R. W. Endebrock, Editor