

W92-17500

NASA SP-5940 (02)

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METALLURGY

A COMPILATION



NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Foreword

The National Aeronautics and Space Administration and the Atomic Energy Commission have established a Technology Utilization Program for the rapid dissemination of information on technological developments which have potential utility outside the aerospace and nuclear communities. By encouraging multiple application of the results of their research and development, NASA and AEC earn for the public an increased return on the public investment in aerospace and nuclear R&D programs.

The items in this compilation, all relating to metallurgy, are presented in two sections. The first presents descriptions of the mechanical properties of various alloys, ranging from TAZ-8B at +2200° F to investment-cast Alloy 718 at -320° F. The second describes methods of analyzing some of the constituents of various alloys, from optical detection of carbide precipitates in René 41 to X-ray spectrographic analysis of the manganese content of high-chromium steels.

Additional technical information on individual devices and techniques can be requested by circling the appropriate number on the Reader Service Card enclosed in this compilation.

Unless otherwise stated, NASA and AEC contemplate no patent action on the technology described.

We appreciate comment by readers and welcome hearing about the relevance and utility of the information in this compilation.

Jeffery T. Hamilton, *Director*
Technology Utilization Office
National Aeronautics and Space Administration

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Section 1. Mechanical Properties of Alloys

COOLING RATE AFTER SOLUTION ANNEALING AFFECTS PROPERTIES OF RENÉ 41

A preliminary study of the effects of variable post-annealing cooling rate (from 1975° to 1400° F) on René 41 alloy has indicated that:

1. The cooling rate profoundly affects many mechanical properties of the alloy. Within the useful range of cooling rate (<150° F/min), yield and ultimate strength vary by 20%.
2. The ductility at 1400° F varies almost linearly with total cooling time.
3. Neither Charpy impact strength nor ambient elongation is significantly changed by slowing the cooling rate from the normal 75° to 20° F/min; elongation at 1400° F was doubled to 36% for the test heat.
4. Minimum ductility does not necessarily occur at 1400° F, but is a function of cooling rate. For 20° F/min it is at $\leq 1100^\circ$ F, while for 75° F/min it is at 1200° F.

It is not known why high-temperature ductility is enhanced by slow cooling, although many alloys exhibit similar effects. The cause may be increased formation of globular carbide, grain-boundary gamma-prime precipitation, for formation of a soft zone adjacent to the grain boundary. Yet the

light microscope reveals only heavier carbide precipitation at the grain-boundaries of specimens cooled more slowly. On the other hand, the overall structure is typically banded, and the amount of carbide varies from location to location even on a single tensile specimen. Slow cooling improves high-temperature ductility and also provides more uniform mechanical properties.

It seems possible to trade strength for high-temperature ductility and to shift the ductility minimum to a harmless location. Only one heat was studied in great detail; thus it may have been chance that ambient elongation and impact resistance were not reduced by the lowest cooling rates. If this heat is an exception rather than the rule, the trade-off becomes more complex and slow cooling becomes less attractive.

Source: M. Prager of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18790)

Circle 1 on Reader Service Card.

DETERMINING SHORT-TRANSVERSE TENSILE PROPERTIES OF RENÉ 41 FORGINGS

Development of a special, composite test specimen has enabled tensile testing to be performed in the short-transverse direction on René 41 forgings from 1/2 to 3 inches thick. The specimen is produced by percussion stud welding René 41 extension rods onto a rough-machined cylinder of test metal. Required heat treatments and finish machining are performed after welding.

In a limited test program involving a turbine manifold flange, eight 1/2 in.-diameter by 1/2 in.-long cylinders were cut from the flange with the cylinders' axes parallel to the short transverse direction. After stud welding, the specimens were

solution heat treated at 1975° F for two hours, then aged at 1400° for 16 hours before final machining. This extended heat treatment was intended to promote diffusion and improve the weld-joint strength; in fact, only three of the eight specimens failed at the weld joint.

Source: E. G. Thompson and
R. E. Fish of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-19021)

Circle 2 on Reader Service Card.

PROPERTIES OF WELDMENTS BETWEEN THREE ALLOYS AND RENÉ 41

Tests comparing the properties of welds between René 41 and Hastelloy C, Hastelloy X, and Inconel 625 have shown the following: 1) The unnotched strengths were not affected by four post-weldment René 41 heat treatments; 2) the Hastelloy X weldment showed superior joint efficiency; 3) changes in notched tensile strengths could be attributed to weld dilution variations; and 4) hold times up to 500 seconds at 1500° F had no significant effect on notched or unnotched tensile strength.

The tests were conducted on specimens TIG welded using Hastelloy W filler metal, and mechanical properties were measured at room temperature and at 1500° F.

Source: E. F. Cook, of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18854)

Circle 3 on Reader Service Card.

EFFECT OF RENÉ 41 HEAT TREATMENTS ON THE TENSILE PROPERTIES OF HASTELLOY C

Single and multiple heat treatment cycles, such as those commonly used in curing René 41 (one hour at 1975° F; air cool; 16 hours at 1400° F; air cool) have been shown to affect the tensile properties of Hastelloy C only moderately. Thus, non-stress-critical parts made of Hastelloy C may be installed in René 41 assemblies prior to heat treatment.

Tensile tests were conducted at room temperature and 1400° F on notched and unnotched specimens. The material was tested in the annealed condition, and after being exposed to one, two, and three René 41 heat-treatment cycles. No significant difference was found between the tensile properties of specimens heat-treated for one, two, or three cycles.

At room temperature, the tensile properties of heat-treated Hastelloy C was below that of an-

nealed material by 84% in elongation, 17% in notched strength, 15% in yield strength, and 9% in ultimate strength. At 1400° F, heat-treated material showed 32% less elongation and slightly higher strength than annealed material.

The notched-to-unnotched strength ratios of heat-treated material were in the range of 0.75 to 0.78 at room temperature. Although these ratios are lower than those of most alloys, notch strengths are still above the yield strengths by 30 to 50%.

Source: R. R. Ferguson and
T. G. McNamara of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18895)

Circle 4 on Reader Service Card.

METALLURGICAL CHARACTERISTICS OF HASTELLOY X

Pertinent information has been compiled on the properties of Hastelloy X through the temperature range -423° to 1800° F. This easily fabricated nickel-base alloy has exceptionally good corrosion resistance at high temperatures, superior strength, good resistance to radiation, and good notch toughness.

The information includes data on the tensile properties of Hastelloy X forgings, strip, weldments, and brazements, presented both as individual test data and as statistical averages. Mechanical properties relating to shear strength, bearing strength, low-cycle fatigue, and thermal cycling are also presented. In addition, the manu-

facturing and fabrication processes, from raw material melting through production of individual hardware components, are discussed. Metallurgical analysis of thermal treatments and processing are also provided, as are specific data on large, heavy-wall forgings and thin strip.

Source: L. F. Glasier, Jr. of
Aerojet-General Corp.
under contract to
AEC- NASA Space Nuclear Systems Office
(NUC-10302)

Circle 5 on Reader Service Card.

LOW-TEMPERATURE PROPERTIES OF CAST HASTELLOY X

Evaluation of Hastelloy X as a cryogenic material has shown that the alloy has low-temperature properties similar to 310 CRES, with good ductility and toughness at temperatures as low as -423° F.

Tensile tests were conducted on 1/4 in.-diameter, investment-cast bars, both as cast and after machining and homogenization at 2225° F. Room-temperature tests were performed conventionally, with load applied at a rate of about 2500 lb/min from zero through the specimen yield strength. Cryogenic tests were performed

similarly at -425° F in liquid hydrogen, at -370° F in liquid nitrogen, and at -110° F in gaseous nitrogen. Results showed that Hastelloy X is suitable for cryogenic applications where high strength is not required.

Source: C. O. Malin of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18915)

Circle 6 on Reader Service Card.

COMPARISON OF 8% COLD-REDUCED HASTELLOY C, HASTELLOY X, AND INCONEL 625

Comparative study of the mechanical properties of 8% cold-reduced Hastelloy C, Hastelloy X, and Inconel 625, before and after four René 41 heat treatment cycles, indicates that: 1) Inconel 625 shows the most stable mechanical properties; 2) both at room temperature and at 1500° F, the René 41 heat treatments improve the ductility and lower the strength of all three alloys; 3) hold times up to 500 seconds at 1500° F have no effect on the properties of the alloys; and 4) for all three alloys,

before and after heat treatment, at room temperature and at 1500° F, the 8% cold-reduced material is superior to the as-cast alloy.

Source: E. F. Cook and
E. F. Green of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18969)

Circle 7 on Reader Service Card.

NICKEL-BASE SUPERALLOY TAZ-8

The development of the TAZ-8 family of nickel-base alloys began more than a decade ago. These alloys with their low-chromium and high refractory content were the prototype of today's high strength cast materials. They were developed for use at temperatures of 1500° to 2200° F. They

have limited workability and possess good strength at high temperatures.

The nominal weight percent composition used as a starting base for systematic alloying studies was 8 molybdenum, 6 chromium, 6 aluminum, 1 zirconium and the balance nickel. The first phase

of this investigation, completed in the early 1960's, examined the effects of carbon, titanium, vanadium, tungsten and tantalum as alloying elements. All of these early experimental alloys were induction melted in an inert atmosphere.

The most promising of this group of alloys was designated TAZ-8 and had the following composition: 6 chromium, 6 aluminum, 4 molybdenum, 8 tantalum, 4 tungsten, 2.5 vanadium, 1 zirconium, 0.125 carbon and the balance nickel. This alloy's stress-rupture life, use temperature, and tensile strength at high temperatures, exceeded or compared favorably with the then-available commercial nickel-base alloys. TAZ-8 also demonstrated limited workability in both rolling and forging tests. Bars were forged at room tem-

perature to a reduction of 50% and 0.100-in. thick sheets were reduced by rolling to 0.015 in. sheet.

The oxidation-resistance of this alloy is somewhat less than that of the better nickel-base alloys, and as with most alloys it would require a protective coating for prolonged use at high temperatures.

The later alloys in the TAZ-8 series investigated, examined the effects of additional alloying elements, and some of these alloys showed marked improvement in oxidation and thermal shock resistance.

Source: J. C. Freche of
Lewis Research Center
(LEW-90226)

Circle 8 on Reader Service Card.

SUPERALLOY TAZ-8A SHOWS IMPROVED OXIDATION RESISTANCE UP TO 2200° F

To provide a high-strength, high-temperature, workable nickel-base alloy with improved oxidation resistance, the chemistry of the original TAZ-8 alloy was further modified, and vacuum melting techniques were employed. The basic formula was modified by substituting 2.5% niobium for the vanadium constituent and by adding 0.004% boron.

The new alloy, designated TAZ-8A, generally retained the good elevated temperature strength and workability of TAZ-8, while showing excellent high temperature oxidation resistance. After 310 hours exposure to air at 1900° F, TAZ-8A had a weight gain of 1.8 mg/cm². The depth of the total affected zone, external oxide scale plus depletion zone, was 0.4 mil. This compares with a weight gain of 3.0 mg/cm² and a total affected zone depth of 3.3 mils for René 41 after 100 hours exposure at 1900° F. In sheet form, its oxidation resistance after 8 hours at 2200° F was approxi-

mately the same as that of René 41 at 1900° F.

Tensile strengths of TAZ-8A after rolling and heat treatment ranged from an average of 185,000 psi at 1400° F to 3000 psi at 2200° F. Maximum elongation was 55% and occurred at the latter temperature. At 1900° F, the average tensile strength was 64,500 psi in the as-cast condition, and 54,000 psi after rolling and heat treatment.

Stress rupture data for low and intermediate stress levels were obtained in the as-cast condition. Use temperatures for 500-, 100-, and 10-hour life at 15,000 psi are 1815°, 1895°, and 2010° F, respectively. At 8000 psi and 2125° F, rupture life was 13 hours.

Source: W. J. Waters and
J. C. Freche of
Lewis Research Center
(LEW-10115)

Circle 9 on Reader Service Card.

HIGH-STRENGTH SUPERPLASTIC SUPERALLOY

A high-strength superplastic superalloy has been produced by extruding pre-alloyed TAZ-8A powder. The alloy was remelted and converted to pre-alloyed powder by inert gas atomization. The

powder was then extruded into bar stock and evaluated, both as-extruded and after various heat treatments.

Tensile test specimens machined from extruded

pre-alloyed TAZ-8A powder showed very high ultimate tensile strengths (228,000 psi at room temperature; 164,000 psi at 1400° F). These values are higher than those obtained with any known cast or wrought nickel-base alloy, and are about 1-1/2 to 2 times greater than for as-cast TAZ-8A.

The material also exhibits superplastic behavior at high temperature. Neck-free elongations greater than 450% at 1800° F and 600% at 1900° F have been produced. This superplastic property can be used to form the alloy into an endless variety of configurations by applying low strain rates and very low deforming loads while the material is held

at high temperature. The alloy can thus be readily shaped at very low processing cost, and the superplasticity can subsequently be removed by heat treatment. This superplastic property, achieved by pre-alloyed powder techniques, contributes to the attractiveness of the alloy for wide commercial use.

Source: J. C. Freche,
J. Waters, and
R. L. Ashbrook of
Lewis Research Center
(LEW-10805)

Circle 10 on Reader Service Card.

HIGH-TEMPERATURE PROPERTIES OF NICKEL-BASE SUPERALLOY TAZ-8B

A recently developed, high-temperature nickel alloy, designated TAZ-8B, has the desirable properties of high strength, ductility, good impact and oxidation resistance, microstructural stability, workability potential, and the ability to show improved strength and ductility when directionally solidified. The alloy's nominal composition in weight percent is 8 tantalum, 6 chromium, 6 aluminum, 4 molybdenum, 4 tungsten, 1.5 niobium, 5 cobalt, 1 zirconium, 0.125 carbon, 0.004 boron, and the balance nickel. The alloy achieves high-temperature strength by a combination of mechanisms. Besides forming the Ni₃Al intermetallic phase, solid-solution strengthening occurs due to the presence of heavier elements such as Ta, W, and Mo, which form stable carbides such as TaC.

The high-temperature properties of TAZ-8B are achieved by modifying the composition of the earlier alloy, TAZ-8A. Cobalt is added so that the gamma-prime phase remains stable to higher temperatures, and the niobium content is reduced to increase ductility.

A directionally oriented grain structure can be achieved through controlled solidification. Directional solidification further increases high-temperature strength and intermediate-temperature ductility by effectively eliminating intergranular failures along grain boundaries transverse to the loading axis. In the directionally solidified form, TAZ-8B at 1400° F has an ultimate tensile strength of approximately 180,000 psi and an elongation of about 6% (1400° F is the alloy's minimum ductility point). Furthermore, the alloy appears to be free from embrittlement by sigma phase formation, a problem encountered with other high-strength nickel-base alloys.

Source: J. C. Freche and
W. J. Waters of
Lewis Research Center
(LEW-10355)

Circle 11 on Reader Service Card.

CRYOGENIC PROPERTIES OF THE 18% NICKEL MARAGING STEELS

The 18% nickel maraging steels develop their maximum cryogenic properties under relatively simple heat treatment. When annealed for one hour and air cooled from 1500° F, the austenite phase is transformed by a diffusionless shear

mechanism into bodycentered cubic martensite, resulting in an alloy with exceptional toughness and moderate hardness. On aging for 6 hours at 900° F, strength and hardness are substantially increased by interaction of the nickel with harden-

ing elements cobalt, molybdenum, and titanium.

As-forged and heat-treated material both show good tensile and notch toughness properties at room temperature. At -320°F , the heat-treated material has been reported to show increased strength with little loss of ductility or notch toughness.

Tests have shown the tensile properties of aged 18% nickel (300-grade) maraging steel to be somewhat lower than has been previously reported. The room-temperature tensile strength is increased by aging, but the ductility is diminished below 10%. The tensile strength of aged material increases to over 350 ksi at -320°F , but ductility decreases still more. Similarly, low impact notch properties measured at room temperature become still lower at -320°F .

Dilatometer tests at cryogenic temperatures

reveal that the thermal contraction of 18% nickel maraging steel is greater in the as-forged condition than in the aged condition. At liquid nitrogen temperature, the contraction of maraging steel is 15% less than AISI 4340 steel. Thermal expansion on heating age-hardened 18% nickel (300-grade) steel to 1093°F is less than the thermal expansion of AISI 4340 steel. Thermal contraction occurs as the temperature is increased from 1150° to 1225°F , reflecting the reversion of martensite to austenite.

Source: E. F. Green of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18280)

Circle 12 on Reader Service Card.

TUNGSTEN-HAFNIUM-CARBON ALLOY RETAINS HIGH STRENGTH AT TEMPERATURES ABOVE 2500°F

A recently developed tungsten-hafnium-carbon alloy (W-0.20Hf-0.26C) shows tensile strengths of 88,200 psi at 3000°F and 62,500 psi at 3500°F .

In earlier work, it was shown that (1) the tensile and creep strengths of arc-melted tungsten at 2500° to 3500°F were increased (in decreasing order of effectiveness) by adding hafnium, tantalum, niobium, and rhenium, and (2) adding carbon to a nominal tungsten-1% niobium alloy resulted in a further increase in strength due to stabilization of the cold-worked structure by carbide precipitates.

More recent work has examined the tungsten-hafnium system in greater detail, including investigation of several ternary alloys. It was found that the effectiveness of solid-solution strengthen-

ing could be correlated with the difference in atomic size between the solute and tungsten, with the larger differences promoting greater strengthening. Carbon additions to the alloys produced various degrees of strengthening, with the largest effect found in the tungsten-hafnium-carbon alloys. Differences in degree of strengthening were found to be correlated with carbide particle size, with the finest particle size (most strengthening) observed in the tungsten-hafnium carbon alloys.

Source: P. L. Raffo and
W. D. Klopp of
Lewis Research Center
(LEW-366)

Circle 13 on Reader Service Card.

TUNGSTEN-FIBER-REINFORCED NICKEL SUPERALLOY

A new tungsten-fiber reinforced, nickel-base superalloy has a 1000-hour strength of 37,000 psi at 2000°F . This is equivalent to the 1000-hour strength of the best previous composite, and is six times as strong as commercially available

nickel superalloys. Because of its high strength-to-density ratio, the composite can be considered for uses where reduced weight, rather than greater strength, is desired.

The properties of fiber-reinforced materials

are adversely affected by fiber-matrix reactions. The strength of the composite is determined by the retained strength of the fiber; fiber-matrix reactions can substantially reduce fiber strength. Accordingly, one of the efforts being made to improve the strength of composites is to develop stronger fibers. The stronger the fiber, the stronger the composite for given degree of fiber-matrix reaction.

Matrix composition, fabrication technique and fiber diameter are selected to minimize fiber-matrix reactions and preserve composite strength. The fibers are 0.015-inch in diameter and specially drawn for high strength. The matrix composition in weight percent is: nickel 15, tungsten 25, chromium 15, titanium 2, and aluminum 2; the

fibers are tungsten with 2% thorium oxide. The composite is prepared by slip-casting a powdered superalloy-water slurry into an array of fibers. The dried slip casting is consolidated by pressing and heating. Fiber content can be varied from zero to 80 volume percent to achieve the desired reinforcement. The 70 volume percent unidirectional-oriented fiber composite has a 100-hour rupture strength of 49,000 psi at 2000° F, and a 1000-hour strength of 37,000 psi at 2000° F.

Source: D W. Petrasek and
R. A. Signorelli of
Lewis Research Center
(LEW-10424; 10933)

Circle 14 on Reader Service Card.

HIGH-STRENGTH TUNGSTEN ALLOY SHOWS IMPROVED DUCTILITY

As the metallic element with the highest melting point, tungsten is of interest for load-bearing components which must operate at extremely high temperatures. A disadvantage of tungsten is that it becomes brittle at temperatures below about 250° F, and alloying to increase strength often also increases the ductile-to-brittle transition temperature.

Extensive studies on alloying effects in arc-melted tungsten have led to the development of an alloy (W-4Re-0.35Hf-0.024C) which combines superior strength at elevated temperatures with improved ductility at lower temperatures relative to unalloyed tungsten. The alloy is prepared by consumable-electrode vacuum arc-melting, and

can be fabricated into rod, plate, and sheet. At 3500° F, the alloy has demonstrated a maximum tensile strength of 75,400 psi, an eightfold strength advantage over the 9,000 psi strength of unalloyed tungsten. Sheet material of this alloy can be bent at temperatures as low as 175° F, compared to 250° F for unalloyed tungsten.

Source: W. D. Klopp,
L. S. Rubenstein,
P. L. Raffo, and
W. R. Witzke of
Lewis Research Center
(LEW-10257)

Circle 15 on Reader Service Card.

COBALT-TUNGSTEN, FERROMAGNETIC HIGH-TEMPERATURE ALLOY

A new cobalt-base alloy which combines both good high temperature strength and magnetic properties has a composition in weight percent of 7-1/2 tungsten, 2-1/2 iron, 1 titanium, 1/2 zirconium, 1/2 carbon, and the balance cobalt.

Systematic alloying modifications were made to an alloy which had been developed for high temperature service as a structural material for space power systems. Magnetic induction was deter-

mined as a function of test temperatures up to 1800° F. The new alloy in the aged condition (72 hours at 1700° F) has a magnetic induction of 8.4 kilogauss when exposed for 1000 hours at 1300° F. Its coercive force in the aged condition was 10.5 oersteds at room temperature. At projected use temperatures of 1200°, 1300°, and 1400° F and a stress of 42,500 psi, the stress-rupture life was 8000, 850, and 4 hours, respectively.

This alloy can be rolled into sheet form, and at 1300° F and 42,500 psi the stress-rupture life of the annealed sheet was about 80 hours. This alloy compares favorably in stress-rupture properties with the strongest high-temperature magnetic alloy commercially available today.

Source: R. L. Ashbrook, A. C. Hoffman, G. D. Sandrock, R. L. Dreshfield, and J. C. Freche of Lewis Research Center (LEW-10378)

Circle 16 on Reader Service Card.

MECHANICAL PROPERTIES OF INVESTMENT-CAST ALLOY 718

Data are now available on the cryogenic and room-temperature properties of investment-cast Alloy 718. Minimum mechanical properties have been determined for this alloy, in both the aged and as-welded conditions, from room temperature to -320° F. Test specimens of the alloy were solution annealed and aged, then TIG welded using Alloy 718 filler metal.

Fractures obtained during tensile testing generally occurred in the weld metal, and the greatest elongation occurred within the base metal, in the

heat affected zone. The joint efficiency of welds in aged Alloy 718 is about 50% that of welds heat-treated after welding. Weld-joint efficiency can be significantly improved if the alloy is solution annealed and aged after welding.

Source: E. F. Green of North American Rockwell Corp. under contract to Marshall Space Flight Center (MFS-19020)

Circle 17 on Reader Service Card.

SHORT-TRANSVERSE MECHANICAL PROPERTIES OF ALLOY 718 PLATE

A series of tests has been conducted at room temperature to evaluate the short-transverse mechanical properties of Alloy 718 components machined from 0.250-in. plate. Results indicate that:

1. The ultimate tensile strength (UTS) in the short-transverse direction is 174 to 177 ksi, or 88% of the UTS in the longitudinal direction.
2. The maximum fiber stress is over 200 ksi.
3. Ductility at a transverse fracture surface in the plate material is about 7%, as compared to 20% in the longitudinal direction (determined from elongation based on local deformation between 0.050-in. gage lines).
4. Because of these results, it has been determined that electron-beam welding of Alloy 718 cannot

produce joints of adequate strength and integrity to join loading stubs to Alloy 718 plate in the short-transverse configuration.

The short-transverse strengths achieved in this series of tests are all higher than the 100 ksi value assumed for Alloy 718 during previous fabrication of rocket-engine components. The actual ductility is difficult to measure on 0.025-in. plate, particularly where a considerable fraction of the plate thickness is influenced by fusion welds.

Source: J. G. Fisher of North American Rockwell Corp. under contract to Marshall Space Flight Center (MFS-19005)

Circle 18 on Reader Service Card.

PRODUCING GRAPHITE WITH DESIRED PROPERTIES

Precise control of particle size, shape, and distribution, using such modern metallurgical techniques as X-ray diffraction, electron microscopy,

and porosimetry, has enabled the synthesis of graphite structures, both isotropic and anisotropic, with very unusual properties.

Isotropic graphites show the same mechanical, thermal, and electrical properties in all directions. Some newly produced graphites are almost perfectly isotropic, have thermal expansion coefficients two or three times those of ordinary graphites, and show high electrical resistivities. Because these graphites can be extruded, limitations on the production of certain sizes and shapes can be reduced considerably. A wide range of commercial application is possible, in components for turbine seals, pump impellers and housings, gas compressors, and tools for electric-discharge machining.

Anisotropic graphite does not exhibit uniform properties. Its strength may lie in one direction while its desirable heat-conduction characteristics lie in another. Such anisotropic graphites approach the anisotropy of pyrolytic graphite, and have the added advantages of lower cost,

much greater section thickness, higher across-grain strength, freedom from delamination, and flexibility in accepting additives such as radiation absorbers and oxidation inhibitors. They may be used to form high-temperature heat shields, since they can be molded by low-cost processes. They also appear to have potential uses in high-temperature piping and support structures, and as furnace electrodes for arc melting of steel.

Source: M. C. Smith,
R. J. Imprescia,
R. D. Reiswig, and
J. M. Dickinson of

Los Alamos Scientific Laboratory
under contract to

AEC-NASA Space Nuclear Systems Office
(NUC-11001)

Circle 19 on Reader Service Card.

COMPARATIVE CRYOGENIC PROPERTIES OF 310 CRES WELDS

Comparison of the cryogenic properties of 310 CRES welds filled with Inconel 92, versus those filled with Hastelloy W, has shown the latter to be markedly superior.

More specifically, the findings were:

1. The ultimate tensile strength (UTS) and yield tensile strength (YTS) of Inconel-filled weldments equaled or exceeded those of the parent metal only at room temperature. At -423°F , both the UTS and YTS of Inconel 92 welds were considerably less. The YTS of Hastelloy W welds was considerably higher than that of Inconel 92 welds at all test temperatures, and was much higher at -423°F .
2. The ductility of the Inconel 92 welds was equivalent to that of the parent metal and the Hastelloy W welds at room temperature. The ductility of Inconel 92 was appreciably lower at -320°F and -423°F , but elongation values at these temperatures remained acceptable.

3. Notch toughness for Inconel 92 weldments was better than that for Hastelloy W at -320°F (both have good service records), but Hastelloy W was markedly better at -423°F .
4. Inconel 92 showed greater susceptibility to weld-restraint cracking than Hastelloy W.

These data were statistically analyzed using the FORTRAN IV GMIN computer program. The superiority of Hastelloy W as a welding filler for 310 CRES components to be used at cryogenic temperatures was definitely established.

Source: R. E. Fish and H. L. Romig of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18989)

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CRYOGENIC FATIGUE PROPERTIES OF SHEET, BAR, AND CAST MATERIALS

The cryogenic fatigue and tensile properties of nine materials in sheet form, seven materials in wrought bar form, and two materials in cast bar

form have been determined for operating lifetimes of 10^4 to 10^7 cycles at room temperature, at -320°F , and at -423°F . Sheet materials with two sur-

face finishes were evaluated for fatigue under full reverse bending. Bar materials were tested in axial-loading and/or rotating-beam fatigue.

Materials evaluated were: 6061-T6 sheet and wrought bar; 7075-T73 sheet and wrought bar, and Tens 50-T6 cast bar aluminum alloys; Ti-5Al-2.5Sn (extra low interstitial) wrought bar; AISI 310 CRES sheet and wrought bar; A286 sheet; Inconel 718 sheet and wrought bar; iron and nickel-base age-hardenable alloys; and AISI 4340 wrought bar high-strength steel.

Results, correlating stress versus number of cycles to failure, are available in graphical and tabular form.

Source: E. F. Green of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18427)

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Section 2. Analysis of Metals and Alloys

ULTRASONIC METAL ETCHING FOR METALLOGRAPHIC ANALYSIS

Selective metal etching for metallographic analysis may be achieved by an ultrasonic etching method. The ultrasonic etch delineates microstructural features that may not be discernible in specimens prepared by standard chemical etching procedures.

A magnetostrictive transducer is used to generate ultrasonic vibrations, which are transmitted through a layer of distilled water to the

metal specimen. Cavitation bubbles in the ultrasonically excited water produce preferential etching of the metallurgical phases or grain boundaries, depending on the hardness and toughness of the specimens.

Source: S. G. Young of
Lewis Research Center
(LEW-11230)

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ANALYSIS OF ALLOY 718 FOR SILICON CONTENT

X-ray emission spectrometry provides a simple, accurate means for quantitatively determining the amount of silicon in heat-resistant alloy castings.

Eight Alloy 718 standards of known silicon content were used to establish an analytical curve. The standards and samples were polished normally using 320-grit silicon carbide paper. A final polish of 600-grit Al_2O_3 powder was employed to remove any traces of silicon carbide from the metal surface.

The silicon K_{α} emission-line intensity was determined for each standard, and the best linear least-squares fit was constructed.

The silicon content of test specimens can now be easily established by comparison with this calibration line. The minimum quantity of silicon in Alloy 718 detectable with the process actually used

is 0.04% (two sigma confidence level). This limit could be reduced by testing a larger sample surface area. The accuracy of the method is estimated to be $\pm 0.01\%$ for a silicon content of 0.20% (one sigma confidence level). Improved accuracy could be achieved with larger sample surface areas and increased integration periods. A single analysis can be made in 45 min., but multiple samples measured in parallel could significantly reduce the time per sample.

Source: R. T. Kessler of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18827)

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RENÉ 41 CARBIDE PRECIPITATES METALLOGRAPHICALLY IDENTIFIED BY COLOR TINTING

The ductility of René 41 is believed to be largely governed by the nature of its grain boundary carbide precipitate particles. However, observation of conventionally prepared metallographic samples with the light microscope does not provide conclusive evidence as to the tensile elongation. One handicap has been the inability of metallurgists to distinguish between the two principle carbide precipitate phases, M23C6 and M6C.

Color tinting has now been applied to solve this

problem. Using an electrolytic chromic acid etchant followed by a dip in an alkaline permanganate solution, it has been found that the M6C phase is colored; the other is not.

Source: M. Prager of
North American Rockwell Corp.
under contract to
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HEAT TREATMENT OF RENÉ 41 INVESTIGATED BY ELECTRON MICROSCOPY

Electron microscopy has been employed to detect microstructures in René 41 alloy that are detrimental to welding. Microstructural changes resulting from variations in heat treatment have also been investigated, because the strain-age cracking mechanism in René 41 appears to be related to the formation of gamma-prime and metal carbide precipitates.

Electron microscopy was used in the study because the René 41 gamma-prime precipitate is so fine that 10,000 \times magnification is needed to observe phase morphology.

The electron micrographs produced in this study constitute an extensive catalog of the microstructure of René 41 as influenced by various heat treatments, and form the basis for a better understanding of acceptable fabrication practices.

Source: W. E. Hensley of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18633)

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X-RAY EMISSION SPECTROMETRY: DETERMINING CHROMIUM AND ZIRCONIUM IN COPPER ALLOYS

X-ray emission spectrometry has recently emerged as a valuable technique for rapid chemical analysis. Essentially qualitative in nature, the technique can be made quantitative in specific cases by means of various calibration devices. The remainder of this compilation presents a number of these specific cases and calibration devices.

In analyzing for chromium (Cr) and zirconium (Zr) in a copper (Cu) alloy, no suitable standard samples were available for calibrating the spectrometer. Instead, three series of synthetic standards were created, with known concentrations of Cr and Zr in: 1) water; 2) a 25 mg/ml Cu solution; and 3) a 50 mg/ml Cu solution. The intensities of the Cr and Zr K_{α} spectral lines were measured

for each solution, and were plotted against concentration for each element to provide a calibration curve.

It is important to note that, while such solution methods as this permit the analysis of materials using readily prepared synthetic standards, the possibility of absorption reducing the x-ray line intensity must be considered. Other solution techniques apply to materials having the determined elements present in concentrations of 5-25%. These relatively high percentages permit the use of dilute solutions, which reduces absorption while retaining sufficient line intensity. The copper casting alloys analyzed by this technique, however, have an anticipated percentage range of 0.1

to 0.5% chromium and zirconium. It is necessary to analyze concentrated solutions, in order to achieve sufficient spectral line intensity. Under such circumstances, careful control of the solution or alloy matrix composition is required for accurate analysis. In this particular case, not only does matrix (Cu) concentration affect the Cr and Zr line intensities appreciably, it also affects the slope of the calibration line, with increasing copper concentration decreasing the slope. Therefore, in order for the synthesized-standard calibration

to remain valid, precise constant-weight alloy test specimens must be dissolved in known volumes of acid in order to achieve a solution matrix concentration matching that of the standard.

Source: R. T. Kessler of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
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NEUTRON ACTIVATION ANALYSIS TRACES ORIGIN OF COPPER ARTIFACTS

A simple method of identifying the source of copper artifacts employs neutron activation analysis to identify impurities that occurred in the ores and were passed by processing into the metallic copper. Three basic types of ore are involved: 1) naturally occurring metallic copper, from which the oldest copper implements were made; 2) oxidized ore, mined more recently and converted to metal by heating with charcoal; and 3) reduced (mainly sulfide) ores, mined still more recently and converted to metal by first roasting to produce oxides and then heating the oxides with charcoal.

In order to identify the impurities that would be passed into the metal, samples of various ores were converted to metallic copper by the most primitive methods known for each ore type. Optical spectroscopy and neutron activation analysis were used to determine quantitatively the impurities present in the copper metal and those lost in the slag.

The impurities most likely to be found in a copper artifact are silver, arsenic, bismuth, iron, antimony, and lead. These impurities have been categorized into nine concentration levels. A frequency distribution has been tabulated, relating impurity and level of concentration to ore type.

Artifacts can thus be analyzed to determine the concentration level of each impurity and the probable ore type from which it was made can be determined from the tables.

Source: M. Conway, P. Fields,
M. Kastner, J. Milsted, and
D. Metta of
Argonne National Laboratory and
E. Olsen of
Chicago Natural History Museum
(ARG-119)

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DETERMINING ALUMINUM IN HIGH-TEMPERATURE NICKEL ALLOYS

In this application, an X-ray vacuum spectrometer was used, equipped with a 9 Å ultrathin-window flow-proportional counter and a low noise preamplifier. This provides increased sensitivity to X-ray radiation from low atomic number elements. A chromium-target X-ray tube was operated at 50 kV and 60 mA for efficient excitation of the aluminum K spectral series. A 0.030 in. beam tunnel and a large window, 0.020 by 1.5 in.

Soller slit, were selected to provide optimum balance between resolution and line intensity. A Pentaerythritol (PET) crystal having a 2d spacing of 8.750 Å was used as the analyzer. Pulse height selection was required to discriminate against higher order interference; the conditions were selected to detect the aluminum K_{α} radiation with a nominal pulse amplitude of 15 V centered in an 8 V window.

The alloy standards used to establish the calibration curve were prepared by polishing with 600-grit silicon carbide paper. A National Bureau of Standards Inco 713C standard containing 4.34% aluminum was chosen as an intensity reference, and the intensity ratio of I_x / I_{434} was plotted vs weight percent aluminum. (This practice will simplify the use of data accumulated over an extended period of time.) Seven of the calibration standards used were NBS primary standard samples, while

the balance were secondary standards obtained by the Spectrographic Laboratory from various sources.

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North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18824)

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DETERMINING ZIRCONIUM IN HIGH-TEMPERATURE NICKEL ALLOYS

Again, the vacuum spectrograph was used. A tungsten-target X-ray tube was operated at 75 kV and 50 mA to excite the zirconium K spectral series. A 0.030 in. beam tunnel and a 0.005 by 3 in. Soller slit with a lithium fluoride analyzing crystal were required to completely resolve the zirconium K_{α} line from the adjacent niobium and molybdenum spectral lines. Vacuum pathway, scintillation counter, and pulse height selection were utilized to gain maximum line intensity. Background correction was made by measuring its intensity adjacent to the zirconium peak. An integration

period of 100 seconds was chosen for an optimum balance between precision and speed.

The calibration curve was found to be linear from 0.0 to 0.46% Zr. The threshold detection level was 0.005%, and the precision was 0.003% at a concentration of 0.05%.

Source: R. T. Kessler of
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under contract to
Marshall Space Flight Center
(MFS-18825)

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DETERMINING COPPER IN 2219 ALUMINUM ALLOY

A platinum-target X-ray tube was operated at 70 kV and 40 mA to excite the copper K series. The K_{α} spectral line was resolved using a LiF crystal, with a 0.010 in. beam tunnel and a 0.005 by 3.0 in. Soller slit.

Standard 2000 series aluminum alloys were used for calibration purposes. Both standards and test specimens were prepared by wet polishing techniques, using 600-grit silicon carbide. The

technique yields a linear calibration curve over the range 3.96 to 6.56% copper, with an accuracy of 0.03% at 6.20%.

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QUANTITATIVELY ANALYZING LOW-ALLOY STEELS

The same general technique as that described in the preceding item was employed to quantitatively analyze for Cr, Ni, Mn, and Mo in low-alloy steels. NBS low-alloy steel spectrographic standards were used to establish calibration curves.

Source: R. T. Kessler of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
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DETERMINING NICKEL CONTENT OF ELECTROFORMING BATHS

X-ray emission spectrometry enables rapid analysis of nickel concentration in electroforming baths. Very accurate results can be obtained by calibrating the system against known standard solutions and by incorporating an internal zinc standard to reduce the effects of selective X-ray absorption.

Calibration standards are prepared by dissolving known weights of pure nickel metal in 10 ml of 1:1 nitric acid, and adding 10 ml of a 1 mg/ml solution of zinc oxide in 1:4 nitric acid. Sufficient ammonium sulfate is added to approximate the absorption coefficient of an electroforming bath of the same concentration, and the solution is diluted to 100 ml in a volumetric flask.

Test samples are prepared by adding the same quantity of zinc solution to 10 ml of electroforming bath, and diluting to 100 ml.

The same procedure is used to analyze both standard and test samples. The spectrogoniometer or standard X-ray fluorescence apparatus is set to record the K_{α} line intensity of first nickel, then zinc, and the ratio of the two readings is plotted against nickel concentration. The known nickel concentrations in the standards establishes a calibration line, from which the concentrations of the test samples can be read. The accuracy of the technique is estimated to be ± 0.02 oz/gal, for electroforming baths with nickel concentrations from 5 to 15 oz/gal.

Source: R. T. Kessler of
North American Rockwell Corp.
under contract to
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DETERMINING MANGANESE IN HIGH-CHROMIUM STEELS

X-ray spectrometric analysis of small amounts of manganese (Mn) in steels containing a high percentage of chromium (Cr) is difficult because of the proximity of the chromium K_{β_1} line (2.085 Å) to the analytical K_{α} line for manganese (2.103 Å). Separation of the two lines by pulse height selection is precluded by the fact that Cr and Mn are adjacent elements in the periodic table.

However, accurate analyses can be made by increasing the angular dispersion and the resolving power of the X-ray spectrometer. The first is accomplished by using an analyzer crystal with

a small d-spacing (i.e., LiF or topaz), and by using higher-order refractions. The second implies the use of a narrow slit. In this case, a 0.005 in. Soller slit, and corresponding long integration times are needed to resolve the two spectral lines.

Source: R. T. Kessler of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18837)

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ANALYSIS OF BRAZE ALLOYS

A solution method of X-ray spectrometry has been found practical for analyzing the zinc content of braze alloys. Using the synthetic-standard technique described earlier, standard solutions were prepared with varying zinc and silver content, and were used to construct a calibration curve for zinc concentration. The curve was found to be linear over the range of 2.0 to 2.85 mg/ml.

Using carefully prepared constant strength test solutions, results were obtained with an accuracy of 0.01%.

Silver content also could be analyzed, but extremely long integrations times would be required.

Source: R. T. Kessler of
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