

NASA SP-5940 (03)

N 7 3 2 5 5 9 8

TECHNOLOGY UTILIZATION**CASE FILE
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Foreword

The National Aeronautics and Space Administration and Atomic Energy Commission have established a Technology Utilization Program for the 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 investment in aerospace and nuclear research and development programs.

This document is intended to provide such technical information. The compilation is arranged in two sections, one on the effects of hydrogen on a variety of metal alloys, and the other on the mechanical properties of some recently developed alloys. In section one, hydrogen at both low and high pressure is shown to have adverse effects on alloys such as ultrahigh-strength steels, irradiated steels, columbium, inconel alloys, titanium alloys, and certain stainless steels. Section two treats of the mechanical and physical properties of a wide range of alloys, their performance at elevated temperatures, and some of the processes involved in their development.

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

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

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Section 1. Effects of Hydrogen on Metal Alloys

PROPERTIES OF MATERIALS IN HIGH-PRESSURE HYDROGEN AT CRYOGENIC, ROOM, AND ELEVATED TEMPERATURES

A program was established to determine degradation of specific material properties and to enable general observations in regard to the susceptibility of a particular material to hydrogen degradation. Certain tests failed to indicate any positive degradation due to the hydrogen environment although, for the materials concerned, other tests showed appreciable degradation. An example is the fracture toughness of INCO 718 with the 1227 K (1750°F) Solution-plus-Age heat treatment. While this test indicated negligible degradation, the low and high cycle fatigue, creep-rupture, and tensile tests indicated extreme hydrogen degradation.

An A-286 material exhibited negligible degradation at 300 K (80°F) in all tests. At elevated temperature, the creep-rupture test indicated severe to extreme degradation, thus emphasizing the fact that no one test can be used as a basis for declaring a material free of hydrogen degradation. This makes mandatory a wide range of tests in a high-pressure hydrogen environment before an accurate conclusion can be made regarding the degree of hydrogen degradation.

All testing was conducted on solid specimens exposed to external gaseous pressure. Specific mechanical properties determined and the testing methods used are summarized below:

- Low Cycle Fatigue – Low cycle fatigue life was established by constant total-strain testing using smooth specimens and total-strain closed-loop testing machines.
- High Cycle Fatigue – High cycle fatigue life was established by load (stress) controlled tension (tension testing) using smooth specimens and servo-actuated, closed-loop machines.
- Fracture Toughness – Fracture toughness tests were conducted using single-edged, notched, fatigue-precracked, compact tensile specimens.
- Creep-Rupture – Creep rate and time-to-failure were determined using smooth specimens and a standard creep-rupture machine equipped with a recording extensometer.
- Tensile – Smooth and notched tensile tests were conducted on solid specimens using ASTM tensile testing techniques.

Source: J. A. Harris, Jr. and
W. C. Van Wanderham of
United Aircraft Corp.
under contract to
Marshall Space Flight Center
(MFS-21591)

Circle 1 on Reader Service Card.

STUDY FOR MINIMIZATION OF HYDROGEN EMBRITTLEMENT IN ULTRAHIGH-STRENGTH STEELS

Atomic hydrogen is capable of entering steel and many other metals and alloys; when it does, any of several undesirable phenomena can occur. If large quantities of hydrogen are introduced, there may be a general loss in ductility or, if the hydrogen accumulates in certain localized areas, internal bursts or blisters may be produced. At elevated temperatures,

hydrogen may react with and remove so much carbon from the steel that the material is no longer capable of supporting the design stresses. Under certain circumstances, hydrogen introduced into steel during its manufacture, subsequent fabrication, or in service, may result in brittle failures at applied stresses far below the yield strength or the nominal design stress

for the alloy. All of these phenomena are collectively referred to as hydrogen embrittlement. However, only the last, i.e., the catastrophic, hydrogen-induced, brittle failure of ultrahigh-strength structural steels at relatively low applied stresses is of interest in the present study. Since this phenomenon frequently occurs in materials that exhibit no appreciable loss in ductility (as measured by a conventional tensile test), it is often termed hydrogen-induced, delayed brittle failure, or hydrogen-stress cracking.

Several conditions must be satisfied for hydrogen-stress cracking of steels to occur:

- (1) The steel must be processed to a strength level above some as yet not clearly defined minimum; generally, as the strength level of the steel is increased above this minimum, the time-to-failure decreases.
- (2) The steel must be subjected to an applied tensile stress above some minimum value that is dependent on the strength level of the steel; as the strength level of the steel increases, the minimum applied stress that will result in hydrogen-stress cracking decreases.
- (3) The steel must contain hydrogen in excess of some minimum amount, and this hydrogen must be free to diffuse through the steel.

In view of these conditions, it would be expected that any condition that alters the strength, applied stress, or hydrogen content of a given material could influence its sensitivity to hydrogen-stress cracking. The trend toward higher tensile strengths, higher design stresses, and the use of materials in applications requiring prolonged exposure to high sustained loads insures that two of the conditions necessary for the occurrence of hydrogen-stress cracking will be present in steel parts intended for certain aerospace and aircraft applications. All that remains is for an adequate supply of hydrogen to be available and for this hydrogen to be free to diffuse through the steel. Thus, it appears that, for high-strength steels sensitive to hydrogen-stress cracking, the most important factors tending to promote hydrogen-stress cracking under these conditions are the hydrogen content of the material and the propensity of the material to absorb hydrogen from its environment, either during processing or in service.

The problem of hydrogen-stress cracking of ultrahigh-strength steels has become quite serious in the aerospace and aircraft industries because many of the components fabricated from these high-strength steels have to be protected from corrosion in their service environments. The preferred method of providing this protection is cadmium electroplating. However, the application of electrodeposited coatings to solve corrosion problems can make the part susceptible to failure by hydrogen-stress cracking because, frequently, hydrogen is introduced during the cleaning and electroplating operation.

Earlier studies showed that most ultrahigh-strength steels were embrittled to various degrees by virtually all of the common electroplating processes, including cadmium, chromium, zinc, tin, nickel, lead, copper, and silver. These studies also showed that the amount of hydrogen entering steel specimens during certain electroplating processes may be as great as that introduced during severe cathodic charging, and that more hydrogen sometimes was introduced during pickling or cathodic cleaning prior to electroplating than during the actual plating operation. In addition, these studies showed that the sustained-load tensile test employing notched bars was the most sensitive method for evaluating the embrittling tendencies of cleaning and electroplating processes.

The results of a literature and industrial survey of reportedly low-hydrogen-embrittling and nonhydrogen-embrittling cleaning, pickling, and electroplating processes (including cadmium, chromium, and nickel electroplating) and of various hydrogen-embrittlement relief treatments are described in: "A Review of the Literature on Cleaning, Pickling, and Electroplating Processes and Relief Treatments To Minimize Hydrogen Embrittlement of Ultrahigh-Strength Steels," by T. P. Groeneveld, E. E. Fletcher, and A. R. Elsea, October 15, 1965, Batelle Memorial Institute.

Source: T. P. Groeneveld, E. E. Fletcher,
and A. R. Elsea
of Batelle Memorial Institute
under contract to
Marshall Space Flight Center
(MFS-2455)

Circle 2 on Reader Service Card.

THE COLUMBIUM-HYDROGEN SYSTEM AND HYDROGEN EMBRITTEMENT OF COLUMBIUM

Columbium and columbium alloys have desirable properties which make them quite attractive for industrial application, especially where high heat fluxes and high temperatures are encountered. In these applications, it is usually more efficient to use a cooling fluid, such as hydrogen, which has excellent heat-transfer characteristics, than to rely on methods which employ ablative materials and porous walls. However, it is well known that columbium and columbium alloys can be severely embrittled by hydrogen, particularly at low cooling temperatures; under certain conditions, fragmentation may even occur.

A thorough investigation of the available literature was undertaken and laboratory efforts were conducted to provide a comprehensive treatise on the columbium-hydrogen system and the effects of hydrogen on the mechanical properties of columbium.

Laboratory methods were developed to charge columbium specimens uniformly with hydrogen and to measure accurately the hydrogen content by a

procedure which involved the removal of hydrogen from flowing argon at 1366 K (2000°F).

The columbium-hydrogen phase diagram that included a miscibility gap and an eutectoid transformation, was determined for two different columbium purity levels. A peritectoid transformation was postulated at the hydrogen-rich end of the diagram. The effects of hydrogen content on the ductile-to-transition temperature, deduced from slow strain-rate tensile test data, were determined for temperatures between 366 and 588 K (200° and 600°F). Brittle fracture occurred at 644 and 700 K (700° and 800°F) for specimens containing large amounts of hydrogen. The strength and ductility of columbium was found essentially to be independent of hydrogen content.

Source: R. J. Walter of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18659)

Circle 3 on Reader Service Card.

EFFECTS OF HIGH PRESSURE HYDROGEN ON METALS

Failures of large, welded hydrogen storage vessels at and below design pressures of 345×10^5 to $414 \times 10^5 \text{ N/m}^2$ (5000 to 6000 psi) have occurred with enough frequency to bring attention to the embrittlement of metals by high-pressure hydrogen at ambient temperatures. Previously, notched steel specimens tested in tension in the presence of high-pressure hydrogen exhibited a considerable decrease in fracture strength. This decrease was more pronounced in cases with sharper notches and steels of higher strength levels.

Thirty-five alloys have been investigated for their susceptibility to high-pressure hydrogen environment embrittlement at ambient temperature. In $69 \times 10^5 \text{ N/m}^2$ (10,000 psi) hydrogen, the ranking in order of decreasing embrittlement was: (1) high-strength steels and nickel-base alloys; (2) moderate- and low-strength

iron-base alloys, pure nickel, and titanium alloys; (3) nonstable AISI type 300 stainless steels, beryllium-copper, and commercially pure titanium; and, (4) aluminum alloys, pure copper, and the stable AISI type 300 stainless steels.

The degree of hydrogen environment embrittlement was found to increase with increasing pressure, while low-alloy steels were reduced considerably in notch strength by 69×10^5 and $69 \times 10^6 \text{ N/m}^2$ (1,000- and 10,000-psi) hydrogen environments.

Source: R. J. Walter and W. T. Chandler of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18612)

Circle 4 on Reader Service Card.

LOW-TEMPERATURE EMBRITTLEMENT OF Ti-6Al-4V AND INCONEL 718 BY HIGH-PRESSURE HYDROGEN

Tensile properties are reported of notched or unnotched specimens of titanium alloy Ti-6Al-4V (STA) at 294, 195, and 77 K (70°, -109°, and -320°F) under air at 1 atm or hydrogen or helium at 69×10^5 and $138 \times 10^5 \text{ N/m}^2$ (1000 and 2000 psi). Under $138 \times 10^4 \text{ N/m}^2$ (200 psi) hydrogen, the notch strength was reduced by about 45% at 294 K (70°F), 8% at 195 K (-109°F), and nil at 77 K (-320°F); unnotched specimens essentially were not embrittled at these temperatures.

Information is also available on tensile properties of notched or unnotched specimens of Inconel-718 at temperatures of 333, 294, 195, and 77 K (140°, 70°, -109°, and -320°F) under air at 1 atm or hydrogen or helium at 69×10^5 and $138 \times 10^5 \text{ N/m}^2$ (1000 and 2000 psi). Under hydrogen at $138 \times 10^5 \text{ N/m}^2$ (2000 psi) the notch strength was reduced by

about 15% at 294 and 333 K (70° and 140°F) and almost nil at 195 and 77 K (-109° and -320°F); unnotched specimens were not embrittled. This degree of embrittlement is much lower than earlier observations under $69 \times 10^5 \text{ N/m}^2$ (1000 psi) hydrogen.

The 1000-cycle fatigue strength of precracked specimens of Inconel-718 was 22% less under hydrogen at $138 \times 10^5 \text{ N/m}^2$ (2000 psi) than under the same concentration of helium.

Source: R. J. Walter and W. T. Chandler of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18753)

Circle 5 on Reader Service Card.

EFFECTS OF HYDROGEN ON ELI TITANIUM ALLOY Ti-5Al-2.5Sn

The effects are reported of hydrogen on ELI titanium alloy Ti-5Al-2.5Sn under three sets of conditions.

1. Tensile tests followed abrasion under hydrogen, and temperature cycling between 21 and either 477 or 700 K (-423° and either 400° or 800°F). The metal was abraded with titanium, Inconel-718, 440-C, Tens-50 aluminum, A-286, or iron files. These treatments lowered the tensile strength by $\leq 21\%$, and increased ductility by $\leq 38.7\%$ as measured by reduction in area. These effects are believed to be too great to reflect data scatter only. Effects of abrasion in air or helium were similar. The tensile tests revealed no evidence of embrittlement. Gas analyses and electron and light microscopy of the failed specimens showed no absorption of hydrogen, no formation of TiH_2 , and no evidence of brittle fracture.
2. When the metal was fretted on itself in flowing hydrogen at ambient temperature and pressure, a powder formed that was identified as TiH_2 . It is

proposed that the fretting between two surfaces so raised the surface temperature that enough hydrogen was absorbed to form the hydride. The same fretting in air formed TiN and TiO_2 powders.

3. When the metal was abraded with an iron file in flowing hydrogen at 477 or 700 K (400° or 800°F), TiH_2 formed on the surface. Here the ambient temperature was high enough for absorption of sufficient hydrogen for formation of the hydride phase in the metal structure.

This information may interest the aircraft industry, fabricators in titanium, and manufacturers and users of containers or handling equipment for cryogenic liquids and gases.

Source: W. E. Hensley and W. T. Chandler of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18815)

Circle 6 on Reader Service Card.

HYDROGEN EMBRITTLEMENT OF STAINLESS STEEL 301 AND INCONEL 718

Inconel 718, in the presence of ultrapure hydrogen gas, has a "threshold" pressure (for a given tension and thickness) below which a crack will propagate due to hydrogen embrittlement. A test program was established to (1) determine the maximum safe hydrogen operating pressure for cryogenic pressure vessels made of Inconel 718, and (2) provide definitive information concerning flaw growth characteristics under the most severe conditions of temperature and pressure. Flaw growth characteristics of cryogenically formed stainless steel under similar conditions were also evaluated.

Tensile tests of 26 Inconel 718 and four cryo-formed stainless steel specimens were conducted. The specimens were subjected to hydrogen pressures of from 1.73×10^6 to $6.9 \times 10^6 \text{ N/m}^2$ (250 to 1000 psi), temperatures ranging from ambient to 200 K (-100°F), and specimen stress levels of from 155×10^6 to $828 \times 10^6 \text{ N/m}^2$ (22,500 to 120,000 psi). Each specimen was deliberately flawed and then fatigued by cycling until the desired crack growth [approx. 0.152 cm (0.060-in.) deep] was obtained. To determine crack propagation, test conditions were selected to give maximum flaw growth without failure of the specimen. In an attempt to define the critical areas of flaw growth for a specific application, the test conditions for each sample were based on the performance of the previous specimen.

Using fracture mechanics techniques, the results of these tests may be used to develop the characteristics of flaw growth as a function of exposure time, stress level, gas pressure, environmental temperature and type of material. To provide maximum information for fracture mechanics analysis techniques, a wide range of hydrogen pressures and tensile stresses was used to bracket the areas where flaw growth would occur without specimen failure.

For Inconel 718 welded specimens, the extreme conditions that would permit flaw growth without failure were (1) ambient temperature, $5.2 \times 10^6 \text{ N/m}^2$ (750 psi) hydrogen and $460 \times 10^6 \text{ N/m}^2$ (67,500 psi)

for at least 20 hr, and (2) 200 K (-100°F), $5.2 \times 10^6 \text{ N/m}^2$ (750 psi) hydrogen and $470 \times 10^6 \text{ N/m}^2$ (67,500 psi) for at least 72 hrs.

The extreme conditions for the parent Inconel 718 specimen were (1) ambient temperature, $3.9 \times 10^6 \text{ N/m}^2$ (570 psi) hydrogen and $470 \times 10^6 \text{ N/m}^2$ (67,500 psi) for at least 20 hr., and (2) 200 K (-100°F), $5.5 \times 10^6 \text{ N/m}^2$ (800 psi) hydrogen and $500 \times 10^6 \text{ N/m}^2$ (72,000 psi) for at least 231 hrs.

The Stainless Steel 301 specimens were all parent metal. The maximum safe condition at ambient temperature was $6.9 \times 10^6 \text{ N/m}^2$ (1000 psi) hydrogen and $276 \times 10^6 \text{ N/m}^2$ (40,000 psi) for at least 21 hours of exposure. The first Stainless Steel 301 parent metal specimens tested failed on loading at ambient temperature, $6.9 \times 10^6 \text{ N/m}^2$ (1000 psi) hydrogen and at 690×10^6 , 414×10^6 , and $345 \times 10^6 \text{ N/m}^2$ (100,000, 60,000 and 50,000 psi) stress levels. The specimens that did not fail were "fatigue-marked" before being pulled to failure to permit measuring the crack growth under sustained load.

Conclusions of this test program showed that flaw growth in cryogenic storage system pressure vessels constructed of Inconel 718 or Stainless Steel 301 material in a hydrogen environment is a function of temperature, pressure and stress; that the welded areas of the pressure vessel (welded specimens) have a lower resistance to crack propagation than the parent metal; that the maximum operating pressure which will permit flaw growth without failure is determined by the initial flaw size and the ability to determine the largest flaw size existing in the pressure vessel; and that the extreme conditions for safe extended operation of specific cryogenic storage system pressure vessels are $3.1 \times 10^6 \text{ N/m}^2$ (450 psig) at 200 K (-100°F) after a cryo-proof test with liquid nitrogen at $13.8 \times 10^6 \text{ N/m}^2$ (2000 psig).

Source: R. Forman and R. K. Allgeier
Manned Spacecraft Center
(MSC-13557)

Circle 7 on Reader Service Card.

EFFECTS OF HYDROGEN ON METALS

A search is being made to develop a method for minimizing failures in storage tanks and related hardware caused by high-pressure hydrogen effects, the formation of hydrides in titanium, and hydrogen absorption through various metals processing techniques.

As yet no general solution has emerged, but much experience in the field and many series of tests have led to development of several rules to guide choice of materials, methods of welding, electroplating, heat treatment, and other processing.

The problem is complicated by the existence of various possible sources (and combinations of sources) of hydrogen. Large quantities of hydrogen may reduce ductility in a metal; accumulations in localized areas may cause internal bursts or blisters. In some circumstances hydrogen reacts with the metal or alloy phases to form brittle compounds resulting in brittle fractures at relatively low stress levels.

The hydrides may result from the storage of high-pressure hydrogen, imperfections in the surfaces of metals, use of incorrect weld fillers, inappropriate methods of welding, cleaning, pickling, electroplating, or heat treatment, or original choice of susceptible materials; or from the synergistic effect of two or more of these factors. Clearly some steels are more susceptible than others during fabrication and service. Alpha titanium differs from beta titanium in absorption of hydrogen; use of commercially pure filler wire for welding of Ti-6Al-4V alloy tends to formation of more hydride than does Ti-6Al-4V filler.

When high-pressure hydrogen is present, entry into the metal is possible when molecular hydrogen is dissociated into atomic hydrogen by catalytic reaction with fresh metallic surfaces. Atomic hydrogen may be formed by the localized energy released by microcracking or in slippage in the metal; it enters the lattice of the metal, and such entry is strongly influenced by temperature and lattice defects and by metals in the process of transformation or under stress. Such conditions provide the energy necessary for the endothermic process of dissolution of the hydrogen into the metal, either as interstitial solid solution or as metal-hydrogen compounds on the basis of ionic bonding.

Regarding the difference in solubility of hydrogen between alpha and beta titanium, the commercially pure alpha alloy has no stabilizing elements such as

the aluminum in Ti-6Al-4V. Aluminum belongs to a group of metals (such as Fe, Cu, Ni, and Mo) in which the hydrogen is endothermically formed and dissolved as interstitial solid solution; while titanium belongs to another group (such as Zr, Ta, and Nb) in which hydrogen occurs in the form of positively charged ions. Conditions causing migration of hydrogen and formation of titanium hydride have not been completely defined, although it is generally accepted that Ti-6Al-4V alloy has sufficient aluminum to provide adequate solubility of hydrogen, and that the small amounts of hydrides formed are inconsequential. Migration mechanisms have been postulated and studied under various influencing conditions, but the triggering circumstances are far from being defined for confident prediction of limiting compositions of alloys, or for design of weld-filler alloys to control such mechanisms.

Regardless of the basic mechanism or mechanisms of metal-hydrogen reaction, applications must be approached with extreme caution. The resistance of one alloy to damage by hydrogen under one set of conditions may not apply under another. Special care is needed where welds are used; new alloys and alloy phases may be formed in weldments that are sensitive to hydrogen; and weldments may entrap pockets or narrow bands of hydrogen concentrations that are not readily detectable and from which hydrogen may diffuse even at relatively low temperatures, so that deterioration becomes time dependent.

Every known method of electroplating produces some degree of hydrogen embrittlement of certain alloys, such as the high-strength steel alloys; chromium plating proved to be the most embrittling in a broad evaluation of methods.

Different alloys seem to differ widely in tolerance of hydrogen without failure. Distribution of hydrogen within a sample is a more important factor in hydrogen embrittlement than is average content of hydrogen.

Documentation is available from:

National Technical Information Service

Springfield, Virginia 22151

Price \$3.00

Reference: TSP69-10372

Source: C. E. Cataldo
Marshall Space Flight Center
(MFS-20364)

SUSCEPTIBILITY OF IRRADIATED STEELS TO HYDROGEN EMBRITTLEMENT

An investigation was conducted to determine whether irradiated pressure-vessel steels 4340 and 212-B could become susceptible to hydrogen embrittlement, and if these irradiated structures would then be susceptible to catastrophic failure. Knowledge of the effect of irradiation on various types of steel is extremely important for reactor safety.

The results of the investigation are contained in the report, *Hydrogen Embrittlement in Irradiated Steels*, by A. D. Rossin, NAL-7266, February 1967, Argonne National Laboratory, Argonne, Illinois. This report completely presents the very specialized area of hydrogen embrittlement of 4340 and 212-B steels. Background information, experimental procedures, testing results, and possibilities for future work are included.

Two very different steels were examined. A high-strength steel, quenched and tempered 4340, was chosen because its behavior when charged with hydrogen is well known and documented. It was irradiated, then hydrogenated and tested in a manner which had previously revealed catastrophic embrittlement.

Steel 212-B was chosen because of its use as a vessel material in a number of pressurized- and boiling-water reactors. Further, fully documented material was available. Samples of 212-B were exposed to an irradiation dose large enough to raise its ductile-brittle transition temperature well above room temperature to cause a substantial increase in its yield and tensile strength. It was then hydrogenated and tested in the manner that results in catastrophic failure in 4340.

Hydrogen-charging conditions which completely embrittled 4340 high-strength steel had negligible

effect on 212-B pressure vessel steel in tensile and delayed-failure tests. Much higher hydrogen charges reduced the notch-tensile strength slightly. Delayed failure was observed only at stresses above 90% of the notch-tensile strength of the hydrogenated 211-B. Tests on 212-B, which had been irradiated to give a 35% increase in strength and an NDT temperature shift of 94°C, showed the same relationship between delayed-failure limits and notch-tensile strength as that observed without irradiation.

Catastrophic embrittlement due to hydrogen was not observed in 212-B, even for irradiated material that had been charged to produce high hydrogen content. Therefore, catastrophic hydrogen embrittlement of a well-designed nuclear-reactor pressure vessel is not probable.

The notch-tensile strength of 4340 steel was reduced by irradiation, although the tensile strength increased. Catastrophic delayed failure still occurred, but the sensitivity to hydrogen was slightly reduced.

The following documentation may be obtained from:

National Technical Information Service
Springfield, Virginia 22151
Single document price \$6.00
(or microfiche \$0.95)

Reference:

ANL-7266 (N67-34900) Hydrogen Embrittlement
in Irradiated Steels

Source: A. D. Rossin
Metallurgy Division
Argonne National Laboratory
(ARG-10115)

Section 2. Mechanical Properties of Various Alloys

WELDABLE ALUMINUM ALLOY HAS IMPROVED MECHANICAL PROPERTIES

An attempt has been made to develop a weldable aluminum alloy with mechanical properties superior to those of commercially available weldable aluminum alloys.

This paper covers a series of new alloys based on aluminum alloy 2219-T87, which nominally contains 6.53 percent copper and 0.30 percent manganese. The alloy with the best mechanical properties in this series, designated X2021, was produced by the addition of 0.15 percent cadmium and 0.05 percent tin to the 2219-T87 alloy. The ultimate tensile strength, yield strength, percent elongation, and notch-tensile ratio of the X2021 alloy were found to be from 8 to 14 percent higher than typical values for the basic 2219-T87 alloy. The new alloy has good resistance to stress-corrosion cracking, shows unchanged strength and formability after storage at room temperature, and can be pre-aged, stretched, and aged. The recommended heat treating temperature for this alloy is 805 K (990°F) [(solidus temperature, 811 K

(1001°F)]. With respect to weldability, the new alloy appears to be as good as the 2219-T87 alloy. X2021 is well suited for use at cryogenic temperatures; all of its mechanical properties at 20 K (-423°F) are better than at room temperature.

Several aluminum alloys of the 7000 series, such as 7075 and 7178, have slightly higher ultimate tensile strengths than the new alloy, but are not as weldable. Toxic fumes of cadmium oxide are evolved when the new alloy is welded. Adequate ventilation must therefore be provided to safeguard welding operators from these fumes.

Source: R. W. Westerlund of Alcoa Research Laboratories under contract to Marshall Space Flight Center (MFS-295)

Circle 8 on Reader Service Card.

TANTALUM ALLOYS RESIST CREEP DEFORMATION AT ELEVATED TEMPERATURES

This paper reports the development of refractory metal alloys possessing high strength and good resistance to creep deformation at elevated temperatures in high vacuum environments. Other properties of the alloys are ease of fabrication, good weldability, and corrosion resistance to molten alkali metals.

Dispersion-strengthened (precipitation-hardened) tantalum-base alloys containing a dispersed second phase comprised of carbides and/or nitrides which serve to pin dislocations and thus greatly improve the resistance of the alloys to creep deformation. Strengthening of the tantalum is achieved by solid-solution strengtheners consisting of tungsten, molybdenum, and rhenium, and also by the presence of stable precipitates resulting from the interaction of the

metal additives hafnium and zirconium with nitrogen and/or carbon.

More specifically, the alloys include, in addition to tantalum, the element tungsten and some or all of the elements molybdenum, rhenium, hafnium, zirconium, carbon, and nitrogen. The composition range for these alloys in weight percent is as follows: tungsten, 7-10; zirconium, 0.25-0.6; nitrogen, 0.04-0.1; and tantalum, balance. Up to 1 percent by weight of molybdenum and/or 2-atom-percent rhenium may be substituted for the tungsten, part or all of the zirconium may be replaced with hafnium, and carbon (0.015-0.04 percent) may be substituted for nitrogen. Impurities should be maintained at a low level; the amount of oxygen must be restricted to less than 100

parts per million. Compositions within the given range should have an atom ratio of zirconium to nitrogen maintained at approximately unity for optimum resistance to creep deformation.

The new tantalum-base alloys are significantly superior to T-222, the highest strength commercially available tantalum-base alloy. Specific compositions have excellent room temperature ductility, which enables their cold rolling to sheet form. The alloys have immediate applications such as in the highly stressed rotating turbine components of advanced

space (high vacuum) power systems of the nuclear turbogenerator type. Possible nonaerospace applications for these alloys are structural materials for fluid flow components in chemical processing and nuclear reactor equipment.

Source: R. W. Buckman, Jr. of
Westinghouse Electric Corp.
under contract to
Lewis Research Center
(LEW-350)

Circle 9 on Reader Service Card.

WELDABLE HIGH STRENGTH ALUMINUM ALLOY DEVELOPED FOR CRYOGENIC SERVICE

It was desired to develop a new 7000 series wrought aluminum alloy (Al-Zn-Mg) that would have mechanical properties superior to those of standard 7000 series alloys, which are relatively low in notch toughness and weldability.

An alloy composed of 6.5 Zn, 1.8 Mg, 0.2 Mn, 0.12 Zr, and 0.10 Cu shows great promise.

Tests indicate that the use of zirconium results in improved low temperature notch toughness and weldability. The manganese and chromium serve as supplementary strengthening elements with beneficial effects on grain size and resistance to stress corrosion cracking. This alloy can be mill-fabricated to plate and sheet without difficulty. Moderate amounts of cold forming are possible prior to artificial aging. Solution heat treatment temperature is 733 K (860°F) and the alloy is fully heat treatable to a range of desired tempers. The mechanical properties (approximately at room temperature) of this alloy are:

Tensile strength	511 x 10 ⁶ N/m ²	(74,000 psi)
Yield strength	449 x 10 ⁶ N/m ²	(65,000 psi)
2-inch elongation		12%
Notch-Tensile Ratio		
Room Temperature		1.31
77 K (-320°F)		1.05
20 K (-423°F)		0.95

This alloy was originally designated M826, and an Aluminum Association registration of this alloy as X7007 has been requested. Post-weld aging has been found to improve weld ductility and strength properties. A typical treatment is 8 hours at 380 K (225°F) plus 16 hours at 422 K (300°F).

Source: Aluminum Company of America
under contract to
Marshall Space Flight Center
(MFS-737)

Circle 10 on Reader Service Card.

MATERIALS DATA HANDBOOK, INCONEL ALLOY 718

A comprehensive compilation of technical data on Inconel 718 (a commercial nickel-chromium base alloy) has been presented in a handbook prepared by the Department of Chemical Engineering and Metallurgy, Syracuse University Research Institute, under contract to Marshall Space Flight Center.

This "Materials Data Handbook, Inconel Alloy 718" includes data on the properties of the alloy at cryogenic, ambient, and elevated temperatures, and other pertinent engineering information required for the design and fabrication of components and equipment utilizing this alloy. The handbook information is arranged in twelve chapters under the following headings:

1. General Information
2. Procurement Information

3. Metallurgy
4. Production Practices
5. Manufacturing Practices
6. Space Environment Effects
7. Static Mechanical Properties
8. Dynamic and Time Dependent Properties
9. Physical Properties
10. Corrosion Resistance and Protection
11. Surface Treatments
12. Joining Techniques

Source: J. Sessler and V. Weiss of
Syracuse University Research Institute
under contract to
Marshall Space Flight Center
(MFS-2348)

Circle 11 on Reader Service Card.

MATERIALS DATA HANDBOOK, ALUMINUM ALLOY 7075

A comprehensive compilation of technical data on aluminum alloy 7075 (nominal composition: 90 Al, 5.6 Zn, 2.5 Mg, 1.6 Cu, 0.3 Cr) has been presented in a handbook prepared by the Department of Chemical Engineering and Metallurgy, Syracuse University Research Institute, under contract to Marshall Space Flight Center.

This "Materials Data Handbook, Aluminum Alloy 7075" includes data on the properties of the alloy at cryogenic, ambient, and elevated temperatures, and other pertinent engineering information required for the design and fabrication of components and equipment utilizing this alloy. The handbook information is arranged in twelve chapters under the following headings:

1. General Information
2. Procurement Information

3. Metallurgy
4. Production Practices
5. Manufacturing Practices
6. Space Environment Effects
7. Static Mechanical Properties
8. Dynamic and Time Dependent Properties
9. Physical Properties
10. Corrosion Resistance and Protection
11. Surface Treatments
12. Joining Techniques

Source: J. Sessler and V. Weiss of
Syracuse University Research Institute
under contract to
Marshall Space Flight Center
(MFS-2349)

Circle 12 on Reader Service Card.

MAGNESIUM-LITHIUM ALLOYS DEVELOPED FOR LOW TEMPERATURE USE

Three new magnesium-lithium alloys have been developed for application at cryogenic temperatures [down to 20 K (-423°F)]. These lightweight alloys have approximately double the tensile and yield strengths (at room temperature) of the LA 91A, LA 141A, and LAZ 933A magnesium-lithium alloys described in NASA SP-5068, "Properties and Current Applications of Magnesium-Lithium Alloys" (for sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402; price: 40 cents). The designations and nominal compositions of the new alloys are:

Alloy II4: Mg, 7Li, 1 Zr, 3Th, 6Zn, 5Cd, 6Ag

Alloy IA6: Mg, 9Li, 3Th, 2Zn, 4Al, 4Ag, 1Mn

Alloy ZLH 972: Mg, 7Li, 9Zn, 2Th

Alloy II4, as rolled initially at 630 K (675°F) for ingot breakdown followed by finish rolling at 505 K (450°F), or as solution heat-treated at 616 K (650°F) followed by thermal stabilizing aging at 338 K (150°F), exhibits at 293 K (68°F) tensile strengths in excess of $311 \times 10^6 \text{N/m}^2$ (45,000 psi), yield strengths of more than $242 \times 10^6 \text{N/m}^2$ (35,000 psi), and minimum ductility values of 15 percent. The strength values are increased at 4 K (-452°F), with a reduction in ductility to a minimum of 8 percent. Alloy IA6, after rolling at 400 K (260°F), followed by solution

heat treatment at 588 K (600°F), water quenching, and then aging at 366 K (200°F), exhibits similar strength properties. However, the long aging times needed to obtain the 15 percent and 8 percent ductilities at 293 and 4 K (68° and -452°F), respectively, for this alloy often result in strength degradation. Alloy ZLH 972, when rolled at 505 K (450°F), heat treated at 588 K (600°F), quenched and aged at 366 K (200°F), has satisfactory strength and ductility properties, but is sensitive to embrittlement and has a tendency toward hot shortness at the usual hot working temperatures 588 to 630 K (600° to 675°F). The alloys are easy to weld by the conventional TIG method, are readily machinable and chemically millable, and respond well to conventional metal forming operations (except that ZLH 972 is not readily extruded). The notched/unnotched tensile ratios for each of the alloys are 1.0 and 0.9 at 293 and 4 K (68° and -452°F), respectively.

Source: H. W. Leavenworth, Jr.
and F. J. Dunkerley of
American Machine and Foundry Co.
under contract to
Marshall Space Flight Center
(MFS-1541)

Circle 13 on Reader Service Card.

NICKEL-BASE ALLOY HAS IMPROVED STRESS RUPTURE PROPERTIES

This innovation concerns a superalloy material for jet aircraft turbine blades providing a substantial improvement in stress rupture life over present-day alloys.

It is a nickel base superalloy of the following weight percent composition: 0.13 C, 6.1 Cr, 2.0 Mo, 1.0 Ti, 5.4 Al, 7.5 Co, 5.8 W, 0.5 Re, 0.43 Hf, 0.13 Zr, 0.02 B, 9.0 Ta, 0.5 Cb, and the balance Ni. This alloy is capable of maintaining its strength and its creep, oxidation, and thermal fatigue resistance at high temperature.

A nickel base superalloy has been developed that is particularly adapted for use in turbine engine blades because of its excellent stress rupture properties and other physical characteristics. The alloy, designated "Alloy VI A," (composition cited above),

has attractive high temperature properties and compares favorably with all known commercially available, nickel base alloys—for example, at 1366 K (2000°F), and at an applied stress of $104 \times 10^6 \text{N/m}^2$ (15,000 psi), the average stress rupture life is 63 hours.

The alloy uses substantial amounts of solid solution strengtheners such as tantalum, tungsten, and molybdenum in proper balance to achieve strength properties without depreciating oxidation resistance. The nickel, aluminum, and titanium contents, for best results, must also be controlled. The addition of rhenium is used to improve strength and corrosive resistance of the alloy. The stress rupture life of the alloy is generally more than that of currently-used superalloys.

Alloy VI A provides a higher use temperature than the strongest present-day commercially available high strength nickel base alloys. Tensile strength and impact properties are comparable to current alloys and ductility appears adequate.

Source: H. W. Collins and R. J. Quigg of TRW, Inc. under contract to Lewis Research Center (LEW-10283)

Circle 14 on Reader Service Card.

EFFECTS OF CRYSTAL DEFECTS ON STRESS-CORROSION SUSCEPTIBILITY IN ALUMINUM ALLOY 7075

Research has been conducted to determine the effects of crystal-lattice point defects on the susceptibility of aluminum alloy 7075 to stress-corrosion cracking. Defects were introduced into specimens of the alloy by neutron irradiation, and the growth of stress-corrosion cracks was observed. Three separate stages of the cracking process were defined: pre-growth, from irradiation to crack initiation; slow growth; and catastrophic growth, ending in specimen failure. It was discovered that the duration of the pre-growth phase is closely related to t_f , the total time from irradiation to failure, but that the duration of the slow growth phase is less closely related to t_f .

Point defects were introduced into specimens of three heat-treated tempers of alloy 7075 (-T6, -T73, and a temper intermediate to these) by irradiation with fast neutrons to a fluence of 8×10^{18} nvt. Continuous ultrasonic monitoring of the irradiated specimens, simultaneously using back-reflection and pulse-echo techniques, allowed the growth of the cracks to be observed. Of the three tempers, only -T6, the least aged, showed a significant increase in stress-corrosion susceptibility. Concurrently, the maximum elongation exhibited by the -T6 specimens was

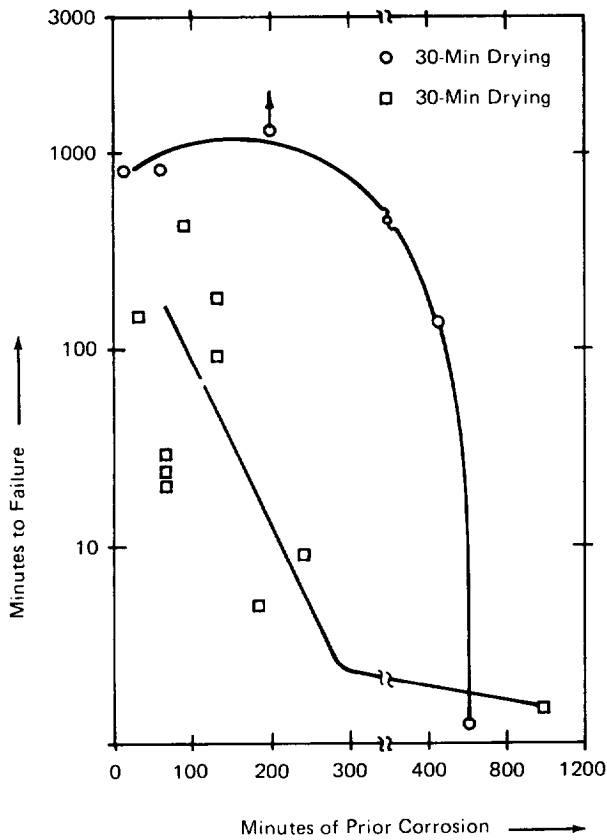
reduced by one-third. Other mechanical properties, such as yield strength and hardness, showed no significant change. In cryogenic tensile tests above 100 K, the yield strengths of both irradiated and control samples of alloy 7075-T6 were much less temperature dependent than were those of samples of the other two tempers. Also the -T6 was the only temper of the three to exhibit negative strain-rate effects in strain-rate sensitivity tests.

Information on related research involving stress corrosion in aluminum alloy 7075 may be found in NASA Tech Briefs B67-10533, Study of Stress Corrosion in Aluminum Alloys, B68-10153, Study of Crack Initiation Phenomena Associated with Stress Corrosion of Aluminum Alloys, and B70-10527, Mechanism of Stress Corrosion Cracking in Aluminum Alloy 7075.

Source: A. J. Jacobs and G. G. Bentele of North American Rockwell Corp. under contract to Marshall Space Flight Center (MFS-18794)

Circle 15 on Reader Service Card.

INCREASED RESISTANCE TO STRESS CORROSION OF ALUMINUM ALLOYS



Time to Failure Versus Time of Prior Corrosion for Specimens Dried After Prior Corrosion

A method of increasing the stress corrosion resistance of high strength aluminum alloys has been developed. The method is based on two observations:

First, surface treatment such as machining or shot peening distorts the surface grain-boundary structure. Since stress corrosion of such alloys (specifically

aluminum-7075) occurs along grain boundaries, it cannot begin before the distorted surface layer is penetrated by pure corrosion.

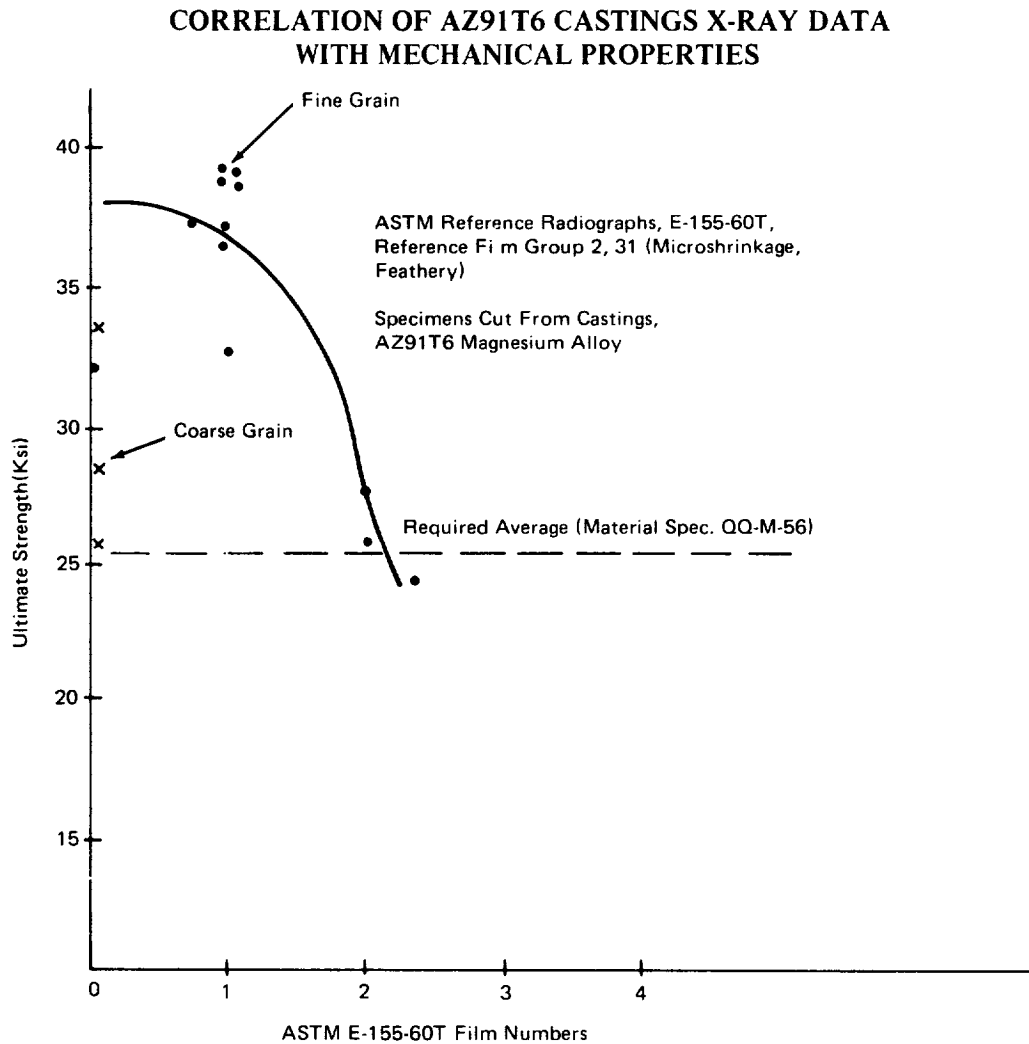
Second, both pure corrosion and stress corrosion start initially at particular sites, beginning with those most favorable. If the pure corrosion and stress corrosion are interrupted, as by removal of the corrosive environment, and the aluminum is later reexposed, the pure corrosion and stress corrosion are not re-initiated at the earlier sites but at completely new ones. These new sites are less favorable than the original sites, so that pure corrosion and stress corrosion now proceed at rates lower than their original rates; thus final failure of the aluminum occurs later than if the corrosion had not been interrupted.

Unstressed specimens of aluminum-7075-T651 were corroded for various times in a 1 M solution of NaCl buffered to pH 4.7 with sodium acetate and acetic acid; the density of the anodic current applied was 0.3 mA/cm². After corrosion, the specimens were removed and dried in a partial vacuum for either 30 min or 14 hr. When the 30-min specimens were re-exposed to corrosion under stress, the time to failure was shorter than if the corrosion had not been interrupted. When, however, the drying was very thorough (14 hr), the time to failure was greatly increased (see fig.). Thus, the anodic treatment increased the aluminum's resistance to stress corrosion.

The aircraft, chemical, and boatbuilding industries, metallurgists, oceanographers, and designers of high-strength aluminum structures may be interested.

Source: S. B. Brummer and F. H. Cocks of Tyco Laboratories, Inc. under contract to Marshall Space Flight Center (MFS-20788)

Circle 16 on Reader Service Card.



Discontinuities, generally referred to as “feathery” and “sponge” microshrinkage, may be present in magnesium castings. These discontinuities were detected by radiographic inspection and related to the mechanical properties of the casting. Radiographs of AZ91T6 magnesium alloy castings showing the type and degree of microshrinkage were used to generate graphs correlating measured mechanical properties with the severity of the microshrinkage.

An X-ray inspection of an AZ91T6 magnesium alloy sand casting revealed various degrees of “feathery” and “sponge” microshrinkage corresponding to ASTM E-155-60T film grades. Specimens were re-

moved and tested for ultimate strength, yield strength, and per cent elongation. The data were used to construct graphs correlating these properties to the severity of the microshrinkage. These graphs, such as that shown in the figure, can be used to predict the mechanical properties of AZ91T6 magnesium alloy castings.

Source: E. S. Scherba of
North American Rockwell Corp.
under contract to
Manned Spacecraft Center
(MSC-17406)

Circle 17 on Reader Service Card.

FATIGUE PROPERTIES OF Ti-5Al-2.5Sn ELI IN SMOOTH, SHOT PEENED, AND NOTCHED SURFACE CONDITIONS AT 294 AND 20 K (70° AND -423°F)

These properties of forged titanium alloy plus the effect of variations in microstructure have been investigated in order to provide design information on fatigue life of turbopump components.

A sample forging of the alloy was obtained from the same billet as the material of the components of the turbopump. The raw material was forged into a 22.47 cm (8.85-in.) diameter by 9.72 cm (3.83-in.) high cylindrical shape and vacuum annealed to 1033 ± 14 K ($1400^\circ \pm 25^\circ$ F) for four hours. Both tensile [(TR-250) and fatigue (FF-155) 0.20 cm (0.080-in.) thick] specimens were removed from the forging. The figure shows the sectioning plan for the test specimens.

The fatigue specimens were carefully prepared so as not to contaminate the surface. They were processed by wet hand grinding with 320 grit, ultrasonic cleaning in acetone, and stress relieving. The stress relieving was done in a tube furnace at 894 ± 6 K ($1150^\circ \pm 10^\circ$ F) for 60 ± 5 minutes in an argon atmosphere. The specimens were cooled in the cold zone of the furnace under an argon atmosphere to room temperature. The tensile properties of the forging were determined at both 294 and 20 K (70° and -423°F). These tensile values were used to set the starting points in the fatigue testing. The modulus of elasticity used for each test was 110×10^9 N/m² (16×10^6 psi) for room temperature 294 K (70°F), and 120×10^9 N/m² for 20 K (17×10^6 psi for -423°F).

The fatigue specimens were tested under three different surface conditions, smooth, shot peened, and notched. All specimens had a 32 RMS or better finish before the surface conditions were changed for testing. The smooth specimens retained the 32 RMS or better finish and were tested in that unaltered condition. The shot peened specimens were peened, using glass beads 0.023 to 0.041 cm (0.009 in. to 0.016 in.) in diameter. The intensity was 0.004A with eight passes on each side. The notched specimens were notched with a scribe in the constant stress area of the specimen. The notch was 0.0013 cm (0.0005 in.) deep and 0.0051 cm (0.002 in.) wide, and the notch was placed directly opposite the strain gage. The room temperature fatigue tests were set up with strain gages and a strain indicator read-out box. All of the three conditions are plotted on the same chart for comparison.

In the cryogenic fatigue testing, the specimen set up was done at room temperature with a strain gage and a strain indicator read-out box. A Type Q plug-in unit and oscilloscope were used to check the strain levels during testing. Allowances were made and set up for contraction during chilldown. The Ti-5Al-2.5-Sn ELI exhibited the typical-shape fatigue curve for titanium alloys, with the curved portion at high stress levels and a knee and flat portion at the lower stress levels. The effects of the two surface conditions imposed on the fatigue specimens were significant on the position of the curve. The smooth curve at both 294 and 20 K (70°F and -423°F) was between the shot peened and the notched curves.

Shot peening was beneficial to the endurance limit of the material. At 294 K (70°F) shot peening raised the endurance limit from 450×10^6 N/m² (66,000 psi) (smooth condition) to 510×10^6 N/m² (74,000 psi) (shot peened). At 20 K (-423°F) shot peening increased the endurance limit from 542×10^6 N/m² (78,500 psi) (smooth) to 597×10^6 N/m² (86,500 psi) (shot peened).

The notching of the fatigue specimens was significantly detrimental to the fatigue properties. At 294 K (70°F) the notched specimen had an endurance limit of 345×10^6 N/m² (50,000 psi) as compared to 455×10^6 N/m² (66,000 psi) for the smooth specimens. At 20 K (-423°F) the notching of the specimens reduced the fatigue limit from 542×10^6 N/m² (78,500 psi) (smooth) to 428×10^6 N/m² (62,000 psi) (notched.)

These results indicated that shot peening did improve the fatigue limit, however, the effect of notching was of a much greater magnitude. Shot peening increased the fatigue limit only 13.8% at 294 K (70°F) while notching decreased the fatigue limit by 23.1%. At 20 K (-423°F) almost the same degree of change was observed. Shot peening increased the fatigue limit by 10.1% and notching reduced the limit by 21.5%.

Source: P. T. Jaracewicz and E. F. Green of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-19019)

Circle 18 on Reader Service Card.

LOW TEMPERATURE MECHANICAL PROPERTIES OF 17-4PH WITH MODIFICATION OF HEAT-TREAT CONDITION H1150



17-4PH is a corrosion-resistant alloy that can be precipitation-hardened to a wide range [931 to 1484×10^6 N/m² (135,000 to 215,000 psi)] of tensile strengths while exhibiting weldability, machinability, and cost below conventional high-strength alloys used for low-temperature applications. Like most body-centered cubic alloys, toughness is reduced at low temperatures.

In this evaluation, two heat-treat conditions were studied, condition H1150 (a standard) and condition H1150M, a modification of the standard. Initial feasibility testing was done on a 1.27 cm (1/2-in.) dia. bar, and additional testing used a small billet to determine if the material would be suitable for a LOX (Liquid Oxygen) valve housing. In addition, a large forging was tested to determine the properties which might be obtained in large sections.

The material 17-4PH in condition H1150M has sufficient notch toughness for most applications at

temperatures down to 78 K (-320°F). It should not be used at lower temperatures without special design considerations to allow for reduced notch toughness. A typical microstructure of condition H1150M is shown in the figure. 17-4PH in condition H1150M should be considered for LOX applications where cost reduction is an important consideration. Although not as strong as Inconel X-750 or Inconel 718, it is much stronger than 347 CRES [typical yield strength of 690×10^6 versus 240×10^6 N/m² (100KSI versus 35KSI)] The machinability of 17-4PH condition H1150M is excellent and far superior to that of 347 CRES or the Inconels.

Source: C. O. Malin and E. F. Green of
North American Rockwell Corp.
under contract to
Marshall Space Flight Center
(MFS-18990)

Circle 19 on Reader Service Card.

COMPARISON OF WROUGHT AND CAST MECHANICAL PROPERTIES OF ANNEALED 21-6-9 ALLOY FROM 20 K (-423°F) TO 922 K (1200°F)

This paper is involved with an investigation into tentative minimum mechanical properties of investment-cast alloy 21-6-9. These tentative minimum mechanical properties are sought for applications within a temperature range from 20 to 922 K (-423° to 1200°F).

In this investigation, the chemical composition of wrought and cast alloy 21-6-9 test materials was determined by quantitative spectrographic and wet-chemical analysis. The carbon content was determined by the Conductometric method and nitrogen content was measured by the Kjeldahl method. Cast-

to-size tensile specimens were annealed at 1338 K (1950°F) for two hours and rapid-air cooled to 644 K (700°F). Three specimens were tested at 20, 78, and 200 K (-423°, -320°, and -100°F), room temperature, 478, 700, and 922 K (400°, 800°, and 1200°F). Tentative minimum mechanical properties for investment-cast alloy 21-6-9 were established from the average of these test results. The mechanical prop-

erties of investment-cast alloy 21-6-9 proved slightly below those of the wrought material.

Source: E. F. Cook and E. F. Green of North American Rockwell Corp. under contract to Marshall Space Flight Center (MFS-18936)

Circle 20 on Reader Service Card.

HIGH-TEMPERATURE NICKEL-BRAZING ALLOY

A brazing alloy for nickel, with a brazing-temperature range from 1188 to 1211 K (1680° to 1720° F), has been developed and used for brazing of nickel-clad silver electrical conductors which operate at temperatures to 922 K (1200°F). The alloy is composed of 77.9% gold and 17.1% nickel, with 5% indium added to depress the melting point.

The alloy was developed for brazing together the ends of nickel-clad silver stator coils in an electromagnetic boiler-feed pump which operates with a hot-spot temperature of 922 K (1200°F). This application required that the brazed joint have both adequate strength and stability at the operating temperature, and electrical conductivity comparable to that of the conductors. The operating environment prohibited the presence of silver either in the brazed joint or on open surfaces within the pump. Therefore the brazing alloy could not contain silver, and the brazing temperature was kept below the melting point of the conductive silver cores to prevent alloying with the braze metal. To prevent corrosion by residual flux, no flux was used.

A review of brazing alloys and techniques indicated need for a gold-based brazing alloy and that brazing

must be done in an inert-gas atmosphere. Available gold-nickel alloys met the requirements except for their high melting points. A review of additives for depression of melting points resulted in the selection of indium. Indium has good solubility with both gold and nickel, and the liquidus temperatures of the gold-indium and nickel-indium binary systems can be substantially reduced by small increases in the indium content. The low content of indium in the selected brazing alloy minimized the effect of indium's high vapor pressure.

This brazing alloy may be used in high-temperature electrical systems in lieu of conventional gold-nickel, gold-copper, or silver brazing alloys; it has low electrical resistivity, does not require a flux, and is less corrosive than other gold-nickel and gold-copper alloys.

Source: S. R. Thompson and A. H. Powell of General Electric Company under contract to Lewis Research Center (LEW-10928)

No further documentation is available.

TANTALUM ALLOY HAS SUPERIOR CREEP RESISTANCE AND GOOD WORKABILITY

A development program on refractory metal alloys produced a significant advance in tantalum technology—ASTAR-811C (Ta-8W-1Re-0.7Hf-0.025C), a carbide-strengthened tantalum alloy with better creep resistance than commercially available tantalum alloys and good fabricating and welding characteristics.

The ductility and fabricating characteristics of the new alloy are similar to Ta-8W-2Hf, a commercially available alloy, yet the creep strength of the new alloy at 1589 K (2400°F) is approximately double that of the commercial alloy.

The ASTAR-811C composition was scaled-up to 10.2 cm (4 in.) diameter ingots, which were then processed to 0.102 cm-(0.040 in.)-thick sheets for subsequent evaluation. Short-term tensile properties, recrystallization, grain-growth behavior, weldability, and creep properties were determined. The results show that ASTAR-811C has useful strength to approximately 1922 K (3000°F), and can be scaled up to larger ingots.

The following documentation may be obtained from:

National Technical Information Service
Springfield, Virginia 22151
Single document price \$3.00
(or microfiche \$0.95)

Reference:

NASA-CR-1641 (N71-20391) Precipitation-Strengthened Tantalum Base Alloy ASTAR-811C

Title to this invention has been waived under the provisions of the National Aeronautics and Space Act [42 U.S.C. 2457(f)], to the Westinghouse Electrical Corp., Pittsburgh, Pennsylvania 15236.

Source: R. W. Buckman, Jr., of
Westinghouse Electric Corp.
under contract to
Lewis Research Center
(LEW-11130)

TUNGSTEN FIBER-REINFORCED COPPER COMPOSITES FORM HIGH STRENGTH ELECTRICAL CONDUCTORS

Investigations to determine the tensile properties of composites utilize a model system, tungsten fibers in a copper matrix, in which both components are mutually insoluble. The results of these investigations show that the ultimate tensile strength, yield strength, and modulus of elasticity of the composites are proportional to fiber content.

While many applications exist for the use of composite materials as structural members, of particular interest is their adaptability to high strength electrical conductors. For such an application, the electrical resistivity and conductivity of the composite materials must be known. Since the tungsten fibers and copper are insoluble in each other, the electrical properties would be expected to follow the example of binary alloys such as Pb-Sn and Zn-Cd. These alloys are mixtures of two phases, and the conductivities, not the resistivities, follow a linear relation with composition expressed in volume percent. Since copper and tungsten act as a mixture in a composite, the conductivity of tungsten fiber-reinforced copper com-

posites should also be a linear function of fiber volume percent.

This investigation determines the electrical resistivity and conductivity of tungsten fiber-reinforced copper composites over a wide range of fiber contents for composites containing both continuous and discontinuous fibers and relates the results to fiber content.

The following documentation may be obtained from:

National Technical Information Service
Springfield, Virginia 22151
Single document price \$3.00
(or microfiche \$0.95)

Reference:

NASA-TN-D-3590 (N66-33983) Electrical Resistivity and Conductivity of Tungsten-Fiber-Reinforced Copper Composites.

Source: D. L. McDanel and
R. A. Signorelli
Lewis Research Center
(LEW-338)

ALLOY REINFORCEMENT PRODUCES THERMAL-SHOCK RESISTANT CERAMICS

A need exists for material systems highly resistant to oxidizing environments at temperatures above 1366 K (2000°F). Many materials, such as the refractory metals, have adequately high melting temperatures and mechanical strength, but deteriorate rapidly due to oxidation at high temperatures. Much effort has been spent in developing oxidation resistant coatings for refractory materials. The very stable oxides, such as zirconia and thoria, which have melting temperatures of 2866 K (4700°F) or greater, are ideal materials for resisting oxidation at very high temperatures. However, the oxides are characteristically brittle and prone to cracking when exposed to thermal shock. Attempts have been made to develop composite materials using oxides reinforced with wires or screens of refractory metals such as tungsten or molybdenum. However, use of such composites has been limited by: (1) high temperature recrystallization of the tungsten and molybdenum reinforcing materials during processing, with consequent major loss of strength and ductility; (2) oxidation of the refractory metal reinforcement causing its deterioration; and (3) separation of the matrix and reinforcement materials due to difference in thermal coefficients of expansion.

In this method, a composite material is made by dispersing short tungsten-rhenium fibers randomly throughout zirconium oxide. The high melting ceramic resists oxidation and the composite material is also thermal stress resistant.

A tungsten-rhenium alloy, generally tungsten-3% rhenium in wire or screen form is used as the material for reinforcement of the oxide matrix materials. The advantages of the tungsten-rhenium materials are:

(1) tungsten-rhenium, when recrystallized during processing as part of a metal-reinforced oxide composite; (2) since the tungsten-rhenium can be recrystallized without serious loss of strength or ductility, the composite materials can be formed by conventional hot pressing and high temperature sintering. Such techniques have proven advantageous for developing optimum densification, bonding, and strength in the oxide matrix. The tungsten-rhenium alloy has a greater thermal coefficient of expansion than that of unalloyed tungsten. This is desirable for a more favorable match with the matrix oxide materials, such as thoria and zirconia. The tungsten-rhenium alloy has greater oxidation resistance than either unalloyed tungsten or molybdenum. Laboratory data for the tungsten-3% rhenium alloy show an improvement of 65% in its oxidation resistance above 1366 K (2000°F) as opposed to that of unalloyed tungsten. This is advantageous in the event of exposure of the reinforcement to an oxidizing environment.

The major use of this development is in the fabrication of thermal-shock resistant oxides by the use of wire reinforcements that do not deteriorate, either by the processing method or by subsequent environment.

Title to this invention has been waived under the provisions of the National Aeronautics and Space Act [42 U.S.C. 2457 (f)], to TRW Inc., 23555 Euclid Avenue, Cleveland, Ohio 44117.

Source: D. N. Crump of
TRW Inc.
under contract to
Lewis Research Center
(LEW-10376)

STUDY OF DUCTILITY LIMITATIONS OF ALUMINUM-SILICON ALLOYS

The relation between microstructure and mechanical properties of cast 356 type aluminum alloys was studied to determine the cause of the variations in properties resulting from differences in solidification rate. It was found that variations in strength are a consequence of variations in ductility, and that ductility is inversely proportional to the dendrite cell size.

There are several ways in which the information gained in this study may be applied to improving the properties of weldments and castings. The most obvious is to reduce the dendrite cell size. In this study, cell-size was controlled by cooling rate. This method cannot always be applied for a variety of reasons, and some other way would be desirable. It is not clear that alloying additions used as grain

refiners would perform this function, since cell size and grain size may not be directly related. For example, in this study, practically no difference in grain size was noted for the A356-T6 for the range of solidification times used. On the other hand, the grain size is certainly the upper limit to the dendrite cell size.

The effect of silicon particle size is not clearly defined. Decreasing the size would decrease the amount of strain required for their fracture, but many more fractures would be required to effect the same decrement of effective cross-sectional areas. A change in distribution of the silicon particles to the interior of the cells would be quite effective since the differential strain of the matrix across the particle would be very small. Decreasing the amount of silicon as particles, either by decreasing the silicon content or by increasing the matrix solubility for silicon, should also be helpful.

As a consequence of studying in detail the interrelation of microstructure and fracture mode of 356 type alloys the following conclusions have been reached:

- (a) Fracture occurs almost exclusively along dendrite cell boundaries, and initiates in the silicon particles present in those boundaries.

- (b) The amount of elongation is inversely proportional to the diameter of the dendrite cells for all sizes smaller than some minimum value.
 (c) Yield strength is not a function of dendrite cell size.
 (d) Ultimate strength is controlled by the dendrite cell limitation on elongation.
 (e) The maximum cell for enhanced elongation is a functioning of the work-hardening rate of the matrix.

The following documentation may be obtained from:
 National Technical Information Service
 Springfield, Virginia 22151
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Reference:

NASA CR-78597 (N66-38791) Ductility Limitations of Aluminum-Silicon Alloys
 Source: S. F. Frederick and W. A. Bailey of
 McDonnell Douglas Corp.
 under contract to
 Marshall Space Flight Center
 (MFS-12524)

TUNGSTEN ALLOY HAS HIGH STRENGTH AT ELEVATED TEMPERATURES

It was desired to develop refractory alloys having high strength at temperatures above 1644 K (2500°F).

A tungsten-hafnium-carbon alloy exhibits tensile strengths of $609 \times 10^6 \text{ N/m}^2$ (88,200 psi) at 1922 K (3000°F) and $431 \times 10^6 \text{ N/m}^2$ (62,500 psi) at 2200 K (3500°F).

On the basis of approximately four years of study of solid-solution and carbide-strengthened, arc-melted tungsten alloys, efforts were concentrated on the tungsten-hafnium-carbon system. The strongest alloy, tungsten-0.20 atom percent hafnium-0.26 atom percent carbon, has tensile strengths of 609×10^6 and $431 \times 10^6 \text{ N/m}^2$ (88,200 and 62,500 psi) in the worked condition at 1922 and 2200 K (3000° and 3500°F), respectively.

In the earlier work, it was shown that (1) the tensile and creep strengths of arc-melted tungsten at 1644 to 2200 K (2500° to 3500°F) were increased

in decreasing order of effectiveness by additions of hafnium, tantalum, columbium, and rhenium, and (2) additions of carbon to a nominally tungsten-1 atom percent columbium alloy resulted in a further increase in strength over that of the tungsten-columbium binary alloy, due to stabilization of the cold-worked structure by carbide precipitates. The present study was aimed at exploring the tungsten-hafnium system in more detail and examining the properties of several ternary alloys containing the above mentioned elements. It was found that the effectiveness of solid-solution strengthening could be correlated with the difference in atom 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. The differences in strength

of the various carbide-strengthened alloys could be correlated with the carbide particle size, where the finest particle size (most strengthening) was observed in the tungsten-hafnium-carbon alloys.

Additional studies are being made to evaluate the effects of hafnium-carbon ratio, total alloying content, and processing variables. Possible industrial applications for these alloys would include electrical components such as switches and spark plugs, die materials for diecasting steels, and heating elements.

The following documentation may be obtained from:

National Technical Information Service
Springfield, Virginia 22151
Single document price \$6.00
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Reference:

NASA-TN-D-3248 (N66-16551) Mechanical Properties of Solid-Solution and Carbide-strengthened Arc-Melted Tungsten Alloys. NASA-TM- X-52131 (N65-34284) Solid Solution and Carbide-Strengthened Arc-Melted Tungsten Alloys.

Source: Lewis Research Center
(LEW-336)

IMPROVED HIGH-TEMPERATURE-STRENGTH NICKEL-BASE SUPERALLOY

A high strength nickel-base superalloy tailored for the 1366-1477 K (2,000-2,200°F) temperature range has recently been developed. The alloy has high tensile strength over the entire temperature range and its strength of $138 \times 10^6 \text{ N/m}^2$ (20,000 psi at 2,200°F) is approximately double the strength of the strongest available cast nickel-base alloys. The alloy possesses very high impact strength. The alloy is not subject to the formation of embrittling phases upon long-time exposure at intermediate temperatures. Its room temperature, unnotched Charpy impact resistance is 183 J (135 ft.-lbs.) after 1,000 hours exposure to a 1144 K (1,600°F) temperature.

This alloy which is based on the nickel-aluminum-tungsten system is designated WAX-20. It has the following nominal composition in weight percent: 17-20 tungsten, 6-7 aluminum, 1.4-1.6 zirconium, 0.10-0.20 carbon, balance nickel. Its incipient melting point is 1575 K (2,375°F) which is approximately 338 K (150°F) higher than that of currently

available cast nickel-base alloys. Also, the alloy is amenable to controlled solidification techniques and in the directionally solidified form exhibits substantially improved tensile and stress rupture properties over the random polycrystalline form.

Although a cast material, the alloy also has reasonable workability potential. Because of this combination of properties, the superalloy has application to such things as stator vanes for advanced gas turbine engines where temperature cycling and long time high temperature operation are required.

Documentation is available from:

National Technical Information Service
Springfield, Virginia 22151
Price \$3.00

Reference: TSP69-10352

Source: W. J. Waters and J. C. Freche
Lewis Research Center
(LEW-10874)

THERMAL TREATMENT AND MECHANICAL PROPERTIES OF ALUMINUM-2021

Mechanical properties, after various thermal treatments, are reported (see documentation) for sheet and plate of copper-rich, high-strength, heat-treatable aluminum-2021 (refs. 1-4). The alloy shows great promise for lightweight structures of great strength; in many applications it may replace alloys 2014 and 2219 (see Table). In strength and cryogenic toughness it is equivalent to aluminum-2014-T6, but its weldability and resistance to stress corrosion are better. In weldability it resembles aluminum-2219, but has greater weld strength.

Alloy, Temper	Tensile Strength, $690 \times 10^3 \text{ N/m}^2$ (100 lb/in ²)		Elongation, %
	Ultimate	Yield	
	294 K (70° F)		
2219-T87	69	57	10
2014-T651	70	60	13
2021-T81	73	63	9
	78 K (-320° F)		
2219-T87	83 ^a	61	15
2014-T651	80	53	28
2021-T81	91	75	10
	478 K (400° F)		
2219-T87	34	25	20
2014-T651	16	13	35
2021-T81	32	28	23

^aOriginally T81

Tensile Properties of Three Aluminum Alloys at
Three Temperatures

Tensile tests were run between 478 and 21 K (400° and -423° F) on samples of 0.16 cm (0.063-in.) sheet and 1.27 cm (0.50-in.) plate either as supplied (tempers 0 and T81) or after various further thermal treatments that might be applied by a fabricator. The results are reported graphically and in tabular form.

The material is so quench sensitive (especially the plate) and so responsive to thermal and mechanical conditions of processing and handling that procedures must be closely specified for consistent

production of strength and other properties. The quench rate is expected to affect its corrosion behavior, the supplier's recommendation of "rapid quenching in cold water" appears to be sound. An earlier suspicion is confirmed that cold working, introduced by flattening of the warpage after quenching, may adversely affect elongation.

Artificial aging from 16 to 24 hr at $436 \pm 5.6 \text{ K}$ ($325^\circ \pm 10^\circ \text{ F}$) produces little variation in strength of either sheet or plate. Sheet is generally higher than plate in both ultimate tensile strength and elongation; in yield strength the two gages may be comparable. Although strengths may be relatively unaffected, corrosion behavior may be readily affected by variations in aging. It is generally recognized that overaging improves the corrosion behavior of aluminum alloys; thus the recommended thermal treatment is usually a compromise for a useful combination of strengths, corrosion behavior, and other desired characteristics.

More comprehensive testing, covering all aspects of fabrication, is required for establishment of realistic data governing all potential uses of the material.

References:

Aluminum Company of America, Inc.: Aluminum Alloy 2021. Alcoa Green Letter, April 1968.

Anon.: Alloy 2021: Another Step Ahead? Iron Age, June 27, 1968, p. 72.

Anon.: Alcoa Develops Alloy for Rocket Structure. Metalworking News, Dec. 23, 1968, p. 10.

Anon.: Two New Cryogenic Alloys Look Promising in Tests. Metal Progress, April 1969, pp. 7-8.

The following documentation may be obtained from:

National Technical Information Center

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Single document price \$6.00

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Reference:

NASA-TM-X-53847 (N69-34671), Thermal Treating Studies on New Aluminum Alloy 2021

Source: M. W. Brennecke
Marshall Space Flight Center
(MFS-20559)

LOW-COST TUNGSTEN-RHENIUM ALLOYS

Applications of the refractory metal tungsten have been limited because of its very low ductility at room temperature. Although binary alloys of tungsten containing approximately 22 to 39 percent by weight of the scarce refractory metal rhenium are considerably more ductile than unalloyed tungsten, the high cost of rhenium has restricted the use of these alloys.

Further research has now shown that tungsten-rhenium alloys with a substantially more dilute rhenium content, 1.0 to 9.1 percent by weight, have ductilities and other mechanical properties which compare favorably with the tungsten-rhenium alloys having much higher concentrations of the costly rhenium. The considerably lower cost of the dilute tungsten-rhenium alloys should make them attractive for a wider range of applications than was practicable with unalloyed tungsten or the more concentrated tungsten-rhenium alloys.

Tungsten alloys containing 1.0 to 9.1 weight-percent rhenium were prepared by electron-beam and arc melting, respectively. These were fabricated into sheet and/or rod and subjected to evaluation tests. A commercial arc-melted 26-percent-rhenium alloy, an electron-beam melted 24-percent-rhenium alloy, and unalloyed tungsten were also evaluated for comparison.

The dilute tungsten-rhenium alloys were significantly more ductile than unalloyed tungsten fabricated in a similar manner. Ductile-brittle bend transition temperatures of 213 and 200 K (-75° and -100° F) were observed for worked sheet of the electron-beam melted alloys with 1.9 and 9.1 percent rhenium, respectively. The dilute arc-melted alloys were slightly less ductile than the electron-beam melted alloys, and room temperature ductility was observed only with the 1.0 percent rhenium alloy. These compared with transition temperatures of 375 and 386 K (215° and 235° F) for worked sheet of un-

alloyed arc-and electron-beam melted tungsten, respectively. Transition temperatures for the arc-melted 26-percent-rhenium alloy and the electron-beam melted 24-percent-rhenium alloy were 172 and approximately 83 K (-150° and approximately -310° F), respectively.

Annealing at 2255 K (3600° F) recrystallized all the alloys and significantly increased the bend transition temperatures. Minima in transition temperature of 505 and 477 K (450° and 400° F) occurred at 2 percent rhenium in the series of electron-beam melted alloys and at approximately 4 percent rhenium in the arc-melted alloys, respectively, compared with 605 to 627 K (630° to 670° F) for unalloyed tungsten. The 24- and 26-percent-rhenium alloys, with transition temperatures of 463 and 450 K (375° and 350° F), respectively, were slightly more ductile than the best dilute alloys after this anneal.

At 1644 to 2200 K (2500° to 3500° F), the short time tensile strength increased with increasing rhenium up to 9.1 percent rhenium.

The creep strength at 2200 K (3500° F) increased with increasing rhenium content up to 5 to 7 percent rhenium. The 24- and 26-percent-rhenium alloys were weaker than the dilute alloys and had about the same creep strength as unalloyed tungsten.

The following documentation may be obtained from:
National Technical Information Service
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Reference:

NASA-TN-D-3483 (N66-37031) Mechanical Properties of Dilute Tungsten-Rhenium Alloys.

Source: W. D. Klopp, W. R. Witzke,
and P. L. Raffo
Lewis Research Center
(LEW-332)

DIRECTIONALLY SOLIDIFIED SUPERALLOY

Metallurgists and industries using metal alloys will be interested in a cast directionally solidified cobalt-tungsten superalloy with improved stress rupture characteristics.

Screened, arc-melted, cast binary alloys were alloyed with selected modifying elements and investment cast into stress rupture test bar clusters. A resulting composition that showed good behavior and properties was Co-15W. Appropriate additions were made to this base alloy using microprobe analyses and a fractional factorial experiment to aid in the selection of alloying elements.

The composition of the resulting superalloy was 85% Co-15W base, 10% Cr, 2% Zr, 2% Ti, and 1% C by weight. This yielded an approximate overall composition of 72.3% Co, 12.7% W, 10% Cr, 2% Zr, 2% Ti, and 1% C by weight.

Stress rupture cast bars of this superalloy with a diameter of 0.63 cm (0.250 inch) and a gage length of 3.17 cm (1.25 inch) were cast conventionally and unidirectionally on a water-cooled, copper chill block in a vacuum after vacuum melting. Stress

rupture tests were carried out. The stress rupture properties of this modified Co-15W alloy in both the conventionally and unidirectionally cast form compared favorably with those of similar types of high quality cast superalloys.

The following documentation may be obtained from:
National Technical Information Services
Springfield, Virginia 22151
Single document price \$6.00
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Reference:

NASA CR-89651 (N67-40235), Dependence of Stress Rupture and Superplasticity on Structure in Co-W Alloys NASA-CR-89650 (N67-39657), Effects of Ti, Zr, and C on Structure and Properties of a Co-35W-3Cr-0.1 B Alloy

Source: J. F. Wallace of
Case Western Reserve University
under contract to
NASA Headquarters
(HQN-10522)

OXIDATION RESISTANT IRON AND NICKEL ALLOYS FOR HIGH TEMPERATURE USE

Iron-base alloys have been developed which have excellent oxidation resistance for extended periods of time at temperatures up to 1533 K (2300°F), and also retain their ductility after exposure. The composition of these alloys, in weight percent, is essentially iron, 25 chromium, 4 aluminum, with small additions of yttrium and tantalum. The addition of yttrium in 0.08 to 0.27 weight percent provided excellent oxidation resistance during exposure to air at 1533 K (2300°F) for 800 hours. Typical results were 2 mils oxide penetration on either side of a 10-mil thick foil and less than 3.5 mg/cm² gain in weight. The addition of tantalum or hafnium as secondary modifier elements provided improved room temperature ductility (after exposure) and had no significant effect on oxidation resistance. One such alloy, composed of iron, 25 chromium, 4 aluminum, 0.08 yttrium and 0.5 tantalum, remained ductile after 800 hours exposure to air at 1533 K (2300°F) including 50 cycles to room temperature. The same iron-chromium-aluminum alloys with additions of

thorium in the same atomic percentages as for yttrium also exhibited good oxidation resistance.

Nickel-base alloys were also investigated in the same program. Alloys composed essentially of nickel, 20 chromium, 5 aluminum, with small additions of yttrium or thorium exhibited considerable oxidation resistance. The best performing alloy of those tested was composed of nickel, 20 chromium, 5 aluminum, and 0.3 thorium. A 10-mil thick foil had a weight gain of 10 mg/cm² in 540 hours, and retained its ductility, but was oxidized internally throughout the foil thickness.

These alloys can be used in applications where inexpensive highly oxidation-resistant materials are needed but high strength materials are not required. They can also be used in applications above the present operating temperatures of the super-alloys [now limited to 1366 to 1477 K (2000° to 2200°F) with special coatings,] and thoria-dispersed nickel [now limited by oxidation to 1366 to 1477 K (2000° to 2200°F)], or they may be used as cladding for these

materials. The observed beneficial effects provided by the additions of yttrium, tantalum (or hafnium), and thorium might be applied to improve conventional iron-chromium-aluminum alloys such as those used in electrical resistance elements.

The following documentation may be obtained from:

National Technical Information Service
Springfield, Virginia 22151
Single document price \$6.00
(or microfiche \$0.95)

Reference:

NASA-CR-72522 (N69-27775), Development of
Ductile Claddings for Dispersion Strengthened
Nickel-Base Alloys

H. L. Wheaton of
IIT Research Institute
under contract to
Lewis Research Center
(LEW-10936)

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