

AFML-TR-77-27



INVESTIGATION OF PROCESSING MICROSTRUCTURES AND PROPERTIES OF OXIDE DISPERSION STRENGTHENED COBALT ALLOY EXTRUDED SHAPES

CABOT CORPORATION STELLITE DIVISION KOKOMO, INDIANA



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Unclassified SECURITY CLASSIFICATION OF THIS PAGE (When Date Entered) READ INSTRUCTIONS BEFORE COMPLETING FORM REPORT DOCUMENTATION PAGE 19 REPORT NUMBER 2. GOVT ACCESSION NO. 3 RECIPIENT'S CATALOG NUMBER 8 9 AFML-17-27 ITLE (and Final Technical Kepet. INVESTIGATION OF PROCESSING MICROSTRUCTURES AND 6 PROPERTIES OF OXIDE DISPERSION STRENGTHENED 1 Sep 74 - 31 Dec 76 6. PERFORMING ORG REPORT NU COBALT ALLOY EXTRUDED SHAPES . UTHOR(.) CONTRACT OR GRANT NUMBER(.) F33615-75-C-5Ø61 15 10 R. /Grierson PERFORMING ORGANIZATION NAME AND ADDRESS PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS Cabot Corporation 62102F Stellite Division \vee 16 73510173 Kokomo, Indiana 46901 11. CONTROLLING OFFICE NAME AND ADDRESS Air Force Materials Laboratory (LLM) Apr 77 Wright-Patterson AFB, Ohio 45433 12 43 14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office) 15. SECURITY CLAS Unclassified 15. DECLASSIFICATION/DOWNGRADING SCHEDULE 16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited. 17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report) 18. SUPPLEMENTARY NOTES 19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Cobalt Alloy Cobalt-Nickel-Chromium Alloy Oxide Dispersion Strengthened Cobalt-Nickel-Chromium-Aluminum Alloy Yttria Dispersion Strengthened Extrusion High Temperature Alloy Mechanical Attrition 0. ABSTRACT (Continue on reverse side if necessary and identify by block number) The present study describes the development of an Oxide Dispersion Strengthened, Cobalt base alloy with the nominal composition of Co-20Ni-18Cr-4.5A1-1.75 Y503. The alloy was produced in extruded form from mechanically attrited powder. 3 IN. In the first part of this study a series of small scale $(\overline{\beta})^{n}$ dia.) extrusions were produced in order to evaluate various compositional and processing parameters. Based upon this study a single powder composition and three sets of extrusion parameters were chosen for use with full scale (7") diameter _ DD 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE -V Unclassified 7 IN. SECURITY CLASSIFICATION OF THIS PAGE (When Date Entered)

Unclassified SECURITY CLASSIFICATION OF THIS PAGE(When Date Entered) extrusion billets. These billets were extruded to give bar which, on decanning, had a cross section of approximately 2.75" x 0.8". A detailed property evaluation of the strongest of the extrusions produced showed that it had a stress rupture capability approximately 20 hrs./10 ksi/2000°F, 20 hrs./7 ksi/2200°F and 20 hrs./4.5 ksi/2400°F. The material did not have the low (<25 x 10^6 psi) modulus of elasticity which was sought as being desirable for aircraft gas turbine vane applications. The actual values achieved were in the range $26-30 \times 10^6$ psi. Dynamic (Mach 0.3), 2100 F oxidation tests showed the material to have excellent surface stability under these conditions, and hot corrosion testing at 16500F showed it to be slightly superior to the analogous Ni base 0.D.S. materials in this latter type of environment. Unclassified SECURITY CLASSIFICATION OF THIS PAGE(When Date Entered)

FOREWORD

This Final Technical Report covers all work performed under Contract F33615-75-C-5061 by the Stellite Division of the Cabot Corporation, Kokomo, Indiana, from 1 September 1974 to 31 December 1976. The report was released by the author on 31 January 1977.

This contract was initiated under Project 7351, "Metallic Materials", Task 735101, "High Temperature Materials", Work Unit 73510173. The work was performed under the technical direction of Mr. William T. O'Hara of the Metals and Ceramics Division of the Air Force Materials Laboratory (AFML/LLM), Wright-Patterson Air Force Base, Ohio.

Dr. Robert Grierson of the Stellite Division, Cabot Corporation, was responsible for the management and execution of the program. Dr. Grierson served as the principal investigator.

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SECTION I

INTRODUCTION

The present program is a continuation of a previous Air Force Materials Laboratory sponsored program on extruded oxide dispersion strengthened, cobalt-base alloys. In the initial studies (1-3) the desirability of such alloys had been demonstrated both by the measurement of the properties of the material, and also by engine testing in the TF-30 engine. The materials examined had compositions based on Co-20Ni-18Cr-2ThO₂ and Co-20Ni-30Cr-2ThO₂. They were produced at the Fansteel Dispersion Strengthened Materials facility. This facility ceased to operate in 1972, and, at that time, the Cabot Corporation purchased much of the available technical know-how and patents relating to the Fansteel method of producing dispersion strengthened alloys.

At the same time, Cabot had been working on the production of Oxide Dispersion Strengthened (ODS) alloys using a technique similar to the Inco mechanical alloying process (4). A nickel-base alloy, HAYNES Developmental alloy No. 8077, was successfully developed. It has the nominal composition Ni-16Cr-4Al-Y₂O₃. Its primary advantages over the earlier nickel base alloys produced by Fansteel (Ni-2ThO₂; Ni-20Cr-2ThO₂) were that it had excellent high temperature (2000°F) dynamic oxidation resistance (due to the aluminum content), and, that it was non-radioactive (due to the use of Y₂O₃ rather than ThO₂ as the dispersoid). The mechanical attrition process being used at Cabot to produce the powder does not suffer from the major disadvantage of the Fansteel production process, which relied upon the ability to reduce the oxides of the matrix metal and was not capable of producing a powder with an aluminum containing matrix.

As the Air Force Materials Laboratory had a continuing interest in the 0.D.S. Cobalt system, a short program was funded at Stellite to study the feasibility of using mechanical attrition to produce such materials. This study is described in a Technical Report AFML -TR-74-34 (5). The main part of this study was concerned with four alloys Co-20Ni-18Cr-1Y₂O₃, Co-20Ni-18Cr-2Y₂O₃, Co-20Ni-18Cr-1Al-1Y₂O₃, and Co-20Ni-16Cr-4Al-1Y₂O₃. It was in the last of these four that the most promising mechanical properties (2000°F) were obtained, and it is this composition which has been the starting point for the compositions studied in the present program.

At the same time as the above program was being carried out, additional studies on the nickel base analogue were being carried out at Stellite. One of these was a NASA sponsored program (6) which had as its aim the optimization of the aluminum content in the system Ni-16Cr-4Al-Y₂O₃. The conclusion reached was that the optimum Al content was between 4.5 and 5%. This fact was used to establish the Al content aim for the present program.

Another study which was being carried out was an in-house investigation of the effect of oxide content in the system Ni-16Cr-4Al-Y₂O₃. As will be described below in a section on the physical metallurgy of 0.D.S. alloys, these materials are highly anisotropic in the extruded form, and the degree of anisotropy can be controlled to a certain extent by the oxide content. It was therefore decided that, in the present program, various oxide contents would be examined initially.

SECTION II

PHYSICAL METALLURGY OF O.D.S. ALLOYS

The basic difference between an oxide dispersion strengthened alloy and a conventional alloy is the presence of a uniform dispersion of fine (100-500A°), thermally stable, oxide particles in the oxide dispersion strengthened alloy. This oxide controls the mechanical properties of the 0.D.S. alloy in two ways. Firstly, they act as barriers to the motion of dislocations, and thus raise the basic flow strength of the material. Secondly, they allow a form of thermo-mechanical processing to be used which is essentially not possible in a conventional alloy.

In this processing, the material is worked to its final shape while it has an extremely fine grain size. This fine grain size exists due to the pinning of grain boundaries by the dispersoid. Material in this form is then annealed at a temperature approaching its melting point. A comparatively unusual form of grain growth can then occur which can lead to a structure which is composed of large, non-equiaxed grains which have a definite crystallographic orientation with respect to the original working (in this case, extrusion) direction.

For applications such as stator vanes in an aircraft gas turbine, it is desired to have the maximum thermal fatigue resistance in a given direction. By suitable design of the part, this becomes the extrusion direction. To maximize thermal fatigue resistance, two properties are controlled - the longitudinal strength and the modulus of elasticity in the longitudinal direction. Maximum longitudinal strength (as defined by creep or stress rupture strength) is achieved when the re-crystallized grains are large and have a length which is great compared with their other dimensions. The desired elastic modulus is the minimum value. The reason for this is that a low elastic modulus allows a greater proportion of any thermally induced strains to be accommodated elastically, and thus results in less plastic deformation of the material during thermal cycling.

The lowest elastic modulus in a Face Centered Cubic material (such as the current Co-20Ni based materials) is associated with the 200 crystallographic direction in the extrusion direction. While this has been achieved in the nickel base alloys (Ni-2ThO₂, Ni-2OCr-2ThO₂, Ni-16Cr-4Al-Y₂O₃), there is no evidence of it having been achieved in a Cobalt base 0.D.S. alloy. One aim of the present program was therefore to produce material with this texture.

The previous studies of the nickel base system had shown that oxide content, extrusion temperature, extrusion speed and extrusion ratio are important variables in controlling the recrystallized structure. These have therefore been studied during the present program.

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SECTION III

EXPERIMENTAL STUDY

3.1 Introduction

The experimental study was composed of the production and evaluation of a number of series of extrusions. The initial extrusions were produced as round rod using the 700-ton extrusion press in the Air Force Materials Laboratory. The final series was a scaled up series produced using the 3850 ton press in the Reactive Metals, Inc. extrusion facility in Ashtabula, Ohio.

The basic format of the program has been that, when one series of extrusions had been produced and evaluated, the conditions for the next series would be selected. This same format will be followed in describing the experimental study and in reporting the results.

3.2 Powder Production

All O.D.S. powders produced in this program have been made by a mechanical attrition process. A detailed description of their production will not be given, however, due to the proprietary nature of the process. All starting powders for mechanical attrition and all mechanically attrited powders have been characterized so that it would be possible to reproduce them. Both in the present program and in the previous program (5) some difficulty has been encountered in eliminating all heterogeneity from these O.D.S. Co powders. No similar difficulty had been encountered in the nickel-base materials. In the present program, changes in starting powders and attrition conditions have been made between different lots in order to try to minimize these heterogeneities. For the purpose of clarification, these changes will be commented on when discussing the different extrusion series.

3.3 First and Second Series of Extrusions

Four 80-1b. lots of powder were produced using a 100S attritor. The first of these (Lot No. AT 223) was a wash heat and was not evaluated, while the others contained three different oxide levels (AT 225-1.75%, AT 229-1%, AT 230.0.6%). Two-pound samples of each of these three powders were canned in evacuated, mild steel containers and extruded using the 300-ton extrusion press at Stellite. Metallographic examination showed all three to be reasonably homogeneous, and so further processing of these powders was carried out. Three cans of each composition were prepared for extrusion using the 700-ton press in the Air Force Materials Laboratory. The extrusion conditions used are listed in Table 1. The prime variables studied in this series were the extrusion temperature and the oxide content of the powder.

Table II lists the nominal and analyzed compositions of the extruded rod. In spite of the use of a wash heat before the production of Heat AT225, carry-over from previous heats produced in the attritor used caused a deviation from the nominal composition. It should be noted that the oxygen present is in excess of that required for stoichemetric Y_2O_3 . In the analogous nickelbased alloy, the dispersoid is actually a mixture of Y_2O_3 and $Y_4Al_2O_9$. Both oxides have good particle size stability on high temperature exposure.

Decanned samples of the extrusions produced at Stellite were used to determine melting point for these and for all subsequent extrusions. A similar result has been observed in all cases. While these materials would ideally be very homogeneous, they are in actual fact no so. This results in small differences in melting point between different regions. Specimens have been annealed at various temperatures and then examined metallographically. the first signs of incipient melting are seen at temperatures just above 2500°F (Fig. 1). Considerable melting can be seen in most materials at 2550°F (Fig. 2. 2500°F has therefore been regarded as the maximum temperature to be used during the recrystallization heat treatment. However, wherever possible, somewhat lower temperatures (2400 or 2450°F) have been used.

The structure of a piece of a given extruded and recrystallized oxide dispersion strengthened alloy is a function, not only of the extrusion parameters used, but also of the recrystallization heat treatment. If a piece is heated rapidly to the maximum temperature, then it will tend to have larger grains (and thus better high temperature strength) but a higher modulus than if it were heated slowly. Two types of heat treatment have therefore been used with most of the extrusions examined. In the fast heat treatment, the specimen is introduced directly into the hot zone of a furnace at the maximum desired temperature, while in the slow heat treatment it is put into the furnace at a temperature where little or no recrystallization will occur, and the temperature is then slowly raised to the maximum desired temperature.

Figures 3, 4 and 5 show AT225C, AT229C and AT 230C, respectively, after being given either a fast (direct insertion at 2500°F, hold 1 hr.), or a slow (in furnace at 1900°F, raise furnace temperature over a period of approximately 2 hrs. to 2500°F, hold for one hour) anneal. The tendency for the fast anneal to give larger grains is obvious. The dark regions, which are particularly obvious in the lower micrographs in Figures 4 and 5, are regions which have a very fine grain size.

Samples from nine extrusions were given both the fast and the slow heat treatments as outlined above, and metallographic mounts prepared. Rather than fill the present report with many micrographs, a numerical system for describing structures will be used. Structures will be divided into four general types as follows:

| Type 1 | large, elongated grains. |
|--------|--|
| Type 2 | primarily medium-sized elongated grains. Some fine equiaxed grains may however be present. |
| Type 3 | a mixture of medium-sized elongated grains and very fine equiazed grains. |

Type 4 fine, primarily equiazed grains.

Using this system, the various microstructures observed are described in Table III. In terms of the high temperature strength of the alloy, Type 1 is expected to be the strongest with Type 4 being the weakest. Figures 3 to 8 are catagorized into the four above listed general structural types. In addition to high temperature strength, the longitudinal sonic modulus of the recrystallized extrusions is of prime concern. Table IV lists the sonic modulus of this first series of extrusions after they had been given either the fast or the slow heat treatment. The only material which had the desired low (<25 x 10^6) modulus was AT225C given the slow heat treatment.

Evaluation of the various structures and modulus values listed in Tables III and IV shows that AT225 is the most promising of the three compositions, presumably due to its higher oxide content. However, due to its having been the first of the three powders produced, it is furthest from the nominal composition. It was therefore decided to produce another powder lot which would be closer to the nominal composition. This powder lot is AT242 and its nominal and analyzed compositions are listed in Table II. While the nickel and cobalt levels are much closer to the nominal than in AT225, the aluminum level is still low and the iron level high.

A second series of extrusions was then carried out. The conditions used are listed in Table IV. The temperature of 2200°F was chosen for extrusions AT225D, AT229D and AT230D due to the fact that, in the previous series, the best structures had been obtained at the maximum temperature used (2100°F). AT242A, B and C were reiterations of conditions used in the first series while AT242D, E, F and G examined the effect of reduction ratio and extrusion speed.

Specimens of all ten extrusions were given the fast and the slow heat treatments. All three 2200°F extruded materials failed to undergo complete recrystallization with either heat treatment. The 1900°F and 2000°F extrusions of AT242 (A and B respectively, Figure 6) have primarily fine-grained structures with the slow heat treatment. AT242A gave a structure similar to AT225A when fast heat-treated (large elongated grains) while AT242B still contained some fine grains.

AT242C-G gave similar structures regardless of which heat treatment was used (Figure 7). The main difference between the different extrusions was the shape of the regions of unrecrystallized material. These regions were more elongated with the higher reduction ratios. The extent of unrecrystallized material is approximately 10% in the fast heat-treated material and 20% in the slow heat-treated material. The structures were thus slightly inferior to those obtained with the equivalently processed AT225C.

Specimens for sonic modulus were taken from all ten extrusions. The three 2200°F extrusions and the 1900°F extrusion of AT242 were given fast heat treatment while the rest (AT242B-G) were given slow heat treatments. The data obtained is listed in Table IV. The low values (<22 x 10^6 psi) obtained for four of the slow heat treated extrusions are regarded as being very promising.

From the nineteen extrusions in the first two series, ten were selected for an evaluation of 2000°F tensile and stress rupture properties. The heat treatment given and the properties obtained are listed in Table V. Table VI summarizes the most important properties. While some specimens had the desired low modulus of elasticity and others easily exceeded the stress rupture goal, no single material possessed the required combination of properties.

3.4 The Third and Fourth Series of Extrusions

While the required combination of high temperature strength and longitudinal modulus was not achieved in any of the material of the first two extrusion series, the results gave a basis for further experimentation. The most promising materials were those which contained the highest oxide level and those which had been extruded at 2100°F. These areas were concentrated upon in the third series of extrusions.

The poor behavior of the materials extruded in the first two series could possibly be a function of the degree of attrition of the powders. A series of six powder lots were produced using a smaller (10S) attritor and smaller powder lots. Two different types of starting powders were used in an attempt to vary the degree of attrition. The first three lots (AT267, AT270 and AT271) contained three different, high oxide levels (2.0 w/%, 1.66 w/% and 1.33 w/% respectively). The fourth powder lot (AT272) consisted of part of Lot AT271 which was re-introduced into the attritor and given an extra 24 hours of processing in order to vary its degree of attrition. The other two powders were produced from a third type of starting blend. The oxide content was 2 w/%, and one lot was processed for 48 hrs. (AT273) and the other for 72 hrs. (AT278).

Two billets of each of the six powder lots were produced for extrusion at 2100°F using the AFML extrusion press. Two sets of extrusion conditions were chosen for use with each powder lot. One involved a reduction ratio of 16:1, and an extrusion speed of 2 inches per second; while the other had a ratio of 20:1 and a speed of 10 i.p.s. In looking toward the future scale up of this alloy system, the first condition is readily achieved using a number of commercial presses; while the second condition, expected to give a better product, would stretch the capability of currently available production presses.

Samples of all twelve extrusions were decanned by acid pickling and then recrystallized by heating slowly (in approximately two hours) from 2100°F to 2450°F. Metallographic chemistry and sonic modulus samples were then taken. The chemistries obtained are listed in Table VII and the sonic modulus values in Table VIII. Only the high extrusion ratio/high extrusion speed version of AT273 and AT278 have the required low modulus values.

Examination of the structures shows a considerable difference between the first four and the last two powder lots. Extrusions of the first four contain a mixture of very fine and fine, equiaxed grains. They are somewhere between the Type 3 and Type 4 structures described earlier, and would be expected to have extremely poor, 2000°F stress rupture strength. For this reason they were not tested. The other two powder lots gave structures which were fully recrystallized although their grain structure was neither as large or as elongated as that structure which it is believed would give the required strength properties. Mechanical testing of these extrusions was, however, carried out and the stress rupture data obtained is listed in Table IX. These materials have similar properties to some of the material produced earlier (see Table VI). However, it should be noted that they have different grain structures and are more homogeneous in grain size. This is regarded as a major step in the required direction.

In order to follow up on the results obtained with powder Lots AT273 and AT278, powder Lot AT285 was produced using those starting powders and attrition conditions that had been used with AT273. The chemistry of AT285 is listed in Table VII. Six extrusion billets of this powder were prepared and extruded under the conditions listed in Table X. Samples for metallography and for sonic modulus were decanned and given a slow heat treatment. The modulus values obtained are listed in Table VII. The moduli were all higher than desired, 25×10^6 pounds/square inch being the maximum acceptable. Metallographic examination showed that the $2100^{\circ}F$ extrusions had fully recrystallized, elongated grain structures while the lower temperature extrusions had fine, more equiazed structures. However, since the moduli of all the materials were high, it was decided not to carry out any additional mechanical testing.

Even though the third and fourth series of extrusions did not produce a material with the required properties, they did demonstrate that the starting powders used could have a pronounced effect upon the properties obtained. Evaluation of the various powder lots showed no relationship between the existence of chemical heterogeneities (possibly due to incomplete mechanical alloying) and the response to processing. However, comparison of the oxygen content of the various Co base powder lots with each other, and with analogous Ni base systems produced at Stellite, suggested that the amount of excess oxygen present was possibly the controlling factor. Excess oxygen means that amount of oxygen which is present in excess of the amount to form stoichemetric Y_2O_3 . For the fifth set of extrusions a further change in starting powders was therefore made in order to reduce the amount of excess oxygen in the attrited powder.

3.5 The Fifth Series of Extrusions

In microprobe and metallographic observation of the earlier lots of powder, no problems were apparent due to the ability to attrit the fine high cobalt content powder being used in the starting charge. This fine powder was one of the major sources of excess oxygen. For the fifth series of extrusions it was therefore decided to replace it with a coarser, lower oxygen content powder produced by inert gas atomization. Two lots of powder(AT327 and AT328) were produced with differing oxide contents (nominally 2% and 1.33% respectively). The analyses compositions are given in Table XI. Three billets of each powder were prepared and extruded at the temperatures listed in Table XII using a nominal ram speed of 5 i.p.s. and a reduction ratio of 16:1. Specimens from each extrusion were decanned and given both the fast and the slow heat treatments. Metallographic examination showed that there was very little difference between the two oxide levels, but the fast heat treatment gave a more fully recrystallized structure than did the slow. Sonic modulus pins of all six extrusions were then prepared with the three AT327 specimens being in the fast heat treated condition, and the three AT327 specimens being in the slow heat treated condition. The modulus values obtained are listed in Table XII. All materials have a modulus appreciably in excess of 25 x 10^6 p.s.i. As the slow heat treatment did not give a low modulus, it was decided to do all mechanical property evaluation on material given the fast heat treatment. The data obtained is listed in Table XIII for 2000°F stress rupture testing, and in Table XIV for 2000°F tensile testing. The 2000°F stress rupture data obtained for this series of extrusions shows that the materials are very little stronger than AT273 and AT278 (Table IX) and not as strong as the earlier produced materials, such as AT225A and AT242A (Table VI).

The next stage in the program called for the scale up of the best of the materials which had been produced in these small scale series. None of the materials had the desired combination of high 2000°F stress rupture strength and low longitudinal modulus which is required for vane applications. However, the high 2000°F stress rupture strengths of materials such as AT225A, coupled with their expected superior surface stability properties, would make this type of material of interest in structural applications where the control of modulus is not of such great importance. For this reason it was decided to scale up a material of the AT225, AT242 type.

SECTION IV

PHASE TWO - SCALE UP

4.1 Production of Extrusions

Phase Two of the program was composed of the production and evaluation of four full scale extrusions. Powder for these extrusions was produced as two 160-1b. lots (AT374 and AT375) using a 100S attritor. Small scale extrusions of each lot were produced to check the powder homogeneity, and, when this was found to be acceptable, two 7" O.D. 6-1/2" I.D. extrusion cans of each powder were prepared. To conform with an inhouse numbering system, the two billets of AT374 as O.D.S. Co-76004 and O.D.S. Co-76006, and the two billets of AT375 as O.D.S. Co-76003 and O.D.S. Co-76005. The billets were evacuated in the normal manner and extruded using the 3850 ton press at R.M.I., Ashtabula, Ohio. Extrusion conditions are listed in Table XV. The die cavity used was rectangular in cross section with an approximate size of 2.93" x 0.95". After extrusion the material was measured at various points along its length. As would be expected, the bars were uniform in cross section. However, they had a slight twist.

4.2 Initial Evaluation of Scaled Up Extrusions

A section was cut from the center of each bar and both micro- and macrographic specimens were prepared using material given the standard fast and slow heat treatments (maximum temperature 2450°F). All material given the fast heat treatment was fully recrystallized while that given the slow heat treatment was not. Some inhomogeneity was present in all the materials with the extrusions 0.D.S. Co-76004 and 75006 being more homogeneous than 0.D.S. Co-75003 and Co-76005. Figure 8 compares the fast heat treated versions of 0.D.S. Co-76004 and 0.D.S. Co-76006. The higher extrusion temperature had resulted in larger, more elongated grains. This was as expected and the same pattern was seen in comparing 0.D.S. Co-76003 and 0.D.S. Co-76005. Chemical analysis samples were taken from all four alloys and the data obtained is listed in Table XVI. A fuller analysis was carried out of 76005 and 76006 than that of 76003 and 76004.

Longitudinal stress rupture and sonic modulus samples were taken from near the middle of the length of each of the four extruded bars. Four specimens were taken from a cross section and note was made as to whether they were from near the center or near the edge. The reason for this is that there is a variation in properties over a cross section. All specimens were tested in the fast heat treated condition and the data obtained is listed in Table XVII.

The 2000°F stress rupture lives obtained showed that the two highest temperature extrusions (76005 and 76006) were slightly stronger than the two extrusions produced at lower temperatures. They also had slightly lower elastic moduli. For these reasons it was decided to use 0.D.S. Co-76005 and Co-76006 to conduct a more detailed property evaluation.

4.3 Detailed Evaluation of Mechanical Properties

Duplicate tensile tests were carried out in air on longitudinal specimens at room temperature 1800°F, 2200°F. The data obtained is listed in Table XVIII. The room temperature and 1800°F tests were carried out using a strain rate of .005 inches/inch/min. to yield and 0.5 inches/inchmin. to failure. These tests were conducted at the Stellite Division. The 2200°F and 2400°F test were performed by Metcut Research Associates, Inc. using a strain rate of 0.005 inches/inch min. to yield and 0.05 inches/inch/min. to failure.

Triplicate stress rupture tests were carried out by Metcut in air at 2200°F and 2400°F. The data obtained is listed in Table XIX. While these materials have limited ductility at 2200°F and 2400°F, both in the tensile and stress rupture test, they do have a useable strength level at both temperatures. This is the most significant observation concerning their mechanical properties.

4.4 Dynamic Oxidation Testing

Dynamic oxidation testing of duplicate samples of extrusions 0.D.S. Co-76005 and 0.D.S. Co-76006 was carried out at Stellite using a flame tunnel type rig which provided a gas velocity of Mach 0.3. The combustible mixture was composed of air and No. 2 fuel oil in a ratio by weight of approximately 54:1. The test duration was 100 hrs. and the test temperature was 2100°F. The test specimens used were flat plates orientated so that the extrusion direction was in the plane of the plate. Four other alloys were evaluated in the same run for the purpose of comparison. Two were 0.D.S. alloys and two conventional alloys. The nominal compositions are given in Table XX.

The specimens were subjected to cycles of 30 minutes in the Test Chamber followed by a two-minute air quench throughout the 100 hour test duration. Periodically, the specimens were removed and weighed to document weight changes. At the conclusion of each test the specimens were sectioned, nickel plated and prepared metallographically to determine oxidation penetration. The definition of the parameters determined by this examination are schematically illustrated in Figure 9.

The data obtained in this test is listed in Table XXI. In terms of the Total Metal Affected, which is the most important parameter, the three O.D.S. alloys are very similar and are superior to the two conventional alloys. This excellent oxidation resistance is due to the deliberate choice of the Al and Cr levels.

4.5 Hot Corrosion Testing

Hot corrosion testing was carried out on the same materials, both O.D.S. and reference that had been used in dynamic oxidation. The gas velocity in the hot corrosion rig is 13 ft./sec. No. 2 fuel oil (0.3 to 0.45%S) was burned with an air to fuel ratio of 3:1 and a test temperature of 1650°F. Five parts per million sea salts were added to the fuel.

The test duration was 200 hours and the specimens were cycled in and out of the rig. For each cycle the specimen spent 60 minutes in the rig followed by five out. Periodically the test was stopped and the specimens were weighed to record weight loss. The evaluation of this test was carried out in a similar manner to that of the dynamic oxidation test with the exception that the weight change is reported in milligrams rather than milligrams/sq. cm. The data obtained is listed in Table XXII.

Looking at the measurement of Total Metal Affected it can be seen that the O.D.S. Co materials have improved hot corrosion resistance as compared to the other O.D.S. materials. They still, however, are not quite as good as the conventional alloys which were tested. It would be expected that, if the chromium level of the O.D.S. Co alloy was increased and, if deliberate, small additions of Si and Mn were made, then a further improvement in hot corrosion resistance would be seen. The improved behavior of the Co base O.D.S. material as compared with the Ni and Ni-Fe base is regarded as significant.

4.6 Thermal Expansion

The coefficient of thermal expansion of a 1"-long specimen of 0.D.S. Co-76006 was measured using a Theta Industries Dilatometer. This equipment produces an apparent length vs. temperature curve for both the specimen being evaluated, and also for a Platinum Standard. The two curves are then computer processed to give the coefficient of expansion between room temperature (68°F in this case) and various elevated temperatures. The data obtained is listed in Table XXIII. It should be noted that the maximum temperature reached in the test was 1926°F, and that the data listed for 2000°F was obtained by extrapolation.

4.7 Density

The specific gravity of a piece of 0.D.S. Co-76006 was determined to be 7.765 grams/cc by the mercury immersion technique

SECTION V

DISCUSSION

The production of 0.D.S. Co-base alloy extrusions with the hoped for combination of low elastic modulus and excellent stress rupture strength was not fully achieved, either in the initial small scale work or in the final full scale extrusions. In general, this system seemed to be more difficult to work with than the analogous nickel base alloy. When processing parameters such as extrusion temperature, reduction ratio and extrusion speed were varied, the material responded in a manner which is similar to that which normally would occur in the analogous Ni base alloys. There are, therefore, some additional processing routes, such as secondary hot working of the extrusions prior to recrystallization which might be expected to give improved properties.

Some comparatively strong (but poorly textured) materials were produced in the early stages of the program. AT225A (see Table V for example) had a 2000°F stress rupture capability of over 200 hrs. at 14 k.s.i. Materials of this type could well be of interest for applications in which high temperature strength rather than thermal fatigue resistance are important. In the scaled up product, however, the same strength levels were not achieved.

One of the hoped for advantages of the Co-base O.D.S. alloy over its Ni-base analogue was that it would have improved hot corrosion resistance with similar dynamic oxidation resistance. This was found to be the case.

TABLE I

EXTRUSION CONDITIONS FOR 1ST EXTRUSION SERIES

| Reduction Ratio | - | 15.8:1 |
|-----------------|---|-------------------------|
| Die | - | 90 ⁰ Conical |
| Lubrication | - | Polygraph |

A LOT A

| Extrusion Number | Nominal ^Y 2 ^O 3 Composition (%) | Extrusion Temperature (^O F) | Extrusion Speed (IPS) |
|---------------------|--|---|-----------------------------|
| AT225A | 1.75 | 1900 | 1.7 |
| В | 1.75 | 2000 | 1.9 |
| С | 1.75 | 2100 | 1.8 |
| AT229A | 1.0 | 1900 | 1.7 |
| В | 1.0 | 2000 | 1.8 |
| С | 1.0 | 2100 | 1.8 |
| AT230A | 0.6 | 1900 | 1.6 |
| В | 0.6 | 2000 | 1.8 |
| С | 0.6 | 2100 | 1.7 |
| | | | |

TABLE II

ANALYSED COMPOSITIONS OF POWDERS USED IN FIRST EXTRUSION SERIES

Nominal Compositions are:

- AT 225 Co-20Ni-18Cr-4.5A1-1.75Y₂O₃ AT 229 Co-20Ni-18Cr-4.5A1-1 Y₂O₃
- AT 230 Co-20Ni-18Cr-4.5A1-0.6 Y203
- AT 242 Co-20Ni-18Cr-4.5A1-1.75 Y203

| Element | AT225 | AT229 | AT230 | _AT242 |
|---------|--------|--------|--------|--------|
| A1 | 3.91 | 4.07 | 4.35 | 3.46 |
| с | .06 | .05 | .04 | .05 |
| Co | 45.3 | 49.80 | 52.03 | 50.34 |
| Cr | 16.5 | 16.81 | 16.78 | 15.82 |
| Cu | < 0.01 | < 0.01 | .01 | .01 |
| Fe | 5.34 | 3.65 | 2.86 | 7.45 |
| Mn | .05 | .04 | .02 | .06 |
| Мо | .03 | .03 | .02 | < .01 |
| N | .06 | .044 | .048 | .039 |
| Ni | 26.26 | 23.43 | 22.26 | 20.91 |
| 0 | .68 | .56 | .50 | - |
| P | < .001 | < .001 | < .001 | < .001 |
| S | .004 | .005 | .006 | .006 |
| Si | .04 | .04 | .05 | .03 |
| W | .03 | .04 | .03 | < .01 |
| Y | 1.22 | 0.80 | .60 | 1.02 |
| | | | | |

| | STRUCTURE | TYPES | OF FIRST SER | LIES OF ODS C | OBALT EX | TRUSIONS | |
|-------|-----------|--------------------|--------------------|--------------------|---------------------------|---------------------------|--------------------|
| | | | | | | | |
| | | -A Fast H.T. | -A Slow H.T. | -B Fast H.T. | -B Slow <u>H.T.</u> | -C Fast <u>H.T.</u> | -C Slow H.T. |
| AT225 | | 1 | 4 | 1 | 3 | 1 | 2 |
| | | | | | | | |
| AT229 | | 2 | 4 | 2 | 4 | 3 | 3 |
| AT230 | | 2 | 4 | 2 | 3 | 3 | 3 |
| | 1.6 | | | | | | |
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TABLE III

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TABLE IV

EXTRUSION CONDITIONS AND SONIC MODULUS VALUES FOR THE FIRST TWO EXTRUSION SERIES

| | Extrusion Temp. | Extrusion Speed | De J | M of E (Fast Heat Treatment) | M of E (Slow Heat Treatment) |
|----------|--------------------|--------------------|-------|------------------------------------|------------------------------------|
| Ext. No. | (⁰ F) | (ips) | Ratio | (psi x 10 ⁶) | (psi x 10 ⁶) |
| | | | | | |
| AT225A | 1900 | 2 | 16:1 | 30.9 | - |
| В | 2000 | 2 | 16:1 | - | 29.4 |
| С | 2100 | 2 | 16:1 | - | 22.5 |
| D | 2200 | 2 | 16:1 | 28.8 | - 101235 |
| AT229A | 1900 | 2 | 16:1 | 31.4 | - |
| В | 2000 | 2 | 16:1 | | 30.1 |
| С | 2100 | 2 | 16:1 | - | |
| D | 2200 | 2 | 16:1 | 29.0 | UK 298. |
| AT230A | 1900 | 2 | 16:1 | 30.1 | - |
| В | 2000 | 2 | 16:1 | - | 27.6 |
| C | 2100 | 2 | 16:1 | - | 25.5 |
| D | 2200 | 2 | 16:1 | 27.1 | - |
| AT242A | 1900 | 2 | 16:1 | 31.1 | - |
| B | 2000 | 2 | 16:1 | | 28.1 |
| С | 2100 | 2 | 16:1 | - | 21.4 |
| D | 2100 | 2 | 10:1 | - | 26.4 |
| Е | 2100 | 2 | 20:1 | - | 20.5 |
| F | 2100 | 5 | 16:1 | - | 20.5 |
| G | 2100 | 5 | 10:1 | - | 21.8 |

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TABLE V

2000°F MECHANICAL PROPERTIES OF TEN SELECTED EXTRUSIONS OF THE FIRST TWO EXTRUSION SERIES

| | | Stress Rupture | | Tensile | |
|---------------|-------------------|-----------------------------|----------------|-----------------|-------------|
| Extrusion No. | Heat Treatment | Stress (ksi)/Life (hrs) | Yield (ksi) | U.T.S. (ksi) | Elong. % |
| 225A | F | Uploaded to 16/7.4: | 19.1 | 19.1 | 3.2 |
| | F | 14/298.7 + 15/4.7: | 19.3 | 19.3 | 3.3 |
| | enviat sing | 14/298.7 + 15/16.3 | Heat | | in Barris |
| В | F | 15/19.4; 14/161.3; 14/197.4 | 16.8 | 17.1 | 4.0 |
| | F | 10.9 | 16.5 | 16.5 | 3.4 |
| С | S | 10/3.6; 8/92.8; 8/95.4 | 12.8 | 14.5 | 4.9 |
| | S | | 12.7 | 14.5 | 4.2 |
| 229A | F | 10/2.1; 9/7.6; 7/113.8 | 19.2 | 19.3 | 2.0 |
| | F | | | 20.0 | 1.9 |
| С | S | 8/0; 4/0.3; 4/0.3 | 6.7 | 10.6 | 3.6 |
| | S | A States | 7.5 | 7.8 | 3.7 |
| 230A | F | 10/2.3; 7/82.8; 7/56.6 | 18.1 | 18.1 | 1.9 |
| | F | | 19.3 | 19.3 | 2.1 |
| С | S | 8/0; 6/0; 4/0.2 | 6.2 | 6.4 | 8.6 |
| | S | | 6.1 | 6.4 | 10.2 |
| 242A | F | 15/13.1; 14.5/29.2; 14/62.3 | 18.7 | 18.7 | 3.7 |
| | F | | 19.5 | 19.5 | 4.3 |
| С | S | 10/0.7; 7/110.6; 7/145.8 | 12.6 | 12.9 | 5.7 |
| | S | | 12.6 | 13.0 | 3.5 |
| Е | S | 10/4.0; 8/88.2; 8/46.5 | 12.2 | 12.6 | 3.9 |
| | S | | 12.0 | 12.7 | 5.7 |

TABLE VI

SUMMARY OF THE PROPERTIES OF THE TEN SELECTED EXTRUSIONS

OF THE FIRST TWO EXTRUSION SERIES

| Extrusion Numbers | Heat Treatment | Modulus (p. s.i x 10 ⁶) | Approximate Stress For 100 Hrs Life (k.s.i.) | Structure Type |
|----------------------|-------------------|---|---|-------------------|
| AT225A | F | 30.9 | 14.5 | 1 |
| AT225B | F | - <u>899</u> 38 : 87 <u>89</u> - | 14.5 | 1 |
| AT225C | S | 22.5 | 8 | 2 |
| AT229A | F | 28.8 | 7 | 2 |
| AT229C | S | 25.6 | <3 | 3 |
| AT230A | F | 30.1 | 6.5 | 2 |
| AT230C | S | 25.5 | <3 | 3 |
| AT242A | F | 31.1 | 13.5 | 1 |
| AT242C | S | 21.4 | 7 | 2 |
| AT242E | S | 20.5 | 7.5 | 2 |
| | | | | |
| Aim | | <25 | 11 | 1 |

TABLE VII

CHEMICAL ANALYSIS OF THE ODS CO-BASE LOTS

USED IN THE THIRD EXTRUSION SERIES

| Element | AT267 | <u>AT270</u> | AT271 | <u>AT272</u> | <u>AT273</u> | AT278 | AT285 |
|---------|-------|--------------|-------|--------------|--------------|-------|--------|
| Al | 3.67 | 3.91 | 3.93 | 4.02 | 4.20 | 3.91 | 4.24 |
| С | 0.02 | 0.11 | 0.09 | 0.02 | 0.10 | 0.10 | .15 |
| Co | 54.94 | - | 52.56 | 54.06 | 49.55 | 52.47 | 50.78 |
| Cr | 18.01 | 17.70 | 18.29 | 17.66 | 19.93 | 18.28 | 18.87 |
| Cu | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 |
| Fe | 0.65 | 0.54 | 0.85 | 0.81 | 0.97 | 0.68 | 0.56 |
| Mn | 0.01 | 0.01 | 0.02 | 0.02 | 0.02 | 0.01 | <0.01 |
| Мо | 0.02 | 0.02 | 0.03 | 0.03 | 0.03 | 0.02 | 0.02 |
| N | 0.069 | 0.060 | 0.044 | 0.083 | 0.067 | 0.068 | 0.08 |
| NÍ | 18.87 | 19.44 | 19.22 | 19.55 | 20.07 | 19.59 | 19.51 |
| 0 | .85 | .76 | .64 | .68 | .73 | .76 | - |
| P | 0.002 | 0.002 | 0.001 | 0.001 | 0.001 | 0.001 | <0.001 |
| S | 0.007 | 0.005 | 0.008 | 0.006 | 0.004 | 0.004 | 0.002 |
| Si | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.04 |
| W | 0.04 | 0.04 | 0.19 | 0.08 | 0.60 | 0.22 | 0.89 |
| Y | 1.46 | 1.32 | 1.34 | 1.12 | 1.27 | 1.33 | 2.0 |

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TABLE VIII

SONIC MODULUS VALVES FOR THE THIRD SERIES OF COBALT BASE EXTRUSIONS (in psi x 10^6)

| Powder Lot | | Extrusion (| Conditions | |
|------------|--|-------------------|-------------|--|
| 18.5 | | <u>16:1/2 ips</u> | 20:1/10 ips | |
| AT267 | | 28.3 | 26.2 | |
| AT270 | | 28.4 | 25.8 | |
| AT271 | | 29.0 | 27.2 | |
| AT272 | | 29.6 | 27.5 | |
| AT273 | | 30.6 | 23.15 | |
| AT278 | | 31.2 | 22.55 | |

TABLE IX

2000°F STRESS RUPTURE PROPERTIES OF AT272 AND AT278

(ALL MATERIAL IN THE SLOW HEAT TREATED CONDITION WITH STRUCTURE OF TYPE 2)

| | Test Temp., F | Stress ksi | Rupture Time Hours | Elong. | R.A. % |
|---------------|------------------|---------------|-----------------------|--------|-----------|
| AT273-1 | 2000 | 10.0 | 1.8 | 1.9 | 2.2 |
| e, <u>Car</u> | 2000 | 8.0 | 17.5 | 3.0 | 2.2 |
| • 8. | 2000 | 8.0 | 13.0 | 3.1 | 2.8 |
| AT278-1 | 2000 | 10.0 | 20.5 | 3.1 | 2.9 |
| - H | 2000 | 9.0 | 39.0 | 2.7 | 2.0 |
| H | 2000 | 9.0 | 73.7 | 1.8 | 2.8 |
| AT273-2 | 2000 | 10.0 | 0.3 | 2.3 | 3.1 |
| H | 2000 | 8.0 | 1.7 | 2.0 | 2.3 |
| " | 2000 | 8.0 | 1.7 | 1.5 | 3.0 |
| AT278-2 | 2000 | 10.0 | 7.3 | 3.3 | 5.8 |
| " | 2000 | 8.0 | 87.3 | 2.5 | 2.8 |
| н | 2000 | 8.0 | 65.3 | 1.3 | 2.0 |

TABLE X

PARAMETERS FOR EXTRUSION THE FOURTH SERIES OF EXTRUSIONS

| Billet No. | Extrusion Temp., F | Reduction Ratio | Ram Speed (ips) | Sonic Modulus (psi x 10 ⁶) | Structure Type |
|---------------|-----------------------|--------------------|--------------------|--|-------------------|
| AT285 A | 2100 | 16:1 | 5 | 28.8 | 1 |
| AT285 B | 2100 | 16:1 | 2 | 30.3 | 1 |
| AT285 C | 2000 | 16:1 | 5 | 29.6 | 2 |
| AT285 D | 2000 | 16:1 | 2 | 30.8 | 2 |
| AT285 E | 1900 | 16:1 | 5 | | 2 |
| AT285 F | 1900 | 16:1 | 2 | 32.1 | 2 |

(ALL MATERIAL IN THE SLOW HEAT TREATED CONDITION)

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TABLE XI

CHEMICAL ANALYSIS OF POWDERS USED IN THE FIFTH EXTRUSION SERIES

| Element | Powder <u>AT327</u> | Lot AT328 |
|---------|------------------------|--------------|
| Al | 4.85 | 5.20 |
| C | .04 | .07 |
| Co | 49.70 | 50.38 |
| Cr | 17.54 | 17.78 |
| Cu | .01 | .01 |
| Fe | .62 | .72 |
| Mn | .01 | .01 |
| Мо | <.01 | .01 |
| N | .052 | .047 |
| Ni | 23.95 | 23.49 |
| 0 | .58 | .48 |
| Р | <.001 | .002 |
| S | < .002 | <.002 |
| Si | .03 | .03 |
| W | < .04 | <.04 |
| Y | 1.39 | 1.08 |

TABLE XII

MODULUS OF ELASTICITY OF AT327 AND AT328

| Material | Extrusion Temp. ([°] F) | Heat Treat. | Modulus (psi x 10 ⁶) |
|----------|--------------------------------------|----------------|-------------------------------------|
| AT327 A | 1900 | Fast | 28.7 |
| В | 2000 | " | 30.0 |
| с | 2100 | " | 32.6 |
| AT328 A | 1900 | Slow | 32.3 |
| В | 2000 | " | 27.8 |
| C | 2100 | " | 27.2 |

No.

TABLE XIII

STRESS RUPTURE LIVES OF AT327 AND AT328 TEST CONDITION -2000°F, 10 KSI.

| Material | . N | S-R Lives (in hrs.) | e.at | Avg. Life (hrs.) |
|----------|-----|---------------------|------|------------------|
| AT327A | | 17.5, 20.5, 29.7 | | 22.5 |
| 327B | | 57.6, 73.3, 59.5 | | 63.5 |
| 327C | | 21.1, 32.0, 27.4 | | 26.8 |
| | | | | |
| AT328A | | 73.1, 75.7, 116.6 | | 88.5 |
| 328B | | 29.1, 31.0, 22.2 | | 27.4 |
| 328C | | 42.1. 11.9. 35.8 | | 29.9 |

TABLE XIV

2000⁰F TENSILE PROPERTIES OF AT327 AND AT328

(ALL MATERIAL IN THE FAST HEAT TREATED CONDITION)

| <u>Material</u> | Yield (0.2%) ksi | U.T.S. (ksi) | Elong% |
|-----------------|---------------------|-----------------|--------|
| AT327A | 16.9 | 17.4 | 5.3 |
| A | 20.0 | 20.2 | 3.4 |
| AT327B | 17.1 | 17.4 | 3.9 |
| В | 17.2 | 17.5 | 5.7 |
| AT327C | 15.5 | 16.2 | 8.7 |
| С | 15.9 | 16.7 | 9.3 |
| AT328A | 19.0 | 19.2 | 3.7 |
| A | 19.0 | 19.1 | 6.2 |
| AT328B | 21.6 | 21.7 | 5.3 |
| В | 16.3 | 16.9 | 5.5 |
| AT328C | 14.7 | 15.3 | 8.0 |
| с | 14.4 | 15.2 | 6.4 |

TABLE XV

EXTRUSION PARAMETERS FOR THE SCALED UP BILLETS

| Billet No. | Extrusion Temp. (°F) | Transfer Time(Secs) | Extrusion Time(Secs) | Extrusion Tonnage (Start) | Extrusion Tonnage (Run) | Extrusion Speed (i.p.m.) |
|---------------|----------------------------|------------------------|-------------------------|---------------------------------|-------------------------------|--------------------------------|
| 76003 | 1900 | 39 | 5 | 2670 | 2605 | 140 |
| | | | | | | |
| 76004 | 2000 | 39 | 4.5 | 2560 | 2515 | 150 |
| | | | | | | |
| 76005 | 2100 | 41 | 4.5 | 2790 | 2645 | 150 |
| | | | | | | |
| 76006 | 2100 | 43 | 4 | 2675 | 2630 | 150 |

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TABLE XVI

CHEMICAL ANALYSIS OF FULL SCALE EXTRUSIONS

| | 76003 | 76004 | 76005 | 76006 |
|----|-------|---------|-------|-------|
| Al | 5.05 | 5.08 | 4.95 | 5.11 |
| с | .036 | .024 | .023 | .025 |
| Со | 53.49 | 53.76 | 54.03 | 53.27 |
| Cr | 18.10 | 17.78 | 17.90 | 17.97 |
| Cu | - | - | .01 | .01 |
| Fe | .35 | .35 | .22 | .44 |
| Mn | - | - | <.01 | .03 |
| Мо | - | _ 27.95 | .02 | .02 |
| N | .079 | .061 | .033 | .079 |
| Ni | 19.50 | 19.35 | 19.51 | 19.62 |
| 0 | .66 | . 58 | .64 | .66 |
| P | - | - | .003 | .001 |
| s | .006 | .007 | .006 | .006 |
| Si | - | - | .03 | .04 |
| W | - | - | <.04 | <.04 |
| Y | 1.45 | 1.47 | 1.48 | 1.46 |

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TABLE XVII

MECHANICAL PROPERTIES OF FULL SCALE EXTRUSIONS

ALL MATERIALS IN THE FAST HEAT TREATED (2450°F) CONDITION

| Material | Location | 2000°F/10 ksi Stress Rupture Life (hrs.) | F Mod | Room Temperature dulus of Elasticity (ksi x 10 ⁶) | Structure Type |
|----------|----------|---|----------|---|-------------------|
| 76003 | Center | 0.9 | | 31.0 | 2 |
| | Edge | 0.4 | | 28.1 | 2 |
| | | | | | |
| 76004 | Center | 0.7 | | 31.7 | 2 |
| | Edge | 3.8 | | 27.9 | 2 |
| | | | | | |
| 76005 | Center | 18.3 | | 30.2 | 1 |
| | Edge | 3.1 | | 27.3 | 1 |
| | | | | | |
| 76006 | Center | 56.5 | | 30.2 | 1 |
| | Edge | 19.9 | | 26.4 | 1 |
| | | | | | |

TABLE XVIII

TENSILE PROPERTIES OF O.D.S. CO-76005 AND O.D.S. CO-006 AS A FUNCTION OF TEMPERATURE

(ALL MATERIAL IN THE FAST HEAT TREATED CONDITION)

| Extrusion No. | Location | Test Temp. (°F) | 0.2% Yield (ksi) | U.T.S. (ksi) | Elong. (%) | R.A% |
|------------------|----------|-----------------------|---------------------|-----------------|---------------|------|
| 76005 | C | RT | 103.8 | 154.5 | 11.4 | 10.4 |
| | Е | RT | 101.0 | 150.2 | 10.6 | 13.3 |
| 76006 | С | RT | 105.6 | 162.2 | 12.3 | 9.5 |
| | Е | RT | 98.8 | 153.6 | 12.4 | 12.6 |
| 76005 | C | 1800 | 17.5 | 21.6 | 2.9 | 4.3 |
| | E | 1800 | 17.3 | 22.6 | 6.1 | 12.6 |
| 76006 | С | 1800 | 19.3 | 25.7 | 11.5 | 15.5 |
| | Е | 1800 | 16.5 | 27.9 | 9.4 | 14.4 |
| 76005 | С | 2200 | 13.6 | 13.6 | 2.1 | 1.6 |
| | Е | 2200 | 12.8 | (a) | 1.6 | 1.3 |
| 76006 | с | 2200 | 13.4 | 13.4 | 2.1 | 1.6 |
| | E | 2200 | 12.4 | 12.4 | 1.8 | 3.2 |
| 76005 | с | 2400 | 10.5 | 10.5 | 1.6 | 1.3 |
| | E | 2400 | 8.0 | (a) | 1.0 | 0.7 |
| 76000 | с | 2400 | 10.7 | 10.7 | 1.3 | 0.4 |
| | E | 2400 | 9.7 | 9.7 | 1.4 | 1.6 |

(a) Failed before 0.2% Y/S was obtained.

C = Center

Contraction of the second second second

E = Edge

TABLE XIX

$\frac{\text{STRESS RUPTURE PROPERTIES OF 0.D.S. CO-76005 AND}{76006 \text{ at } 2200^\circ \text{F} \text{ and } 2400^\circ \text{F}}$

(ALL MATERIAL IN THE FAST HEAT TREATED CONDITION)

| Extrusion No. | Location | Test Temp. (°F) | Stress (ksi) | Life (hrs.) | Elong. (%) | R.A. (%) |
|------------------|----------|-----------------------|-----------------|----------------|---------------|-------------|
| 76005 | Е | 2200 | 8 | 1.4 | 1.7 | 2.2 |
| | Е | 2200 | 7 | 5.3 | 0.6 | 0.7 |
| | С | 2200 | 7 | 87.8 | 1.2 | (a) |
| 76006 | E | 2200 | 7 | 28.4 | 1.6 | 0.4 |
| | E | 2200 | 7 | 4.5 (b) | 0.3 | 1.1 |
| | C | 2200 | 7 | 81.5 | 0.8 | 0.3 |
| 76005 | E | 2400 | 4 | 15.5 | 1.6 | 1.8 |
| | E | 2400 | • 4 | 8.7 | 1.1 | (a) |
| | С | 2400 | 4 | 0.5 | 2.9 | 0.7 |
| 76006 | Е | 2400 | 4 | 72.5 | 2.6 | (a) |
| | Е | 2400 | 4 | 172.6 | 1.9 | (a) |
| | C | 2400 | 4 | 71.5 | 1.3 | nil |
| | | | | | | |

(a) Failed near radius

(b) Timer failed to shut off - time taken from temperature recorder

E = edge

C = center

19 C .

TABLE XX

NOMINAL COMPOSITIONS OF ALLOYS EVALUATED IN THE DYNAMIC OXIDATION TESTS

| | 0.D.S. Co | H.D.A. 8077 | <u>M.A. 953</u> | <u>H.A. 188</u> | HASTELLOY X |
|----|--------------|------------------|-----------------|-----------------|-------------|
| A1 | 5 | 4 | 5 | - | - |
| С | .03 | .08 | .04 | 0.1 | 0.1 |
| Co | 54 | - | - | .40 | 1 |
| Cr | 18 | 16 | 19 | 22 | 22 |
| Fe | 0.4 | 0.5 | 35 | <3 | 17.5 |
| La | 1.0 <u> </u> | - A | 0.25 | 0.05 | - |
| Mn | 8-9 - | - ¹ | -944 | <1.25 | <1 |
| Мо | 8.1 - | 695 - 195 | -0004 | - 4 | 9 |
| Ni | 20 | 78 | -0044 | 22 | - |
| 0 | 0.6 | 0.5 | 0.5 | - | - |
| Si | - | - | - | 0.35 | <1 |
| Ti | - | | 0045 | - | - |
| W | - | - | 2400 | 14.5 | 0.5 |
| Y | 1.45 | 1.0 | - | - | - |
| | | | | | |

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TABLE XXI

2100°F DYNAMIC OXIDATION TEST RESULTS

| | | | Weigh | t Loss | | | Continuous | Total |
|----|-----------------------------|------------------------|-----------------------|-----------------------|-----------------------|-------------|-------------|----------------|
| | | 20 hrs. | 40 hrs. | 60 hrs. | 100 hrs. | Metal Loss | Penetration | Metal Affected |
| | Alloy | $(\overline{mg/cm}^2)$ | (mg/cm ²) | (mg/cm ²) | (mg/cm ²) | (mils/side) | (mils/side) | (mils/side) |
| | 76005 | .417 | 2.51 | 3.60 | 4.37 | 6. | .43 ± .23 | 1.33 |
| | 76005 | .301 | 2.18 | 3.16 | 4.89 | | .34 ± .11 | 1.04 |
| | 76006 | .241 | 3.17 | 4.55 | 6.56 | .7 | .45 ± .09 | 1.15 |
| | 76006 | .669 | 3.20 | 4.51 | 6.29 | .5 | .50 ± .17 | 1.0 |
| | 8077(Lot 67) | 1.96 | 3.6 | 4.24 | 5.15 | 1.05 | .18 ± .06 | 1.23 |
| | 8077(Lot 57) | 2.34 | 3.19 | 3.56 | 4.25 | .65 | .15 ± .07 | 0.80* |
| 33 | MA 953E | .343 | .703 | 668. | 1.64 | 1.0 | .76 ± .11 | 1.26* |
| | HA 188 (Lot 4-1668) | .45 | 1.79 | 3.97 | 9.11 | .40 | 2.58 ± .27 | 2.98 |
| | HA 188 (Lot 3-1637) | .785 | 2.68 | 7.23 | 48.6 | 1.2 | 1.63 ± .87 | 5.33 |
| | HASTALLOY X (Lot 5-5108) | 1.01 | 52.2 | 83.6 | 152.6 | 7.5 | 2.05 ± .82 | 11.9 |
| | | | | | | | | |

* Some Kirkendall Porosity

103.9 M

CELER.

TABLE XXII 1650°F HOT CORROSION TEST RESULTS

Metal Affected (mils/side) 1.99 3.35 3.85 7.83 5.00 7.15 2.62 1.95 3.12 3.27 Total Penetration (mils/side) Continuous .67 ± .29 .77 ± .16 .66 ± .27 .65 ± .13 1.07 ± .19 1.05 ± .49 1.37 ± .44 1.79 ± .47 .64 ± .29 .60 ± .16 Metal Loss (mils/side) .55 1.75 4.65 4.75 .45 1.15 1.25 1.5 1.2 ~ mg after 200 hrs. + .0007 + .0044 + .0024 + .0039 + .0153 - .5609 - .2836 - .3281 + .0002 - .0551 mg after 132 hrs. + .0162 + .0163 + .0161 + .0138 - .1155 - .9473 - .0553 + .0052 e0000. + + .0501 Weight Loss mg after 84 hrs. 1100. + + .0052 + .0051 + .0032 + .0041 + .0016 + .0030 0000. + + .0014 - .0034 mg after 43 hrs. + .0052 + .0052 + .0045 + .0043 + .0022 + .0030 - .0010 + .0018 + .0034 + .0027 (Lot 4-1668) (Lot 3-1637) HASTALLOY X (Lot 5-5108) 8077(Lot 67) 8077(Lot 57) MA 953 E Alloy HA 188 HA 188 76005 76005 76006 76006

34

- 4 12000

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TABLE XXIII

COEFFICIENT OF THERMAL EXPANSION OF O.D.S. CO-76006 IN LONGITUDINAL DIRECTION FOR TEMPERATURES UP TO 2000°F

| Temperature | Expansion | |
|-------------|-------------------|------------------------|
| (°F) | (inches per inch) | (Inches per inch/°F |
| 200 | 0.0011911 | 9.02×10^{-6} |
| 400 | 0.0028311 | 8.53×10^{-6} |
| 600 | 0.0045884 | 8.62×10^{-6} |
| 800 | 0.0061524 | 8.40×10^{-6} |
| 1000 | 0.0079976 | 8.58×10^{-6} |
| 1200 | 0.0101084 | 9.00×10^{-6} |
| 1400 | 0.0134080 | 10.07×10^{-6} |
| 1600 | 0.015739 | 10.26×10^{-6} |
| 1800 | 0.0181579 | 10.48×10^{-6} |
| 2000 * | 0.0211320 | 10.94×10^{-6} |

* Extrapolated





Mag. = 100X



Fig. 3 AT 225C given fast (upper) and slow (lower) heat treatments





Neg. 37551

Mag. = 100X

Type 2

Structure

Neg. 242-74

Mag. = 100X



Type 3 Structure

Type 3 Structure

Neg. 37553

the second s

Mag = 100X

27 2 400

Fig. 5 AT 230C given fast (upper) and slow (lower) heat treatment.





Neg. 37549

the state of the second st

Mag. = 100X



Type 3 Structure

Neg. 37550









Neg. 36094

Mag. = 100X



Type 2 Structure

Neg. 36079

Mag. = 100X





Type 2 Structure

Neg. 37566

Mag. = 100X



Type 1 Structure

Neg. 37567

Mag. = 100X



Fig. 9 Definitions of attack in oxidation and corrosion testing.

1. Metal Loss (mils/side), [(A-B)/2]

2. Continuous Penetration (mils/side),

3. Maximum Penetration (mils/side), [D]

4. Total Metal Affected (mils/side), ((A-B)/2) + D

[c]

Schematic Of Metallographic Measurement Technique

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