MAY 12 1967

MASTER 29

OAK RIDGE NATIONAL LABORATORY
operated by

UNION CARBIDE CORPORATION
NUCLEAR DIVISION

for the
U.S. ATOMIC ENERGY COMMISSION

ORNL - TM - 1802

MECHANICAL PROPERTIES OF $\text{Al-Al}_2\text{O}_3$ ALLOYS:

TESTING PROCEDURES AND EVALUATION OF DATA

D. G. Harman

THIS DOCUMENT HAS BEEN REVIEWED.
NO INVENTIONS OF PATENT INTEREST
TO THE A.E.C. ARE DISCLOSED THEREIN.

2835/12/67

NOTICE This document contains information of a preliminary nature and was prepared primarily for internal use at the Oak Ridge National Laboratory. It is subject to revision or correction and therefore does not represent a final report.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

— LEGAL NOTICE —

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

- A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or
- B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

ORNL-TM-1802

CESTI PRICES

Contract No. W-7405-eng-26

METALS AND CERAMICS DIVISION

H.C. \$ 3.00; MN. 65

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

MECHANICAL PROPERTIES OF $\text{Al-Al}_2\text{O}_3$ ALLOYS: TESTING PROCEDURES AND EVALUATION OF DATA

D. G. Harman

MAY 1967

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
operated by
UNION CARBIDE CORPORATION
for the
U.S. ATOMIC ENERGY COMMISSION

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

CONTENTS

	Page
Abstract	1
Introduction	1
Strain Rate Dependence	2
Fractographic Analysis	8
Summary	11
Acknowledgement	11

MECHANICAL PROPERTIES OF Al-Al₂O₃ ALLOYS:
TESTING PROCEDURES AND EVALUATION OF DATA

D. G. Harman

ABSTRACT

Techniques have been developed to evaluate the elevated-temperature mechanical properties of Al-Al₂O₃ (SAP-type) alloys. Varying the strain rate over two orders of magnitude has explained much of the reported scatter in mechanical property data and provides data for creep-rupture predictions. The strain rate, $\dot{\epsilon}$, in SAP is an exponential function of the stress σ : $\dot{\epsilon} = A \sigma^n$ with n being very high. This high n value is correlated with short-time tensile and creep-rupture behavior. Fractographic analysis shows ductile fracture under all testing conditions.

INTRODUCTION

Our mechanical properties investigations regarding the SAP program began in July 1965. We have under way general deformation studies on both commercial and experimental materials as well as evaluations of the effects of certain material and process parameters. Because of the relative importance of the fracture mechanics of SAP materials, we are also pursuing extensive fractographic analysis of failed test specimens. A meaningful mechanical property evaluation of SAP materials, because of their unique behavior, requires information gained through all of these studies. The inadequacy of any superficial mechanical property study is pointed out in this report.

Experimental materials with oxide contents ranging from 1 to 10% have been tested as well as commercial SAP alloys. High-temperature properties were found to be sensitive to changes in temperature or strain rate. High temperature-low strain rate ductility has ranged from less than 1% total elongation for the higher oxide contents to as high as 17% for the lower oxide contents.

STRAIN RATE DEPENDENCE

At high temperatures SAP type materials exhibit pronounced strain-rate dependence of mechanical properties. Much of the relatively large scatter in reported SAP data can be attributed to differences in testing conditions. Figure 1 shows our data on commercial XAP-001 superimposed on a scatter band obtained from the literature.¹⁻⁹ The change in strain rate from 0.2 to 0.002 min⁻¹ has a large effect on high-temperature properties and covers a good portion of the scatter band. A strain-rate dependence is not unique to SAP but is significant at certain temperatures for many alloy systems, including austenitic stainless steels and nickel-base superalloys.

¹E. A. Bloch, "Dispersion-Strengthened Aluminum Alloys," Met. Rev. 6(22), 193-239 (1961).

²R. J. Towner, Alcoa's APM Alloys, ALCOA Research Laboratories, New Kensington, Pennsylvania, Sept. 15, 1960.

³E. G. Kendall and W. H. Friske, "Fabrication and Properties of APM Products," Met. Soc. Am. Inst. Mining, Met. Petrol. Engrs., Inst. Metals Div., Spcc. Rept. Ser. 10, 39-45 (1960).

⁴D. G. Boxall and S. Mocarski, The Present Status of SAP in the Canadian Organic Cooled Reactor Program, R61CAP30, Canadian General Electric (June 1961) (limited distribution).

⁵D. M. Guy, Alcoa's Aluminum Powder Metallurgy (APM) Alloys, Alcoa Green Letter, ALCOA Research Laboratories, New Kensington, Pennsylvania, March 1959 (not released for publication).

⁶W. H. Friske, Interior Report on the Aluminum Powder Metallurgy Product Development Program, NAA-SR-4233 (1960).

⁷N. Hansen, Tensile Properties at Room Temperature and at 400°C of Commercial Sintered Aluminum Products, Risö-96 (December 1964).

⁸Atomics International, SAP Materials Handbook, AI-CE-Memo-24 (March 23, 1966).

⁹D. G. Boxall and J. W. Standish, Mechanical Properties of Dispersion-Strengthened Aluminum Alloys, AECL-1532 (January 1962).

Fig. 1. Short-Time Tensile Strength and Ductility of XAP-001. Extruded rod, 0.40 in. in diameter, tested parallel to its extrusion direction. Hatched area represents large scatter in reported SAP data. Refs. 1-9.

The strain rate, $\dot{\epsilon}$, in SAP is an exponential function of the stress σ : $\dot{\epsilon} = A \sigma^n$. This relationship gives a straight line with slope n on log-log coordinates, as shown in Fig. 2. The filled symbols represent 450°C data from an experimental 5% oxide material; the filled triangles are tensile data at three strain rates and the filled circles are from creep data. The open circles are Battelle's¹⁰ XAP-001 (previously called M-257) data at 427°C. The straight line has a slope of 40

¹⁰J. A. Van Echo and S. W. Porembka, Long-Term Creep Rupture of Sintered Aluminum Powder and Zircaloy-2 Alloys, BMI-X-10 113 (Jan. 25, 1965).

Fig. 2. Stress vs Creep Rate for Two Sintered Aluminum Products.

($n = 40$). This value of n is very high compared to values of 4 to 6 usually found for more conventional alloys; TD nickel, which has a structure similar to SAP, has also shown this high n value.¹¹

This very high value for n is significant in both the tensile and creep situations. Any small amount of area change during a tensile test, by internal void formation or tensile necking, gives rise to locally increasing strain rates, which result in decreased total elongation. Figure 3 shows the local neck observed for a high-temperature tensile test.

¹¹L. P. Rice, Metallurgy and Properties of Thoria-Strengthened Nickel, DMIC-210 (Oct. 1, 1965).

Fig. 3. Interrupted 450°C Tensile Test on XAP-001 Showing Local Necking Just Prior to Rupture. Specimen was electropolished before test.

A high n value can affect the creep test by the acceleration of third-stage creep; SAP material with its low work hardening at elevated temperatures is particularly susceptible to this affect. At the termination of second-stage or linear creep, some slight reduction in cross-sectional area has occurred either through actual necking or more likely through internal void formation. Because the load on the specimen is constant, this reduction in area increases the stress level. A small increase in stress provides a significant increase in strain rate, which results in an immediate rupture via high-strain-rate shear. This type of rupture shown in Fig. 4 exhibits essentially no necking on the portion that failed by the growing and linking of the internal voids. This fracture process is commonly called ductile tearing. Considerable necking is seen on the portion that failed by the high-strain-rate shear.

Fig. 4. Creep Rupture of XAP-001 at 450°C Showing Local Necking at Shear Lip. No neck is evident on that portion that failed by tearing.

Consideration of the low ductility and sensitivity to changes in strain rate has led to a procedure for the evaluation of any particular SAP rod with a given powder and fabrication history. Because of the limited amount of material available per SAP rod, a small button-head specimen design is being used that has been successful for other laboratory programs involving materials with low ductility. Each specimen requires only 2 in. of rod length and has a 1/8-in.-diam $\times$ 1 1/8-in.-long gage length. The specimen design is shown in Fig. 5.

Fig. 5. Tensile Specimen.

Special precautions are taken to ensure a constant test temperature along the entire test specimen during the tensile test. This is necessary because of the temperature dependence of the properties of SAP and because of its overall low ductility. Chromel-P-Alumel thermocouple wires are resistance welded to both specimen grips and are located very close to the specimen gage length. These thermocouples are used for a two-zone temperature control system utilizing separate power controllers coupled with speedomax H temperature recorders. In addition both temperatures are monitored manually using a potentiometer to ensure that both grips are at the same temperature. The tensile test setup shown in Fig. 6 (thermocouples are not shown) has been used to ensure consistent test results from specimens that have ruptured in the center portion of the gage length.

The creep testing setup differs only in that the pull rod guides are not used and a shunt wound furnace is used with a single power controller and temperature recorder. The temperature of both grips is monitored at regular intervals and the furnace is properly shunted to correct any difference.

At least four tensile tests are performed on each rod: one tensile test at room temperature for base-line comparison and at least three tensile tests at 450°C covering a wide range of strain rates. This strain-rate variation not only provides good tensile data but also

Y-52144

Fig. 6. Pull Rod Setup for Elevated Temperature Tensile Testing of SAP.

allows one to extrapolate to obtain expected long-time stress-rupture values. The treatment of tensile data is shown in Fig. 7. Plotted on log-log coordinates is the applied stress vs the time to rupture.

Fig. 7. Stress Rupture of SAP 37 (MD-3100) at 450°C.

This particular stress-rupture plot is for an experimental SAP product produced from a commercially milled flake powder (Metals Disintegrating Grade MD-3100) with about 6% oxide. The open circles are the ultimate tensile strength plotted against the total tensile test time, while the filled circles are dead-load stress-rupture tests. One can see the good correlation obtained by the extension of the tensile data. This treatment of tensile data is invaluable in determining the stress levels for loading stress rupture specimens for reasonably predictable rupture times.

FRAC TOGRAPHIC ANALYSIS

Another very important aspect of SAP behavior is the mode of fracture. A most significant finding has been that under all testing conditions, including high-temperature creep, SAP fractures in a ductile manner. This ductility is confined to a small portion of the specimen and thus provides very little overall extension.

Ductile fracture is generally categorized as separation, tearing, or shear. All three of these modes have been found in SAP. Figure 8 shows typical low- and high-temperature fractographs. The fracture surfaces of the specimens were replicated using the two-stage carbon technique and subsequently viewed and photographed in the electron microscope.

Fig. 8. Fracture of Experimental SAP. (a) Room-temperature shear. 6500 $\times$. (b) Room-temperature tearing. 6500 $\times$. (c) 450 $^{\circ}$ C shear. 8000 $\times$. (d) 450 $^{\circ}$ C tearing. 6500 $\times$. Fractography by T. A. Nolan, Physics Department, ORGDP.

The very fine scale, low-temperature fractures are in contrast to the coarser high-temperature fractures. We now have very good evidence that these high-temperature voids, which initiate fracture, form very early in the tensile test at strains as low as 0.2% and that fracture initiates at the Al-Al₂O₃ boundary. The room temperature shear fractographs are being used routinely to show the oxide distribution within the material. Another technique being used is to anodize the fracture surface for direct replication. Figure 9 shows such a replica for a high-temperature fracture. The platelets of oxide can be seen over the entire surface and are now available for further study, including diffraction analysis and stereophotography.

Fig. 9. Direct Replication of Anodized Fracture Surface of Experimental SAP Tested at High Temperature. Black needle-shaped particles are oxide platelets viewed on edge. Fractography by T. A. Nolan, Physics Department, ORGDP. 6500x.

SUMMARY

The alloy Al-Al₂O₃ is a unique material and requires special considerations in obtaining meaningful mechanical properties data. An understanding of the general deformation behavior is essential. Significant scatter in the data results if certain precautions are not taken during testing; the most significant of which are the strain rate and specimen temperature. The so-called constant load rate tensile tests are not appropriate because of the strain rate effect. The strain rate-stress relationship for SAP can be expressed as $\dot{\epsilon} = A\sigma^n$ where n is about 40. Good correlation is obtained between constant strain rate tensile data and dead-load creep-rupture results.

Fracture of SAP initiates early in the tensile test at internal locations and propagates in a classically ductile manner. However, this ductility is confined very local to the fracture and provides little overall extension.

ACKNOWLEDGMENT

The author is indebted to T. A. Nolan, Physics Department, ORGDP, for the excellent fractographic analysis that he has performed and his technical advice relative to this research. Also acknowledged is the creep and tensile testing performed by C. W. Walker, C. W. Dollins, B. C. Williams and F. L. Beeler and the technical discussions of C. R. Kennedy during the course of this work. Appreciation is extended to E. E. Bloom and C. R. Kennedy for their review of this report.

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

INTERNAL DISTRIBUTION

1-3. Central Research Library	54. H. G. MacPherson
4-5. ORNL - Y-12 Technical Library	55. R. E. MacPherson
Document Reference Section	56. W. R. Martin
6-15. Laboratory Records Department	57. R. W. McClung
16. Laboratory Records, ORNL R.C.	58. H. E. McCoy, Jr.
17. ORNL Patent Office	59. H. C. McCurdy
18. G. M. Adamson, Jr.	60. D. L. McElroy
19. J. H. Barrett	61. C. J. McHargue
20. S. E. Beall	62. F. R. McQuilkin
21. M. Bender	63. A. J. Miller
22. R. G. Berggren	64. E. C. Miller
23. G. E. Boyd	65. F. H. Neill
24. W. H. Bridges	66. S. M. Ohr
25. W. E. Brundage	67. P. Patriarca
26. F. L. Culler	68. A. M. Perry
27. J. E. Cunningham	69. R. E. Reed
28. J. H. DeVan	70. P. L. Rittenhouse
29. K. Farrell	71. M. W. Rosenthal
30. D. E. Ferguson	72. G. Samuels
31. A. P. Fraas	73. J. L. Scott
32. J. H. Frye, Jr.	74. O. Sisman
33. W. R. Grimes	75. G. M. Slaughter
34. J. P. Hammond	76. I. Spiewak
35-39. D. G. Harman	77. J. T. Stanley
40. W. O. Harms	78. W. J. Stelzman
41-43. M. R. Hill	79. J. O. Stiegler
44. N. E. Hinkle	80. D. B. Trauger
45. H. W. Hoffman	81. R. P. Tucker
46. W. R. Huntley	82. J. T. Venard
47. H. Inouye	83. G. M. Watson
48. G. W. Keilholtz	84. A. M. Weinberg
49. B. C. Kelley	85. J. R. Weir, Jr.
50. C. F. Leitten, Jr.	86. G. D. Whitman
51. A. P. Litman	87. J. M. Williams
52. A. L. Lotts	88. F. W. Young, Jr.
53. R. N. Lyon	

EXTERNAL DISTRIBUTION

- 89-90. F. W. Albaugh, Assoc. Director, PNL
- 91. A. J. Alexander, RDT, OSR, AEC, Combustion Engineering,
Windsor, Conn.
- 92-93. D. G. Boyer, DIA, USAEC Scientific Representative, Whiteshell
Nuclear Research Establishment, AECL, Pinawa, Manitoba, Canada
- 94. Chief, Control Mechanisms Branch, AEC, Washington
- 95. D. F. Cope, RDT, OSR, AEC, Oak Ridge National Laboratory
- 96. M. W. Croft, HWOOR Program Mgr., Babcock and Wilcox Company
- 97. P. G. Holstead, RDT, OSR, PNL, Richland, Washington
- 98-100. M. N. Hudson, DIA, USAEC Scientific Representative, Chalk
River, Ontario, Canada
- 101. T. J. Iltis, DIA, Brussels for EURATOM, Division of International
Affairs, AEC, Washington
- 102-103. E. A. Kintner, Chief, Fuel Fabrication Branch, AEC, Washington
- 104. J. E. Konklin, Idaho Reactor Division, Idaho Nuclear Corp.
- 105. J. H. Kruth, RDT, OSR, AEC, Aiken, S. C.
- 106. W. J. Larkin, AEC, Oak Ridge Operations
- 107. R. L. Morgan, RDT, SSR, AEC, AI-CE, Canoga Park
- 108. S. W. O'Rear, Supervisor, Technical Information Service,
Du Pont, SRL
- 109-111. J. M. Simmons, Chief, Fuels and Materials Branch, AEC,
Washington
- 112. E. E. Sinclair, Asst. Director, Reactor Technology, AEC,
Washington
- 113. Franklin Standerfer, Chief, Applications and Facilities Branch,
AEC, Washington
- 114-119. C. L. Storrs, Director, HPO (AI-CE), Canoga Park
- 120-121. A. N. Tardiff, Chief, HWOOR Branch, AEC, Washington
- 122. K. A. Trickett, Chief, Core Design Branch, AEC, Washington
- 123-125. R. P. Varnes, HPO Representative, Combustion Engineering,
Windsor, Conn.
- 126. M. J. Whitman, Asst. Director, Program Analysis, AEC, Washington
- 127. E. A. Wright, AEC, Washington
- 128. Division of Research and Development, AEC, Oak Ridge Operations
- 129-143. Division of Technical Information Extension