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TID-25816

RESEARCH REPORT

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TOPICAL REPORT

on

TID-25816

LMFBR INSTRUMENTATION - STRAIN GAGES
PHASE I - HIGH-TEMPERATURE STRAIN
GAGE DEVELOPMENT PROGRAM

LMEC Contract No. N083-0004
TASK 1 - LITERATURE AND FIELD SURVEY

MASTER

to

RECEIVED BY DTIE OCT 13 1971

LIQUID METAL ENGINEERING CENTER

November 13, 1970

by

D. C. Drennen, C. M. Jackson, Nelson Crites
and J. E. Sorenson

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November 13, 1970

Mr. Gordon Twa
Contract Project Engineer
Liquid Metal Engineering Center
P. O. Box 1449
Canoga Park, California 91304

Dear Mr. Twa:

LMEC Contract No. N083-0004

Enclosed is the topical report covering the work carried out
on Task 1 - Literature and Field Survey of the subject contract.

Sincerely,

J. E. Sorenson
Project Engineer

JES:ph

Enc.

Liquid Metal Engineering Center

*Operated for
the U.S. Atomic Energy Commission by Atomics International*

A Division of North American Rockwell Corporation

October 19, 1970

*In reply refer
70LMEC-1699*

Mr. J. E. Sorenson
Battelle Memorial Institute
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

Subject: High Temperature Strain Gage Development Program (LMEC Contract No. N083-0004), Topical Report, Task 1 - Literature and Field Survey

Dear Mr. Sorenson:

The subject report has been reviewed. It is an excellent report, only minor editorial errors are commented on in the attachment to this letter.

Please incorporate these comments and distribute the report as required.

Sincerely yours,

Gordon J. Twa
Project Engineer

Enclosure
Topical Report, Task 1 -
Literature and Field Survey

cc w/encl: Director, RDT, HQ
Asst. Director, P.E., RDT, HQ
Chief, Instrumentation & Control Br., RDT, HQ
Manager, SAN
RDT Senior Site Representative (AI) (2)
Director, LMFBR Program Office, ANL
Contract Representative, CP-AEC

HIGH TEMPERATURE STRAIN GAGE DEVELOPMENT PROGRAM

(LMEC CONTRACT # N083-0004)

Comments on Task I Report

1. All charts should be made full size.
2. Page 12 - last sentence of 1st paragraph - " Cr_2O_3 -inch scale," is this correct?
3. Page 12 - third paragraph - 1st sentence - what does the 15 to 30 percent refer to?
4. Page 15 - line 10 - "cycles" should be "cycled."
5. Page 15 - last sentence on page - should read ". . . expected also to depend."
6. Page 19 - second to last sentence - typo error "ingestigations" should be "investigations."
7. Page 65 - second paragraph, 3rd sentence - "are" should be "or".
8. Page 67 - the Microdot SG-423 gage is no longer manufactured and in its place should be a discussion of the SG-425 which has corrected many of the faults described here.
9. Page 67 - last complete sentence of this page - the gage and shim assembly is removed from the calibration bar.
10. Page 69 - the unusually low drift rates noted for the LMEC gage were determined only after a stabilization period of 16 hours at 1200°F. This should be noted so as not to be misleading.
11. Page 71 - no mention is made of ultrasonic bonding techniques - refer to Agreements and Commitments from June 24 - 25 meeting, Item 1.
12. Page 58 - Reference 1, date of publication should be noted and is the publication number correct?

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TOPICAL REPORT

on

LMFBR INSTRUMENTATION - STRAIN GAGES PHASE I - HIGH-TEMPERATURE STRAIN GAGE DEVELOPMENT PROGRAM

LMEC Contract No. N083-0004
TASK 1 - LITERATURE AND FIELD SURVEY

to

LIQUID METAL ENGINEERING CENTER

November 13, 1970

INTRODUCTION

The objective of Task 1 - Literature and Field Survey was to obtain information on high-temperature strain gages, high-temperature lead wire, strain gage alloys and Fe-Cr-Al-X alloys (where X represents one or more other alloying elements).

The literature search covered the period 1957 to 1970 and pertinent articles from the following sources were obtained:

1. United States Atomic Energy Commission
Division of Technical Information Extension
Machine Search of Nuclear Science Abstracts (NSA)
2. NASA Scientific and Technical Information Facility
Machine Search (NASA Literature Search Number 11705)
3. Defense Documentation Center Defense Supply Center
Cameron Station, Alexandria, Virginia 22314
Machine Search (Search Contract No. 037361)
4. Defense Metals Information Center
(DMIC) - Battelle-Columbus
5. ASM Review of Metal Literature--Battelle Columbus Library
6. ASM Metals Abstract--Battelle-Columbus Library
7. Engineering Index--Battelle Columbus Library
8. Instruments Abstracts--Battelle-Columbus Library.

The initial search of the above sources uncovered well over 700 references. The search was carried out in two separate parts.

One dealt specifically with strain gage and Fe-Cr-Al-X alloys, while the other part covered high-temperature strain gage and lead-wire research.

In an attempt to obtain more recent data and possibly unpublished information, visits were made to the following organizations that have an interest in high-temperature strain gage development:

- (1) General Electric Company, Aircraft Engine Group
Evendale, Ohio
- (2) Wright Patterson Air Force Base, Dayton, Ohio
- (3) Lewis Research Center, National Aeronautics and Space
Administration, Cleveland, Ohio.

In addition, telephone conversations were held with the following men who have carried out research on high-temperature strain gages, strain gage alloy wires or Fe-Cr-Al-X wires:

- (1) R. Bertodo, Perkins Engines Company, Peterborough, England
- (2) R. Friedman, Aerostructures Department, Naval Air Development Center, Warminster, Pennsylvania
- (3) W. H. Graft, LaSalle Steel Company, Hammond, Indiana
- (4) C. Gross, Langley Research Center, National Aeronautics and Space Administration, Hampton, Virginia
- (5) C. Guettel, Driver-Harris Company, Harrison, New Jersey
- (6) Dr. C. Raynor, Present affiliation unknown (contacted at home), Ann Arbor, Michigan
- (7) G. Steven, Crucible Steel Company, Pittsburgh, Pennsylvania.

With regard to the alloys, the following types of information were sought:

- (1) Drawability to 0.001-inch-diameter wire
- (2) Resistances to oxidation at temperatures to 1200 F
- (3) Metallurgical structure from room temperatures to 1200 F
- (4) Electrical resistivity and temperature coefficient of resistance (TC) at temperatures to 1200 F
- (5) Stability of the metallurgical structure, electrical resistivity and temperature coefficient during thermal cycling from room temperature to 1200 F.

Abstracts of pertinent articles were reviewed and copies of those articles thought most applicable to this study were obtained for a detailed review. A total of 33 references on high temperature strain

gages and 56 references on alloys are cited in this report. A greater number of references were reviewed in detail in the preparation of this report but were determined to be not applicable to this study.

This report is divided into two major portions. The first deals entirely with alloys for high temperature strain gages while the second covers high-temperature strain gages, lead wires, lead wire attachment and insulation and bonding techniques.

IDENTIFICATION AND CHARACTERISTICS OF ALLOYS
FOR HIGH-TEMPERATURE STRAIN GAGES

Alloys for High-Temperature Strain Gages

Tables 1 and 2 list the compositions of alloys that either have been evaluated for use in high-temperature strain gages or possess certain properties that make them candidates for use in high-temperature strain gages. In order to be usable in a high-temperature strain gage, an alloy should exhibit (1) good oxidation resistance at temperatures at which it would be used, (2) metallurgical and structural stability at service temperatures, (3) good fabricability to wire or foil, (4) adequate mechanical strength, (5) high electrical resistivity, (6) the same or nearly the same coefficient of thermal expansion as the material to which it is applied, (7) little variation in gage factor with temperature and strain, (8) a minimum amount of apparent strain with temperature, and (9) a constant or linear temperature coefficient of resistance. It is further recognized that no alloy is presently known that will exhibit all of the above properties at temperatures up to 1200 F.

The following sections discuss some of the important (for high-temperature strain-gage applications) properties of the various types of alloys listed in Tables 1 and 2. Because of the instability caused by the order-disorder and precipitation reactions that occur in nickel-chromium and nickel-chromium-base alloys at temperatures between room temperature and 1200 F, detailed data on these alloys (two of which are listed in Table 1) were not compiled.

CHARACTERISTICS OF ALLOYS FOR
HIGH-TEMPERATURE STRAIN GAGES

Oxidation Resistance

Fe-Cr-Base Alloys

As indicated in Tables 1 and 2, the Fe-Cr-base alloys listed would be expected to exhibit good oxidation resistance at 1200 F (650 F).

TABLE 1. CANDIDATE IRON-, NICKEL-, AND COBALT-BASED ALLOYS FOR HIGH-TEMPERATURE STRAIN GAGES

Alloy Designation	Nominal Chemical Composition, w/o	Electrical Resistivity at Room Temperature, Ω/cmf	Temperature Coefficient of Resistance (0 to 1000 C), $\mu\Omega/\Omega\text{ C}$	Approximate Gage Factor at Room Temperature	Probable Potential for Fabrication to Fine Wire	Probable Oxidation Resistance at 1200 F (650 C)	Coefficient of Thermal Expansion (0-1000 C), $\mu\text{in./in. C}$	Ultimate Tensile Strength at Room Temperature, psi	Reference
Armour A ⁽¹⁾	Fe-25.18Cr-5.0Al-0.38Th	775	+ 127	3.3	Good	Good	14.1	--	1, 2, 3
Armour B ⁽¹⁾	Fe-24.89Cr-7.84Al	1005	- 61	2-2.8	Good	Good	15.8	--	1, 2, 3
Armour C ⁽¹⁾	Fe-24.7Cr-13.0Al	1380	-226	1.8	Poor	Good	21.2	--	1, 2, 3
Armour D ⁽¹⁾	Fe-20.5Cr-10.5Al	1059	-110	1.8-2	Good	Good	16.6	--	1, 2, 3
Armour E ⁽¹⁾	Fe-35.6Cr-6.5Al	--	--	--	Poor	Good	--	--	2
Armour F ⁽¹⁾	Fe-50Cr	360	--	--	Good	Good	--	--	2
Armour G ⁽¹⁾	Fe-40Cr	324	--	--	Good	Good	--	--	2
Armour H ⁽¹⁾	Fe-28.35Cr-10.35Al	--	--	--	Poor	Good	--	--	2
Armour I ⁽¹⁾	Fe-10.01Cr-17.75Al	--	--	--	Poor	Good	--	--	2
Armour J ⁽¹⁾	Ni-12.2Cr-2.03Al	--	--	--	Good	Good	--	--	2
Armour K ⁽¹⁾	Co-27.18Cr-6.26Al	--	--	--	Poor	Good	--	--	2
Karma ⁽²⁾	Ni-20Cr-3.0Al-3.0Fe	800	$\pm 20^{(3)}$	2.0	Good	Good	18.0	130-180,000	2, 4, 5, 6
Nichrome V ⁽²⁾	80Ni-20Cr	650	+110 ⁽⁴⁾	2.0	Good	Good	17.6	100-200,000	2, 4, 6
GE-2541 ⁽⁵⁾	Fe-25Cr-4Al-1Y	--	--	--	Poor	Good	--	79,100	7, 8, 9, 10, 11, 12
GE-1541 ⁽⁵⁾	Fe-15Cr-4Al-1Y	--	--	--	Poor	Good	--	71,500	7, 11, 12
GE-1041 ⁽⁵⁾	Fe-10Cr-4Al-1Y	--	--	--	Poor	Good	--	67,000	7, 8, 11, 12
GE-0561 ⁽⁵⁾	Fe-5Cr-6Al-1Y	--	--	--	Poor	Good	--	64,300	7, 8, 11, 12
GE-254 (0.35) ⁽⁵⁾	Fe-25Cr-4Al-0.35Y	--	--	--	Marginal	Good	--	--	11, 13
GE-253 (0.5) ⁽⁵⁾	Fe-25Cr-3Al-0.5Y	--	--	--	Marginal	Good	--	--	11, 13
GE-2540 ⁽⁵⁾	Fe-25Cr-4Al	--	--	--	Good	Good	--	--	13
GE-1540 ⁽⁵⁾	Fe-15Cr-4Al	--	--	--	Good	Good	--	--	13
GE-0561+3Mo ⁽⁵⁾	Fe-5Cr-6Al-1Y-3Mo	--	--	--	Poor	Good	--	79,000	14, 12
GE-1061 ⁽⁵⁾	Fe-10Cr-6Al-1Y	--	--	--	Poor	Good	--	74,200	14, 12
Kanthal A-1 ⁽⁶⁾	Fe-22Cr-5.5Al-0.5Co	872	--	--	Good	Good	--	110,000	15

TABLE 1. (CONTINUED)

Alloy Designation	Nominal Chemical Composition, w/o	Electrical Resistivity at Room Temperature, Ω/cmf	Temperature Coefficient of Resistance (0 to 1000 C), $\mu\Omega/\Omega \text{ C}$	Approximate Gage Factor at Room Temperature	Probable Potential for Fabrication to Fine Wire	Probable Oxidation Resistance at 1200 F (650 C)	Coefficient of Thermal Expansion (0-1000 C), $\mu\text{in./in. C}$	Ultimate Tensile Strength at Room Temperature, psi	Reference
Hoskins 875 ⁽⁷⁾	Fe-22.5-5.5Al-0.5Si	875	+ 32 ⁽⁸⁾	--	Good	Good	--	115,000	16
--	Fe-23Cr-5.7Al-0.57Co-0.65Si-0.26Mn	--	--	--	Good	Good	--	--	17
--	Fe-22.4Cr-5.1Al-0.59Co-0.74Si-0.27Mn	--	--	--	Good	Good	--	--	17
--	Fe-21.7Cr-4.5Al-0.58Co-0.65Si-0.22Mn	--	--	--	Good	Good	--	--	17
--	Fe-24.6Cr-4.0Al	--	--	--	Good	Good	--	--	18
--	Fe-20Cr-3.0Al	720	+ 164	--	Good	Good	14.2	93,000	5
--	Fe-20Cr-6Al	960	+ 46	--	Good	Good	15.3	100,000	5
--	Fe-20Cr-8Al	1056	- 20	2.19	Good	Good	16.4	118,000	5
--	Fe-20Cr-10Al	1170	- 91	2.22	Good	Good	17.2	120,000	5
--	Fe-24Cr-7Al	1026	- 17	2.21	Good	Good	16.4	115,000	5
--	Fe-25Cr-5Al	918	+ 48	2.37	Good	Good	15.3	94,000	5
--	Fe-25Cr-6Al	966	+ 32	1.88	Good	Good	15.9	100,000	5
--	Fe-25Cr-12Al	1230	- 205	--	Poor	Good	21.2	130,000	5
--	Fe-20.12Cr-9.92Al-1.0Ti-0.13Mn	1170	+ 87	--	Marginal	Good	17.2	112,000	5

(1) Research alloy, Armour Research Foundation (now Illinois Institute of Technology Research Institute), Chicago, Illinois, circa 1955.

(2) A product of Driver-Harris Company, Harrison, New Jersey.

(3) -50 to 500 C.

(4) 20 to 500 C.

(5) Research alloy, General Electric Company, Evendale, Ohio, circa 1964.

(6) A product of The Kanthal Corporation, Bethel, Connecticut.

(7) A product of Hoskins Manufacturing Company, Detroit, Michigan.

(8) 25 to 1316 C.

TABLE 2. CANDIDATE PLATINUM-GROUP ALLOYS FOR HIGH-TEMPERATURE STRAIN GAGES

Alloy Designation	Nominal Chemical Composition, w/o	Electrical Resistivity at Room Temperature, Ω/cmf	Temperature Coefficient of Resistance (0 to 1000 C), $\mu\Omega/\Omega\text{ }^\circ\text{C}$	Approximate Gage Factor at Room Temperature	Probable Potential for Fabrication to Fine Wire	Probable Oxidation Resistance at 1200 F (650 C)	Coefficient of Thermal Expansion (0-1000 C), $\mu\text{in./in. }^\circ\text{C}$	Ultimate Tensile Strength, psi	Reference
--	Pt-5W	258	--	5.23	Good	Good	--	120,000	4, 19, 20
--	Pt-7W	360	--	5.0	Good	Good	--	128,000	4, 19, 20
--	Pt-8W	408	+216-325	4.45	Good	Good	--	130,000	4, 19, 21, 20
--	Pt-8W	330-355	+117	5.3	Good	Good	--	--	22
--	Pt-9W	480	--	4.93	Good	Good	--	132,000	4, 19, 20
--	Pd-40Ag	240	--	1.8	Unknown	--	--	--	4
--	Pd-35Ag-3Pt	228	--	--	Unknown	Good	--	--	4
--	Pd-65Ag-3W	240	--	--	Unknown	Good	--	--	4
--	Pt-9.5W	445	+145	3.74	Good	Good	8.9	142,000	5
--	Pt-45Pd-10Mo	468	+100	--	Good	Good	14.0	--	19, 21
--	Pt-10Ni	192	+350	4.2	Good	Good	--	--	22
--	Pt-8Ni-2W	188	+378	4.2	Good	Good	--	--	22
--	Pt-8Ni-2Cr	228	+245	4.1	Good	Good	--	--	22

In particular, the Fe-Cr-Al and the Fe-Cr-Al-Y alloys are well known for their outstanding resistance to oxidation at temperatures up to 2300 F.

The oxidation resistance of the Fe-Cr-Al alloys has been attributed to the formation of an adherent film of nearly pure $\alpha\text{-Al}_2\text{O}_3$ on the surface of the alloys.(11,23,24,19,18,25,17) Kornilov(26) reported that a film of nearly pure light gray alumina forms at temperatures above 1290 F (700 C). For example, Kornilov reported that the oxide formed on an Fe-25 w/o Cr-5 w/o Al alloy after a 240-hour exposure in air at 2190 F (1200 C) consisted of 98.72 percent Al_2O_3 . The formation of the aluminum oxide is due to the preferential oxidation of aluminum. In low-aluminum alloys, the loss of aluminum can represent a significant fraction of the amount present, whereas in high-aluminum alloys the loss is minor. Figure 1 illustrates the effect of aluminum content on the loss of aluminum by preferential oxidation at 2190 F (1200 C). Unfortunately, neither the size nor the surface-to-volume ratio of the specimens were given.

Other data of Kornilov(26) indicate that the chromium content of the Fe-Cr-Al alloys remains fairly constant even after 1000-to 3000-hour exposure at 2190 F (1200 C).

The oxide film that forms on Fe-Cr-Al alloys below 1290 F (700 C) consists of a solid solution of Al_2O_3 , Cr_2O_3 and Fe_2O_3 in approximately the same proportion as the elements in the alloy.(26,18)

Preoxidation of Fe-Cr-Al alloys at elevated temperatures (above 1290 F) to form a $\alpha\text{-Al}_2\text{O}_3$ film has been carried out or considered as a means of improving the oxidation resistance of these alloys for subsequent use at lower temperatures (below 1290 F).(18,3,11)

Experiments(26) have shown that both chromium and aluminum increase the oxidation resistance of Fe-Cr-Al alloys. However, the effect of aluminum per weight percent is much greater than that of chromium, as illustrated in Figures 2 and 3.

Kornilov(26) also found that carbon, when present in amounts exceeding the limit of solid solubility (about 0.03 w/o), rapidly decreases the oxidation resistance of Fe-Cr-Al alloys.

FIGURE 1. LOSS OF ALUMINUM AS A FUNCTION OF THE ORIGINAL ALUMINUM CONTENT OF Fe-Cr-Al ALLOYS, AFTER 1000-HR EXPOSURES AT 2190 F (1200 C) (Ref. 24,26)

Curve 1 averages results obtained on alloys containing 15-17% chromium and 3-15% aluminum. Curve 2 averages results obtained on alloys containing 25-27% chromium and 1.5-12.0% aluminum. Neither the size nor the surface-to-volume ratio of the specimens was given.

FIGURE 2. EFFECTS OF ALUMINUM AND OF CHROMIUM ON SCALING LOSSES OF CAST Fe-Cr-Al ALLOYS AT 2190 F (1200 C) (Ref. 24, 26)

Duration of tests was 240 hr.

FIGURE 3. EFFECTS OF ALUMINUM AND OF CHROMIUM ON SCALING LOSSES OF CAST Fe-Cr-Al ALLOYS AT 2190 F (1200 C) (Ref. 24, 26)

Additions of yttrium to Fe-Cr and Fe-Cr-Al alloys have been found by many investigators(27,28,9,10,11,29,30,31,32) to substantially improve the oxidation resistance of these alloys. Although yttrium-containing alloys have been studied extensively, the mechanism by which the yttrium improves the oxidation resistance is still not completely understood. A number of investigators(27,11,9,10) have shown that the yttrium oxidizes internally within the grains and at grain boundaries to form Y_2O_3 and that this oxide intimately contacts the surface oxide of aluminum. It is theorized that the oxidized yttrium prevents the Al_2O_3 from spalling by "locking" it into the substrate. Wood, et al(28) obtained data indicating that the improvement in oxidation resistance of Fe-Cr alloys with yttrium additions might be due to a layer of an yttrium compound at or near the base of a Cr_2O_3 -rich scale.

Wukusick(11,9) reported that yttrium additions to an Fe-25 w/o Cr-4 w/o Al alloy increases the initial oxidation rate as a result of oxidation of YFe_9 particles and internal oxidation. However, the improved adherence of the oxide reportedly more than compensates for the higher initial rate of oxidation. Wukusick also reported that alloys with lower yttrium contents showed lower oxidation rates in air, but exhibited poorer scaling resistance. Nevertheless, Perkins and Wukusick(29) state that a 0.1 w/o yttrium addition is sufficient to provide good oxidation resistance in Fe-Cr-Al-Y alloys for long exposures at temperatures up to 2012 F (1100 C). Figure 4 illustrates the effect of yttrium additions on the oxidation resistance in still air at 2012 F (1100 C) of an Fe-15 w/o Cr-4 w/o Al alloy. Figure 5 also shows the effect of yttrium as well as chromium and aluminum on the oxidation of Fe-Cr-Al-Y alloys.

For an optimum combination of workability and resistance to oxidation in air at about 2000 F (1093 C), studies have shown that the chromium content of Fe-Cr-Al and Fe-Cr-Al-Y alloys should be about 15 to 30 percent, and the aluminum content should be 3 to 5 percent.^(24,9) In Fe-Cr-Al-Y alloys, the optimum yttrium content was reported as 0.7 percent⁽⁹⁾.

Fe-Cr-Al and Fe-Cr-Al-Y alloys are generally considered only for use at very high temperatures, where their resistance to oxidation exceeds that of most other alloys. Accordingly, although much information

FIGURE 4. WEIGHT CHANGE OF Fe-15Cr-4Al ALLOYS CONTAINING 0 TO 1 PERCENT Y TESTED AT 1100 C IN AIR FOR 1000 HOURS (Ref. 32)

FIGURE 5. WEIGHT GAIN OF Fe-Cr-Al-Y ALLOYS AS A FUNCTION OF TEST TIME AT 900 C and 1100 C IN STATIC AIR FOR TIMES UP TO 10,000 HOURS (Ref. 11)

The chemical compositions of the alloys are given in Table 1.

was obtained on the rate of oxidation of these alloys at 1650 F (900 C) and above, no oxidation data were found for exposure of these alloys at 1200 F (650 C). Therefore no conclusions can be drawn regarding the minimum chromium, aluminum and yttrium contents that will yield the very low oxidation rate desired in a strain gage alloy at 1200 F.

The effect of thermal cycling on the oxidation resistance of Fe-Cr-Al alloys is important in strain-gage applications. Wukusick^(9,11) found that the oxide on an Fe-25 w/o Cr-4 w/o Al-1 w/o Y alloy cracks after an increase in weight of about 4 mg/cm² if the alloy is thermally cycled, presumably between room temperature and 2400 F (1315 C). However, Al₂O₃ forms under the crack to slow the rate of further oxidation. Saller, Stacy, and Eddy⁽³³⁾ reported that, in general, Fe-Cr-Al alloys oxidize more rapidly during thermal cycling between 850 and 2200 F (454 and 1200 C) than during exposure at a constant temperature of 2200 F (1200 C).

Strain-oxidation tests were carried out by Gulbransen and Andrew⁽¹⁷⁾ on three iron-base alloys containing from 21.7 to 23.0 percent chromium, 4.5 to 5.7 percent aluminum, and minor additions of silicon, manganese, and cobalt. In these tests, the alloys were oxidized about 2 hours at 1650 F (900 C), cooled and strained from 2 to 4 percent, and then reheated and oxidized 2 additional hours at 1650 F. The results of these tests indicate that the oxidation rate of the alloys containing 22.4 w/o Cr-5.1 w/o Al and 21.7 w/o Cr-4.5 w/o Al increased appreciably with only 2 percent strain. On the other hand, the oxidation rate of an alloy containing Fe-23.0 w/o Cr-5.7 w/o Al did not increase until it was strained 4 percent. The effect of strain in each case was attributed not only to cracking of the oxide layer during straining but also to possible localized damage on a microscopic or even submicroscopic level. The quantitative effect of strain would be expected to also depend on the thickness, chemical composition, and degree of adhesion of the oxide coating; thus, straining during exposure at 1200 F might produce different results than straining at room temperature after exposure at 1650 F.

Platinum-Group Alloys

The oxidation resistance of platinum- and palladium-base alloys at 1200 F, as indicated in Table 2, is expected to be good. The use of the precious metal alloys for high-temperature strain-gage applications has been studied extensively by Bertodo(5,19,21,34,20) and by other investigators.(4,35,22,36,37)

Bertodo(20) found that the oxidation rate at 1832 F (1000 C) of platinum alloys containing tungsten reached a minimum at approximately 8 w/o tungsten, as illustrated in Figure 6. Bertoda(5) quoted the oxidation rate of a Pt-9.5 w/o W alloy in still air at 1110 F (600 C) as being 0.00084 g/m²h.

Bertodo(21) evaluated Pd-Mo alloys for use as a high-temperature strain-gage alloy. He found that the oxidation resistance of these alloys was inadequate, although the oxidation resistance of a Pd-45 w/o Pt-10 w/o Mo was reportedly good. Furthermore, this alloy possessed electrical properties that made it more suitable for strain-gage applications than Pt-W alloys.(21) However, the Pd-Pt-Mo alloy has never been evaluated in a strain gage(38).

Figure 7 shows the "oxidation limit" as well as the "workability limits" for Pt-Pd-Mo alloys. Unfortunately, the terms "oxidation limit" and "workability limit" were not defined by the author.

Metallurgical Structure and Stability

The electrical properties of an alloy are dependent upon the composition and metallurgical phases of the alloy as well as upon imperfections in the crystal lattice that interfere with the movement of conduction electrons. Therefore, since good electrical stability is desired for a strain-gage alloy, it is important that little or no change occurs in the microstructure of the alloy during a temperature excursion from room temperature to the service temperature or during holding at the service temperature. Changes in microstructure could also affect the gage factor of the alloy. Accordingly, it is desirable that an alloy for strain-gage applications be a single-phase solid solution and undergo no order-disorder reactions over the entire temperature range of use.

FIGURE 6. INITIAL OXIDATION RATE OF PLATINUM-TUNGSTEN ALLOY IN STILL AIR AT 1000 C (Ref. 20)

FIGURE 7. "OXIDATION LIMITS" AND "WORKABILITY LIMITS" FOR Pd-Pt-Mo ALLOYS

The terms "oxidation limit" and "workability limit" were not defined by the author.

Fe-Cr-Al-Base Alloys

The microstructure and the stability of the microstructure of Fe-Cr-Al alloys have been the subject of many investigations. (7,8,39,1,11,29,30,23,32,42,14,40,33,41) The low-temperature portion of the Fe-Cr binary phase diagram(2) and the effect of a 4 w/o addition of aluminum on the extent of the phase fields, are shown in Figure 8. It can be seen that the addition of aluminum increases the solubility limit of chromium in the α solid solution.

As may be seen from Figure 8, alloys which contain 4 w/o aluminum and between about 17 and 48 w/o chromium will precipitate sigma (σ) phase (FeCr) from the α solid solution over a temperature range which has 968 F (520 C) as its lower limit. (32,7,11,29,30,14,33) The upper limit of the range depends on the chromium content of the alloy. Additions of aluminum appear to decrease the amount of σ formed at any given temperature in the range.

Figure 8 also indicates that at room temperature, under equilibrium conditions, most Fe-Cr and Fe-Cr-4 Al alloys consist of two phases α and α' . [α' is a chromium-rich ferrite phase that forms at temperatures below 968 F (520 C).] However, it has been reported that alloys must contain at least 10 w/o chromium (presumably, at either the 0 or 4 w/o aluminum level) before the presence of α' is noticed under "normal" (nonequilibrium) conditions.(32). The higher the aluminum content, the greater the amount of chromium that the alloy can contain without the formation of α' .

The precipitation of σ and α' results in hardening and a loss in ductility. In addition, such precipitation is a microstructural instability that is believed to cause an undesirable change in the electrical properties of the alloy. Accordingly, it is preferable that the chemical compositions of alloys used in strain gages be such that neither σ nor α' forms in the service temperature range.

The microstructure of alloys containing yttrium consists of a ferritic matrix containing a random dispersion of an yttrium-iron intermetallic compound. Most of the investigations(7,32,11,29,30,42) of Fe-Cr-Al-Y alloys identified this compound as YFe_9 . However, more recent work by Bartos(41) has identified the particles as mixtures of YFe_5 and Y_2Fe_{17} with small amounts of aluminum and chromium in solid solution

FIGURE 8. LOW-TEMPERATURE PORTION OF THE Fe-Cr BINARY PHASE DIAGRAM, WITH DOTTED LINES INDICATING THE EFFECT OF AN ADDITION OF 4 PERCENT ALUMINUM ON THE PHASE FIELDS (Ref. 7)

with the compounds. The ferritic matrices of Fe-Cr-Al-Y alloys consist of a solid solution of iron, chromium, and aluminum; yttrium is practically insoluble (the solubility is less than 0.1 weight percent) in the Fe-Cr-Al matrix.(11,7)

A reduction of the chromium content improves the stability of Fe-Cr-Al-Y alloys in the temperature range where α' precipitation occurs in higher chromium alloys. This improvement in stability is illustrated in Figure 9, which shows the effect of aging time on the hardness of a number of Fe-Cr-Al-Y alloys. In order to maintain good oxidation resistance, however, the aluminum content must be increased when the chromium content is decreased.(11)

Platinum-Group Alloys

Platinum-group alloys often show wide miscibility gaps and have a tendency to form compounds. Therefore, only dilute alloys would be expected to be single phase.(19)

Although platinum-tungsten alloys containing low tungsten contents have been said to be single phase at all temperatures,(4) the results of research by Bertodo(38,21) indicate to the contrary. Specifically, Bertodo(38,21) found that, after 500 hours at 1110 F (600 C), the popular Pt-8 W strain-gage alloy is no longer single phase. A further indication of the instability of this alloy for use as a strain gage for long times at 1200 F (650 C) was provided by Hudson and Axley.(43,44) These researchers found voids in the interior of Pt-8 W wires after "long-time exposure" at temperatures as low as 965 F (520 C). The rate of formation of the voids was found to increase with increasing temperature; the holes were said to form "rapidly" at 1200 F (650 C). Quantitative data on the rate of formation of the holes are not available. Hudson and Axley(43,44) attributed the voids to vaporization of tungsten from the alloy.

Further indication of the instability of platinum-tungsten alloys at 1200 F was shown by Lemcoe and Dickinson(37). Using microprobe analysis, they showed that the concentration of tungsten was essentially constant through a Pt-8 w/o tungsten alloy wire before testing. However, after

FIGURE 9. ROOM-TEMPERATURE HARDNESS AS A FUNCTION OF AGING TIME AT 450 C FOR LOW-CHROMIUM-CONTENT Fe-Cr-Al-Y ALLOYS COMPARED WITH 2541 AND 1541 ALLOYS (Ref. 7)

2541 Alloy: 25Cr-4Al-1Y

1541 Alloy: 15Cr-4Al-1Y

holding for 112 hours at 1200 F it was found that the concentration of tungsten was higher at or near the surface of the wire than at the interior. After 208 hours at 1200 F, it appeared that the average tungsten content of the wire had decreased to about 7 w/o from the original 8 w/o value. Lemcoe and Dickinson⁽³⁷⁾ also obtained indications that oxides or nitrides might have formed on the surface of the wire during exposure at 1200 F.

Bean⁽²²⁾ evaluated the metallurgical stability of a number of platinum-base alloys for strain-gage applications at temperatures up to 1200 F (650 C). Electrical-resistance measurements were taken on strain gages made from wire of the alloys while the gages were being heated to 1200 F at the rate of 20 F/minute and cooled to room temperature at the rate of 10 F/minute. The gages were run through one or two of these cycles. On the basis of the electrical-resistance measurements, Bean concluded that the following alloys were stable at temperatures up to 1200 F:

90 Pt-8 Ni-2 W
 90 Pt-8 Ni-2 Cr
 92 Pt-8 W.

Bean's conclusion in the case of the 92 Pt-8 W alloy does not agree with the aforementioned findings of Bertodo^(38,21), Hudson and Axley^(43,44), and Lemcoe and Dickinson.⁽³⁷⁾ This discrepancy can be explained by the fact that Bean's samples were only exposed to elevated temperatures for a few hours, while the samples of the other investigators were exposed for hundreds of hours.

Electrical Resistivity and Temperature Coefficient

Fe-Cr-Al-Base Alloys

Fe-Cr-Al-base alloys have been considered good candidates for high-temperature strain gages by a number of investigators^(45,46,47,48,5,2,3,1,23) because of their excellent oxidation resistance at elevated temperatures and their favorable electrical properties such as high resistivity, and low-temperature coefficient of resistance. Various electrical properties of Fe-Cr-Al alloys are presented in Figures 10 through 19.

FIGURE 10. ELECTRICAL RESISTIVITY OF IRON-CHROMIUM-ALUMINUM ALLOYS IN MICROHM-CM (Ref. 23)

FIGURE 11. ELECTRICAL RESISTIVITY OF IRON-CHROMIUM-ALUMINUM ALLOYS WITH CONSTANT RATIO IRON: CHROMIUM OF 7:2 (Ref. 23)

FIGURE 12. CHANGE OF ELECTRICAL RESISTIVITY BETWEEN 32 AND 1600 F, PERCENT (Ref. 23)

FIGURE 13. RESISTIVITY VERSUS TEMPERATURE CHARACTERISTICS OF FOUR Fe-Cr-Al ALLOYS, 20 C TO 1200 C (Ref. 2)

FIGURE 14. TYPICAL ELECTRICAL RESISTANCE CHARACTERISTICS OF SOME IRON-CHROMIUM-ALUMINUM ALLOYS (Ref. 5)

FIGURE 15. EFFECT OF TEMPERATURE ON THE PERMANENT ELECTRICAL RESISTANCE CHANGE OF A 20 PERCENT CHROMIUM-10 PERCENT ALUMINUM-IRON ALLOY SPECIMEN (Ref. 5)

FIGURE 16. EFFECT OF THERMAL CYCLING BETWEEN ROOM TEMPERATURE AND 1600 F ON THE ELECTRICAL RESISTANCE OF Fe-Cr-Al WIRE (Ref. 3)

Alloy B Composition: Fe-24.89Cr-7.84Al

FIGURE 17. VARIATION OF PERMANENT RESISTANCE CHANGE WITH CYCLING BETWEEN ROOM TEMPERATURE AND 700 C FOR AN Fe-20Cr-10Al-1.0Ti ALLOY (Ref. 5)

FIGURE 18. EFFECT OF THERMAL CYCLING BETWEEN ROOM TEMPERATURE AND 1600 F ON THE ELECTRICAL RESISTANCE OF AN Fe-Cr-Al STRAIN GAGE (Ref. 3)

Alloy B Composition: Fe-24.89Cr-7.84Al

FIGURE 19. EFFECT OF THERMAL CYCLING BETWEEN ROOM TEMPERATURE AND 1600 F ON THE ELECTRICAL RESISTANCE OF AN Fe-Cr-Al STRAIN GAGE (Ref. 3)

Alloy D Composition: Fe-20.5Cr-10.5Al

The effect of chemical composition on the electrical resistivity of Fe-Cr-Al alloys is illustrated in Figures 10 and 11. Figures 11 and 12 show the effect of aluminum content on the temperature coefficient of resistance. Figures 13 and 14 show the effect of temperature on the resistivity and resistance, respectively. The effect of long-time exposures at elevated temperatures on electrical resistance is shown in Figure 15. Figures 16 and 17 show the effect of thermal cycling on the electrical resistance of samples of two Fe-Cr-Al alloys, while Figures 18 and 19 illustrate the effect of thermal cycling on strain gages made from two Fe-Cr-Al alloys. The tendency of these gages to stabilize after several thermal cycles may be noted.

Platinum-Group Alloys

Platinum-group alloys are characterized by low resistivities, high temperature coefficients of electrical resistance and extreme sensitivity to trace impurities.⁽²¹⁾ However, they have been said to exhibit good electrical resistance stability and high strain sensitivities⁽²¹⁾ which, along with their resistance to corrosion and oxidation, have made them candidate alloys for high-temperature strain gages.

Figures 20 and 21 present the electrical properties of platinum-palladium-molybdenum alloys and platinum-tungsten alloys. Figure 20 illustrates the effect of composition on the resistance coefficients of platinum-palladium-molybdenum alloys. Figure 21 shows the effect of tungsten content on the resistivity, strain sensitivity, and temperature coefficient of resistance of platinum-tungsten alloys.

The comment made earlier in this section regarding the good electrical resistance stability of platinum-group alloys is open to question, at least in respect to platinum-tungsten alloys. For instance, Bertodo⁽²¹⁾ and Lemcoe and Dickinson⁽³⁷⁾ have found that the electrical resistance of the Pt-8 w/o W alloy changes during exposure at 1110 F (600 C) and 1200 F (650 C), respectively. In Bertodo's⁽²¹⁾ experiments, the resistivity and temperature coefficient of resistance of this alloy increased during the first 100 hours at 1110 F, remained constant during the next 100 hours, and then began again to increase gradually. The

FIGURE 20. ELECTRICAL RESISTANCE COEFFICIENTS OF PLATINUM-PALLADIUM-MOLYBDENUM ALLOYS (Ref. 19)

The temperature range over which the indicated temperature coefficients of resistance were measured was not given in the reference.

FIGURE 21. ELECTRICAL AND MECHANICAL PROPERTIES OF ANNEALED PLATINUM TUNGSTEN ALLOYS (Ref. 20)

The temperature range over which the temperature coefficients of resistance were measured were not given in the reference.

resistivity and temperature coefficient did not stabilize even after 500 hours at 1110 F. Lemcoe's and Dickinson's⁽³⁷⁾ data are given in Figure 22 where it may be seen that the electrical resistance of Pt-8 w/o W wires decreases with time of exposure at 1200 F; after about 210 hours at 1200 F the resistance appears to approach a constant value.

Thus, although the results of Bertodo⁽²¹⁾ and Lemcoe and Dickinson⁽³⁷⁾ both indicate that the electrical resistivity of the Pt-8 w/o W alloy is not stable at elevated temperatures the algebraic sign of the change in resistance with time is different in the two cases. This may be due to the wires possibly having been heat treated differently prior to testing in the two cases, to the fact that the tests were carried out at different temperatures, or to differences in the chemical composition and impurity content of the wires used. Changes in the electrical resistivity and temperature coefficient of resistance of Pt-8 w/o W alloys at elevated temperatures may be a result of such factors as the precipitation of second-phase particles, formation of voids, migration and vaporization of tungsten, or internal oxidation or nitridation, as discussed in the section entitled "Metallurgical Structure and Stability".

Fabricability

Fe-Cr-Al-Base Alloys

The fabrication behavior of Fe-Cr-Al-base alloys has been noted by many investigators.^{27,5,8,39,2,3,1,9,49,10,11,29,30,31,32,14,12,50,40,33,25)} Based on a review of the literature, the actual or estimated fabricability of a large number of Fe-Cr-Al-base alloys to fine wire is indicated in Table 1.

Kornilov^(24,26) investigated the fabricability of alloys containing up to 70 w/o chromium and 12.5 w/o aluminum. The results of Kornilov's work are presented in Figure 23. The data show compositional limits for hot and cold working Fe-Cr-Al alloys. For example, alloys containing no chromium and 11 w/o Al out to a maximum of 30 w/o Cr and 3 w/o Al may be worked hot or cold. Outside this area, alloys containing no chromium and

FIGURE 22. ELECTRICAL RESISTANCE VERSUS TIME FOR 92Pt-8W WIRE AT 1200 F (Ref. 37)

FIGURE 23. FABRICABILITY LIMITS OF IRON-CHROMIUM-ALUMINUM ALLOYS
(Ref. 24, 26, 40)

15 w/o Al up to a maximum of 40 w/o Cr and 12 w/o Al can be hot worked only. The results of later investigations have shown that the extent of the composition field of alloys that can be cold worked as well as hot worked is somewhat broader than indicated in Figure 23, however.

Wukusick and Collins⁽⁹⁾ found that, for optimum workability and oxidation resistance, the composition of Fe-Cr-Al-Y alloys should not exceed 25 w/o chromium, 5 w/o aluminum and 0.7 w/o yttrium. This material has been hot and cold worked to 5-mil-thick foil and 25-mil-diameter wire. However, it is very doubtful that an alloy with approximately this chemical composition could be drawn to 1 or 2 mils in diameter,^(51,52) because of the presence of large particles of yttrium-iron compounds; these particles were discussed in a previous section. On the other hand, it may be possible to draw an yttrium containing alloy of this type, provided the yttrium content is reduced to a lower level such as 0.1 w/o. The oxidation resistance of Fe-Cr-Al alloys containing only 0.1 w/o yttrium would be expected to be good at 1200 F (650 C), since, as mentioned previously, such alloys exhibit good oxidation resistance for long exposures at temperatures up to 2012 F (1100 C).⁽²⁹⁾

General Electric Company, Evendale, Ohio, is currently investigating methods for preparing Fe-Cr-Al-Y alloys by powder metallurgical techniques, under the sponsorship of the Naval Air Systems Command.⁽⁵¹⁾ It was reasoned that the drawability of the powder metallurgical product might exceed that of material that is made by melting and casting, after the powder techniques have been optimized. The reason for the possible improvement in drawability is that the Fe₃Y particles are smaller in the powder metallurgical product. However, no wire has been made from this type of material thus far; General Electric's experiments have been carried out on relatively large specimens.

In regard to hot working, a variety of Fe-Cr-Al-base alloys have been extruded, hammer forged, press forged, rolled and swaged with success into bars, rods, and strip. General Electric^(10,9,32,25) has extruded their Fe-Cr-Al-Y alloys into rods at approximately 1832 F (1000 C), using extrusion ratios as high as 24:1. Hammer and press forging of Fe-Cr-Al ingots has been carried out at temperatures ranging from 2400 F (1316 C) down to 1800 F (980 C) and lower, depending on

the investigator and the chemical composition of the alloy. (33,53,50,2,3,49,14) Rolling and swaging of Fe-Cr-Al-base alloys have been conducted at temperatures varying between 2500 F (1371 C) and 1100 F (593 C). (2,3,33,30,14, 32,5) As was the case in forging these alloys, the working temperature varied with the investigator and the chemical composition of the alloy.

On the basis of a review of the literature on the hot working of Fe-Cr-Al-base alloys; it is concluded that, in addition to the chemical composition of the alloy the quality of the ingot (as indicated by purity, grain structure, and the presence or absence of porosity) is an important determinant of the fabricability and optimum working temperature of the alloy. This conclusion is made because the reported hot working temperatures of alloys having the same composition were quite different. In fact, Strawley⁽⁴⁾ reported that severe cracking was encountered at temperatures which others gave as the optimum working temperature for the same alloys.

Alloys that possess cold-working characteristics have either been swaged, rolled or drawn. Intermediate anneals at 1200 to 1800 F (650 to 982 C) have been used during these cold-working processes. (14,55,2,3,50) It should be noted that the mere fact that an alloy has been cold worked does not imply that it can be drawn into 1- or 2-mil-diameter wire.

Platinum-Group Alloys

Although platinum-group alloys have been evaluated for high-temperature strain gages, no detailed information was found regarding their fabrication. The investigators (34,20,19,4,22,21) of these alloys have simply stated that certain alloys in the form of wire varying from 1 to 24 mils in diameter were evaluated. Bertodo⁽¹⁹⁾ gave an indication of the fabricability of platinum-palladium-tungsten alloys on a ternary diagram of these elements which is shown in Figure 7. In another article, (20) Bertodo reported that platinum alloys containing up to 15 w/o tungsten could be drawn into fine wires.

The observed or estimated fabricability to fine wire of a number of platinum-base alloys is indicated in Table 2.

Mechanical Properties

Fe-Cr-Al-Base Alloys

The mechanical properties of Fe-Cr-Al-base alloys have been determined by many investigators. (7,8,39,9,4⁹,10,11,29,30,31,32,42,14,12,50,40,33,25,26) Thus, a voluminous amount of data was uncovered in this search. A limited amount of these data is presented to illustrate the range of values that can be obtained.

Figure 24 shows the effect of temperature on the tensile properties of an Fe-25 w/o Cr-5 w/o Al alloy.

The effect of temperature on the tensile and yield strengths of Fe-Cr-Al-Y alloys containing various levels of chromium and aluminum is illustrated in Figure 25. These data indicate that the tensile and yield strengths of these alloys increase with increasing amounts of chromium and aluminum.

The creep properties of an Fe-25 w/o Cr-4 w/o Al-1 w/o Y alloy were determined by General Electric⁽³¹⁾ at temperatures of 1090 F (590 C) and 1200 F (650 C). This alloy exhibited 0.5 percent creep in 5400 hours under stress of 3000 psi at 1090 F and 0.4 percent creep after 5765 hours under a stress of 1990 psi at 1200 F. An Fe-15 w/o Cr-4 w/o Al-1 w/o Y alloy exhibited 7 percent creep after 5200 hours under a stress of 7000 psi at 1000 F (540 C).

The creep-rupture properties of a number of Fe-Cr-Al-Y alloys were studied by General Electric⁽¹²⁾ and are presented in Figure 26. These data indicate that at 1020 F (550 C) the creep-rupture strengths of the alloys were about the same with the exception of the molybdenum-containing alloy which had a higher strength. However, at 1380 F (750 C) the strengths of the Fe-25 w/o Cr-4 w/o Al-1 w/o Y and the Fe-5 w/o Cr-6 w/o Al-1 w/o Y-3 w/o Mo alloy were greater than those of the other alloys.

The effect of chemical composition on the hardness of Fe-Cr-Al alloys was determined by Kornilov.⁽²⁶⁾ The data, as shown in Figure 27, indicate that chromium and aluminum both increase the room-temperature hardness of the alloys. Chubb, et al⁽⁴⁰⁾ determined the hot hardness of

FIGURE 24. TENSILE PROPERTIES OF IRON-17 TO 25 w/o CHROMIUM-5 w/o ALUMINUM ALLOYS (Ref. 24, 26, 40)

FIGURE 25. THE EFFECT OF TEMPERATURE ON THE TENSILE AND YIELD STRENGTHS OF Fe-Cr-Al ALLOYS (Ref. 12)

The chemical compositions of the alloys are given in Table 1.

FIGURE 26. CREEP RUPTURE DATA FOR Fe-Cr-Al-Y ALLOYS (Ref. 12)

The chemical compositions of the alloys are given in Table 1.

FIGURE 27. HARDNESS AT ROOM TEMPERATURE OF IRON-CHROMIUM-ALUMINUM ALLOYS AS A FUNCTION OF COMPOSITION (Ref. 26, 40)

two Fe-Cr-Al alloys. Their data, presented in Figure 28, indicate that the hot hardness of an Fe-35 w/o Cr-7 w/o Al alloy was higher than that of an Fe-25 w/o Cr-5 w/o Al alloy at temperatures above 500 F (260 C).

General Electric^(7,8,11,29,14) studied the effect of aging time and temperature on the hardness of Fe-Cr-Al-Y alloys. Figure 29 and 30 show the effect of aging time at 840 F (450 C) on the hardness of a number of their alloys. These data indicate that Fe-Cr-Al-Y alloys containing 15 percent chromium or more age harden, while those with less than 15 percent chromium do not age harden at 840 F. The degree of hardening was related primarily to the chromium content and increased with the chromium content. The hardness increased slightly with increased amounts of aluminum. Yttrium and aluminum both increased the rate of precipitation hardening, although they had little effect on the ultimate hardness.^(11,14) The addition of molybdenum to the Fe-5 w/o Cr-6 w/o Al-1 w/o Y alloy promotes the aging reaction; however, the alloy overages rapidly to eliminate any problem with loss of ductility.⁽³³⁾

The effect of aging time at 1020 F (550 C) on the hardness of Fe-Cr-Al-Y alloys is illustrated in Figure 31. These data indicate that only the Fe-25 w/o Cr-4 w/o Al-1 w/o Y age hardened significantly.

Fe-Cr-Al and Fe-Cr-Al-Y alloys containing from 15 to 25 w/o chromium exhibit ductile-to-brittle behavior with decreasing temperature as a result of alpha prime or sigma precipitation and, in the case of yttrium-containing alloys, on the size and distribution of the Y-Fe secondary phases.⁽¹¹⁾

Kornilov⁽²⁶⁾ and Chubb, et al⁽⁴⁰⁾ studied the effect of carbon and aluminum, respectively, on the ductile-to-brittle transition temperature (DBTT) of Fe-Cr-Al alloys by means of impact tests. Kornilov and Chubb, et al, found that increased carbon and aluminum contents result in an increase in the DBTT, as illustrated in Figures 32 and 33.

General Electric^(11,31,32,42,12) determined the DBTT of Fe-Cr-Al-Y alloys, also by conducting impact tests. Figure 34 shows the effect of alloy composition on the absorbed energy at temperatures up to about 570 F (300 C). These data indicate that yttrium increased the

FIGURE 28. HARDNESS OF SELECTED IRON-CHROMIUM-ALUMINUM ALLOYS AS A FUNCTION OF TEMPERATURE (Ref. 40)

FIGURE 29. HARDENING CHARACTERISTICS OF Fe-Cr-Al-Y ALLOYS DURING AGING AT 450 C (Ref. 14)

The chemical compositions of the alloys are given in Table 1.

FIGURE 30. HARDNESS VERSUS TIME AT 450 C FOR VARIOUS Fe-Cr-Al-Y ALLOYS (Ref. 29)

The chemical compositions of the alloys are given in Table 1.

FIGURE 31. HARDNESS VERSUS TIME AT 550 C FOR VARIOUS Fe-Cr-Al-Y ALLOYS (Ref. 29)

The chemical compositions of the alloys are given in Table 1.

FIGURE 32. EFFECT OF CARBON ON THE IMPACT STRENGTH OF IRON-25 w/o CHROMIUM-5 w/o ALUMINUM ALLOY (Ref. 26, 40)

FIGURE 33. EFFECT OF ALUMINUM ON THE TRANSITION TEMPERATURE OF IRON-25 w/o CHROMIUM ALLOYS (Ref. 40)

FIGURE 34. ABSORBED ENERGY AS A FUNCTION OF TEST TEMPERATURE FOR NOTCH-IMPACT TESTS OF DISPERSION-STRENGTHENED O563 ALLOY COMPARED TO CAST AND EXTRUDED ALLOYS (Ref. 11)

Alloys tested in the annealed condition

DBTT about 200 C, while the chromium content had little apparent effect on the DBTT. Also, the data indicate that coarse Y-Fe particles serve as internal notches and increase the DBTT. Small Y-Fe particles present in one of the alloys containing 3 w/o yttrium were not as detrimental as the coarse particles. Other General Electric data⁽³¹⁾ indicated that the DBTT increased about 140 F (60 C) when the aluminum content was increased from 4 w/o Al to 6 w/o Al. However, the data in Figure 34 indicate that aluminum had no appreciable effect on the DBTT.

Platinum-Group Alloys

Very little data on the mechanical properties of platinum-group alloys were found in the technical literature. Bertodo^(19,20) presented Conchie's⁽⁵⁶⁾ data showing the effect of tungsten on the tensile strength of platinum-tungsten alloys. The data in Figure 35 show that the tensile strength increases significantly with increasing tungsten content.

Conclusions

The literature search and field survey revealed that there are three types of alloys that might be considered for high-temperature strain gages. These alloys are nickel-chromium-base alloys, iron-chromium-aluminum-base alloys and platinum-base alloys.

On the basis of the data uncovered and discussed in this report, it is concluded that certain Fe-Cr-Al and Fe-Cr-Al-Y alloys offer more promise for a 1200 F (650 C) strain gage than any alloys from the other two alloy systems. Selected Fe-Cr-Al-base alloys are expected to exhibit good oxidation resistance, good fabricability to fine wire or foil and adequate mechanical properties at 1200 F. In addition to these characteristics, which certain alloys of the Ni-Cr-base and Pt-base systems are also expected to possess, it is believed that Fe-Cr-Al-base alloys can be formulated so that the alloys will be metallurgically stable, exhibit good electrical properties and possess a suitable coefficient of thermal expansion in strain-gage applications at 1200 F.

FIGURE 35. MECHANICAL PROPERTIES OF ANNEALED
PLATINUM-TUNGSTEN ALLOYS (Ref. 20, 56)

On the other hand, nickel-chromium, nickel-chromium-base, and some platinum-tungsten alloys have severe limitations in regard to this application. Nickel-chromium and nickel-chromium-base alloys undergo order-disorder and precipitation reactions between room temperature and 1200 F. Platinum-tungsten alloys, at least in the area of 8 w/o tungsten, possess the following limitations:

- (1) The metallurgical structure is not stable at temperatures up to 1200 F (650 C). Not only doesn't the alloy remain a single-phase solid solution and the tungsten content tends to decrease after holding at these temperatures, but also voids have been found to form within Pt-8 w/o W wires after exposure at temperatures between 965 and 1200 F (518 and 650 C).
- (2) The electrical resistivity and temperature coefficient of the alloy change with time at temperatures up to 1200 F (650 C).
- (3) The thermal expansion coefficient of the alloy from room temperature to 1200 F (650 C) is very much lower than that of the alloys whose strain is to measured. This difference is said to be enough to cause plastic deformation of the wire at service temperatures.(44)

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HIGH-TEMPERATURE STRAIN GAGES

The review of literature on high temperature strain gages covered published material from the Western World and as well as some from Slavic sources. Although the period covered was from 1957 to 1970, a considerable portion of the material obtained centered around the late fifties and early sixties. The number of really useful references were relatively few and often dealt with evaluations of commercial gages rather than the development of new and improved gages or gage materials. However, of the gages discussed, very few were recommended for static applications in the 900 F to 1200 F regions. None were believed to be completely satisfactory for long-term use in a liquid sodium environment.

All commercial gages for use to 1200 F are of the wire type and employ platinum-tungsten alloys. There were, however, other gages not commercially available that were developed for high temperature applications. Notable were the LMEC (Liquid Metal Engineering Center) gages employing basically iron-chromium-aluminum alloy wire in the sensing elements. Remarkable stability was reported for some of these gages over long periods of time.

Another development was the capacitance gage developed at the Space Systems Division of the Hughes Aircraft Company for Wright-Patterson Air Force Base at Dayton, Ohio. The plates of the capacitors are mounted inside a miniature rhombic frame. The diagonal distance across the frame of each gage is the gage length.

The dielectric material used principally for insulating and bonding the wire-type gages was high-purity alumina. A notable exception is the Microdot SG 420 gage. This gage employs magnesium oxide to insulate the wire from its stainless steel sheath. The Rockide process using alumina rod material for flame spraying has probably been used most extensively for bonding, although powdered flame spraying of alumina was discussed. Better adhesion of the alumina has been reported by using a substrate of nickel aluminide, where the gages are used particularly on refractory alloys containing nickel.

The half-bridge concept seems to be useful for compensation purposes. However, there seems to be some promise of obtaining successful compensation by the use of negative TC (temperature coefficient of resistance) wire such as certain iron-chromium-aluminum wires with positive TC wire such as platinum. One way this was accomplished was by dual or three element configurations.

Lead wires above 1000 F are usually enclosed in a stainless steel metallic sheath and employ magnesium-oxide as insulation. Copper leads have been used to 1200 F where a vacuum or protective atmospheres can be maintained. Where protective atmospheres are not possible, the copper lead wires are usually coated with stainless steel. Other investigators reported using Nichrome V or Karma materials for leads.

One of the principal problems still seems to be associated with the joints between the fine sensing wires and the tabs of the gage or the lead wires. For the most part these joints are still made by capacitance discharge welding. Although other means of joining, such as by lasers and electron beams, were mentioned, no successful results were reported.

Some success has been obtained using ultrasonic welding to bond fine wires to larger diameter wires or to flat surfaces^(1,2). Much of the work was apparently with gold and aluminum for the microelectronic industry. It was apparently not necessary to remove the aluminum oxide from the metal prior to bonding. One of the main problems appears to be fatigue cracks around the welds caused by the high alternating stresses at the edges of the weld areas.

In the past few years there has been a renewed interest in high-temperature strain gages corresponding somewhat to the effort in the late fifties and early sixties. This current interest stems from the need for high temperature strain gages to implement the work on liquid metal reactors, re-entry vehicles, and jet engine research. Some of this work is being released from private sources and soon will add to the knowledge of the subject.

High Temperature Strain Sensing Systems

Two basic types of transducing systems were reported in the literature for remotely indicating strain at high temperatures. One was

based on electrical resistance as a function of strain and the other was based on capacitance as function of strain. The greater proportion of the work was done employing resistance devices; however, the advantages of capacitance devices cannot be ignored.

Strain measurement problems with electrical resistance systems multiply rapidly when larger excursions of temperature are encountered. This is especially true where the changes in temperature are rapid. Some of these problems can be minimized with capacitance devices. Where metal parts are used to effect the change in the spacing of the miniature capacitance plates, difficulties are still present. Also, the small changes in capacitance induced by the strain tend to be masked by capacitance changes in the lead wires.

Electrical Resistance Gage Systems

When considering the resistance systems for measuring strain, the emphasis often is placed on the gage itself. However, the accuracy of the measurements depends upon the entire measuring system, including the leads, the means of attachment, and the readout system. High temperatures or rapid changes in temperature result in many complications not associated to such a degree with room-temperature, electrical-resistance gages. Some of these complications are:

- (1) The temperature coefficients of resistance of many alloys are so high that even compensation systems do not prevent larger errors from masking the resistance changes caused by strain.
- (2) Oxidation problems of the sensing alloys and the lead materials result in changes in resistance causing errors.
- (3) Instability in the sensing and lead materials may be induced by metallurgical phase changes or migration of elements within the alloys.
- (4) The relief or development of internal strains in the sensing alloys from causes other than the applied mechanical strains may result from strain or work hardening or from differences in thermal coefficients of expansion. Many alloys exhibit marked changes in resistance between the elastic and plastic states.

- (5) Changes occur in the dielectric characteristics of the insulating materials with temperature. As the temperatures increase above given levels the dielectric materials tend to become conductive, even acting as rectifiers depending on conditions of purity, etc.
- (6) Other problems arise from the slippage of the sensing alloy materials in the dielectric bonding materials. This results in a different gage factor between tension and compression strains.
- (7) Shear lag effects often result from the attachment of the gages. This is encountered where the gages must be attached by spot welding or other means.
- (8) Development of stress concentrations around the attachment weld areas results in changes in the variations in strain transfer between the structure and the gage.
- (9) Changes in resistance at the welded connections between the leads and the gage wires or tabs have been the cause of gage inaccuracies and failures.

In view of all of the above problems, work has been continuing on resistance gage systems with the principal effort to date being on wire gages. Only a small amount of effort has been placed on foil gages. These gages require ceramic cements, since the ultra-thin materials are damaged by flame or plasma spraying⁽³⁾. Also, a curing cycle is required for ceramic cements, making "in situ" installations difficult or impossible for many applications.

Another type of high temperature electrical resistance gage was reported by Leszynsk⁽⁴⁾. This gage employed flame-sprayed and plasma-arc-deposited gage alloys. The gages were deposited through a mask on ceramic substrates. Although gages made by this means were operative, major problems were encountered. These problems stemmed from high porosity and poor ductility. Apparently the metallic particles were to some extent attached to each other by oxide bonds. Only by hot-hydrogen-reduction techniques could the gage grids be fused into continuous conductors.

Still other attempts have been made to make high temperature resistance strain gages by physical vapor deposition of the sensing alloys or elements through masks on ceramic substrates. Bray and Calcaelli⁽⁵⁾ report success using the vacuum deposited films of platinum and chrome-silicon.

Successful gages were made where film thicknesses exceeded 1000 Å. Another attempt to deposit high temperature evaporated film gage was made by D.F.A. MacLachlan⁽⁶⁾. He evaporated platinum resistance grids sandwiched between layers of silicon-monoxide.

With the development of new equipment these approaches may open up attractive new possibilities. Both sputtering and electron beam vaporization techniques offer new techniques for making high-temperature gages.

Out of all of the above approaches, only the wire gages are currently available commercially for the temperature range between 900 F and 1200 F. Most of these gages make use of platinum-tungsten alloy wire sensors. The gages are installed by flame spraying high purity alumina as an insulating substrate, then covering the wire grid with the same material. One different approach is the Microdot SG 420 or SG 423 gage. Among those commercial gages that have been evaluated, the Microdot gage has been reported⁽⁷⁾ to provide some of the best combinations of characteristics for sodium environments. The gage is of a half-bridge design, and also employs platinum-tungsten wire sensing elements. One element is the dummy element and provides temperature compensation. The elements are hermetically sealed and sheathed in an Inconel X-750 strain tube. The insulation is magnesium-oxide tightly packed around the sensing wires. However, since silver brazing is used to join the parts of the gage and the leads, the liquid sodium metal attacks these joints. Electroplating the gage assembly with nickel and nichrobrazing provided some limited protection. For extended periods of time, huts⁽⁸⁾ of stainless steel were required to protect the gage assembly. For predictable results it was necessary to spot weld the Microdot gages to large shims and then both thermally and mechanically cycle the gages. After the gages are thus stabilized, the gage and shim assembly is removed from the calibration bar. Each gage requires individual calibration⁽⁹⁾. The investigations cited above were conducted with the SG-423; however, the new

SG-425 gage has overcome many of the problems associated with the older design. The new gage has eliminated the silver brazed joint between the strain tube and the weldable flange. The two parts are now TIG welded. Also the sensing element, after being enlarged in diameter, continues on back to an evacuated and TIG welded joint between the gage and the stainless steel sheathed lead wire.

The selection of platinum-tungsten alloys for the above commercial gages was based upon extensive investigation in England and the United States. Bertodo^(10,11,12) and Easterling^(13,14) in England and recently Bean⁽¹⁵⁾ in the United States report on the development of platinum-base alloys for strain gages.

Most alloy systems were rejected for very high temperature applications because of either poor oxidation characteristics, phase changes including ordering and disordering effects, or excessively high TC's with low specific resistivity⁽¹⁴⁾.

The nickel-base alloys had received much attention in the early high temperature strain gage work, with Nichrome V (80% Ni, 20% Cr) being one of the more important of the early strain gage alloys. Also, additions of aluminum and iron to the nickel-chromium resulted in Karma-type alloys. The nickel content was reduced to 73-76%, and aluminum and usually iron made up the balance. The aluminum tends to provide a protective coat against further oxidation at high temperatures and the iron adds to the corrosion resistance⁽¹⁶⁾. However, because of phase changes, neither of these alloys can be recommended for static strain measurements in the 1200 F region.

The dilute platinum-tungsten-base alloys have emerged as superior to many others since they are single phase and provide excellent oxidation resistance. Considerable experimental work has demonstrated that between 8 and 9 percent of tungsten seems to provide the optimum proportion. Other additions to platinum have been recommended by Bertodo⁽¹⁷⁾ for high temperature applications; these include palladium and molybdenum.

Some recent unpublished work has indicated that the tungsten at very high temperatures will selectively oxide, leaving a somewhat porous structure having the properties of pure platinum. Other problems with the platinum-tungsten alloys are sometimes associated with a mismatch in thermal

coefficients of expansion. The platinum alloys have a comparatively low coefficient of expansion as compared to many of the refractory alloys.

Another approach to obtain a suitable high temperature alloy for strain gage applications was begun by investigating various alloys in the iron-chromium-aluminum systems. In the initial work eleven alloys were prepared^(18,19). Among those investigated, the one now called Armour D, seemed to provide some of the more promising characteristics. This alloy had a nominal composition of 70Fe-20Cr-10Al. Larger amounts of aluminum tend to increase oxidation resistance, but tend to interfere with the workability of the material. It has been said that at least 20% chromium and 6% aluminum would be needed to provide adequate oxidation resistance. Gages were fabricated from these alloys at the General Electric Company at Evendale, Ohio. Also, since that time, other investigators have used these materials^(3,20).

Recently, a concerted effort has been made at LMEC by Lemcoe et al⁽²⁰⁾ to develop a useful gage from the Armour D alloy. Since Armour D has a negative coefficient of resistance, an advantage was made of this characteristic to produce, in addition to single gages, two and three element gages in which materials having positive coefficients of resistance are used to produce a balancing effect of the apparent strain. These gage variations included a three-element gage in which platinum wire was used for two outside loops and the Armour D for the inside loops. Another approach was to use positive TC platinum for lead tabs. Still another variation made use of a coating of platinum over the Armour D to effect compensation.

All of the above gages seemed to offer some promise. A much better match in coefficient of expansion can be effected by Armour D alloy with austenitic stainless steels than with platinum alloys. Excellent temperature compensation was obtained using Armour D and platinum wire. Also, with these gages an unusually low drift rate was obtained after a stabilization period of 16 hours at 1200 F.

With the increased need for suitable high temperature strain gages for atomic reactors, jet engines, and re-entry vehicles, additional work in alloy improvements and gage design is in order.

Capacitance Strain Gage System

One high temperature strain gage system was reported using electrical capacitance as the means of transducing the strain⁽²¹⁾. This is one of the recent strain gage developments and was first initiated to attempt to bypass the formidable technical obstacles associated with the electrical resistance strain systems.

The latest design of the transducer is composed of a stress frame having a nominal one-inch rhombic configuration employing a capacitance wafer composed of four stainless steel plates separated by five mica-dielectric insulators. The geometry of the stress frame is such that mechanical strain amplification is obtained. It is reported that 3-5 pounds of force is required to apply strains of 5000 microinches. The gage is attached by small, spot welds through stainless steel shims located at diagonal corners.

Although the measurement of capacitance is much more difficult than the measurement of resistance, strain measurements were indicated to 1500 F using these gages to within ± 5 to 10 percent of the readings at room temperature. Additional work has been in progress to improve the readout equipment.

High-Temperature Lead Wires

The literature search provided only a minimum of references on lead wires. However, the lead wires are most important to the overall system. Almost all references agreed that a three-wire system was necessary. Some authors suggested that the lead wires should have the same alloy composition as the gages^(16,22). This, of course, would be impractical where long leads are involved since the overall resistance could become too great. The advantage of the same alloy would be to help avoid thermoelectric effects. Dykes⁽¹⁶⁾ and others have suggested that the 1100 F stainless steel clad cooper wire be used with woven glass insulation. He mentions that above 1100 F Nichrome V and Karma wire be

used. A further recommendation is that a stainless steel sheath with magnesium oxide insulation be used. Almost all investigators recommend that lead tabs be used since it is ordinarily impossible to join the fine gage wire to the leads. Some suggested Nichrome V^(15,20). Others employed lead-tab materials iron-chromium-aluminum, and platinum foil⁽²⁰⁾.

The Microdot gage S-G 423 with the continuous leads uses solid copper wire in a stainless steel sheath with magnesium oxide insulation. In this special usage a hermetic seal with argon atmosphere is maintained. Broad⁽²³⁾ in his investigation of static strain measurements under rapid heating, made use of 30 S.W.G. solid core nickel plated copper wire with fiber glass insulation.

It is considered important for many applications to be able to monitor the gage lead resistance so that it is known. The wires should be attached to the structure so that the thermal condition can be held or at least repeated.

Lead Wire Attachments

Most lead wires for high temperature applications are spot welded by capacitance discharge techniques. Other techniques have been investigated such as lasers. The lasers for welding are quite expensive and difficult to control the weld on fine size wires. The laser beams are reported to either burn holes or go out of control⁽²⁴⁾.

Other techniques are worth considering such as TIG (tungsten-inert-gas) welding^(25,26). Equipment is commercially available for making miniature welded joints using a binocular microscope. Excellent control can be exercised with this equipment. Oxidation of the materials being joined is minimized because of the blanket of inert gas.

Another means that offers possibilities is plasma-needle-arc welding equipment^(27,28,29). Here the arc length is not as critical as for the TIG welding technique. Equipment is available commercially for both techniques.

Although discharge welding is convenient and can make excellent joints the nugget formed by the resistance heating can be the source of

a stress concentration and can cause microscopic fatigue cracks, which in turn, cause resistance changes. Some of the principal causes of gage failure can be traced to the spot welded joints between the fine sensor wires and the lead tabs⁽²⁰⁾.

Insulation and Bonding Techniques

One of the more important considerations in a successful high temperature electrical strain gage system is the means employed for insulation and bonding. If the resistance-to-ground drops too low, excessive errors can result.

A desirable insulation material should be impervious to gases and liquids, should transmit all of the strain from the structure to the sensing alloy and maintain its dielectric properties. Ideally, it would have the same coefficient of thermal expansion as the substrate. It should be able to withstand thermal shock especially on cooling.

A number of types of insulators and bonding agents are available; these include (1) ceramic cements, (2) ceramic glazes, and (3) flame or plasma sprayed metal oxides.

The ceramic cements are often two part materials containing a powder composed of a mixture of materials of some or all of the following: alumina, silicates, magnesium oxide, and chromium oxide. The liquid binders are usually phosphates or phosphoric acid⁽²²⁾. The coating thicknesses of these cements are critical since water must be released⁽³⁰⁾. Also, the curing rates and temperatures are critical.

A ceramic glaze type coating Brimor U-529 was developed by the Protective Coatings Section of the Engineering Research and Development Laboratory at Fort Belvoir, Virginia⁽³⁰⁾. This is applied in thin coats, is dried at 150 and fired at 1550 F. This coating results in a glossy refractory coating of very high hardness and excellent adhesion. When used to apply wire gages, the coating was reported to provide excellent protection from abrasion and was a good dielectric.

A ceramic material that appears to be used in a slurry form and dried is called Glass Rock. This material was mentioned twice in

this search as having the best high temperature dielectric properties of all of the materials tested.

The Rokide Process for applying wire strain gages was probably the most often mentioned and is apparently the most used^(31,32).

Thermospray processes using powders and plasma jet systems were also employed. One of the problems using the Rokide process is to learn the art of making successful and uniform gage applications. Also, problems are found in connection with adhesion of the coating to the substrate. A much better bond was obtained by first putting down a substrate of nickel aluminide before flame spraying the alumina.

Mention was made of using a laminating technique with the alumina and stainless steel. The objective here was to improve the thermal properties. An interesting Slavic reference described a flame-spraying powder mixture composed of 93-96 percent alumina by weight with the remainder phosphorous pentoxide⁽³³⁾. It was alleged that a substrate made by this manner had unusual adhesion and dielectric properties.

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