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DMIC Memorandum 203

RECENT INFORMATION ON LONG-TIME CREEP DATA FOR COLUMBIUM ALLOYS

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Director

RECENT INFORMATION ON LONG-TIME CREEP DATA FOR COLUMBIUM ALLOYS

J. J. English and E. S. Bartlett*

Introduction

This memorandum summarizes recent information relating to long-time creep parameters for columbium alloys. The available creep data for columbium alloys through mid-1963 were previously reported in DMIC Memorandum 170.

Long-time-creep test programs are being conducted by NASA-Lewis Research Center, Thompson Ramo Wooldridge, and Oak Ridge National Laboratory. Data from these and other selected sources for times greater than 10 hours are compiled in this memorandum.

Comparisons among many of the creep data are obscured by uncertainties relating to material parameters (i.e., was the material stress relieved, recrystallized, or processed according to some "optimized" schedule?). Other uncertainties accrue from methods of testing and reporting. Most frequently, external readout of creep strain (dial gages or differential transformers) is used rather than direct optical measurements of strain gages mounted on reduced sections. The accuracy of compensation for heterogeneous creep throughout the specimen is questionable in some cases, even when such corrections are made. In addition, at least two laboratories, CANEL and Martin-Denver, have reported observation of anisotropic behavior in columbium alloys related to creep straining. Such observations could be most important in compensating for external readout techniques. In most sources, it is not clear whether creep deformation includes elastic as well as plastic deformation.

NASA-Lewis Research Center

Table 1 gives some creep data obtained by NASA.[1] Test apparatus capable of a vacuum about 10^{-9} torr was used in the investigation. Creep strain was measured by direct optical techniques. Although limited, these data do provide some indication of relative long-time creep behavior of most columbium alloys of current interest.

Thompson Ramo Wooldridge

TRW is conducting long-time creep tests in the 10^{-9} to 10^{-10} torr pressure range under NASA sponsorship.[2] Direct optical readout of creep strain is being used. Although the only columbium alloys scheduled for creep analysis are the high-strength forging alloys Cb-132M (Cb-20Ta-15W-5Mo-1Zr-0.1C) and AS-30 (Cb-20W-1Zr-0.1C), TRW has conducted a preliminary equipment check using the FS-85 alloy (Cb-28Ta-10W-1Zr). The FS-85 test specimens were annealed at 2600 F for 1 hour before testing. Creep results at 2000 F for a load of 7000 psi are shown in Figure 1. A tabulation of dimensional changes for FS-85 at 2000 F and 4000-psi stress is given in Table 2. The physical properties determined for FS-85 as a result of the strain measurements are footnoted in Table 2. The generally good agreement with previously reported data attests to the accuracy of the strain measurements.

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The initial test on a specimen machined from AS-30 plate in the stress-relieved condition was conducted at 2000 F and 12,000 psi. Figure 2 gives the creep results for 800 hours' duration.

Under Navy sponsorship,[3] TRW has examined the creep behavior of B-66 (Cb-5Mo-5V-1Zr) and D-43 (Cb-10W-1Zr) when coated with the Pfaudler modified silicide and TRW Cr-Ti-Si coatings. The specimen deformation was not monitored during the tests. Elongation (plastic deformation) was determined by measurement before and after test. The tests were conducted in air; so, the test life of the specimens was dependent on the protectiveness of coatings under stress as well as the creep-rupture characteristics of the two alloys. The cross-sectional area of the uncoated specimens was used to calculate the stresses in these tests. Tables 3 and 4 give the results of these tests. These results demonstrate the importance of considering the behavior of the coated substrate when the columbium alloy is to be used in an oxidizing environment. The TRW data show that both the coating and coating process can affect creep behavior. For example, premature fractures occurred at 1600 F and low stresses for coated D-43 and B-66. These failures at the low temperature were not caused by normal creep rupture but by the inability of the coatings to protect the columbium alloys from oxidation and contamination. In the unstressed condition, the protective lives of these coatings at 1600 F averaged about 100 hours. However, in the stressed condition, coating protection was degraded at 1600 F, presumably by coating crack formation and the absence of a viscous oxide product. At the higher temperatures, better coating performance was obtained in the stressed condition. Also, the TRW data show that the creep behavior of an alloy is dependent on the coating and coating process used to protect the alloy from oxidation.

Union Carbide Corporation

Union Carbide determined the creep properties of heavily worked and stress-relieved 0.065-inch-thick D-43 sheet, and lightly worked and stress-relieved 0.068-inch-thick B-66 sheet.[4] Test equipment included a vacuum capability of 10^{-7} to 10^{-8} torr. An externally mounted dial indicator was used to measure strain. The creep values reported in Figures 3 through 10 reflect plastic deformation only. These design-type data show a greater (negative) slope for B-66 in the condition tested than for D-43. As a result, B-66 appears the more attractive material for very short-time (<1 hr) strength, and D-43 appears superior at longer times. Despite the more severe cold work, D-43 was much more resistant to recrystallization during testing than B-66, according to this investigation.

References

(1) Hall, R. W., and Titran, R. H., "Creep Properties of Columbium Alloys in Very High Vacuum", Report TP 15-63, NASA-Lewis Research Center, Cleveland, Ohio (December 9-10, 1963).

(2) Sawyer, J. C., and Philleo, C. H., "Generation of Long-Time Creep Data of Refractory Alloys at Elevated Temperatures", Report CR-54123, Fourth, Fifth, and Sixth Quarterly Reports, Thompson Ramo Wooldridge, Inc., Cleveland, Ohio, under Contract NAS-3-2545 (March 26 through December 26, 1964).

(3) Warmuth, D. B., "Design Data Study for Coated Columbium Alloys", Final Summary Technical Report ER-5885, Thompson Ramo Wooldridge, Inc., Cleveland, Ohio, under Contract NOw 63-0471-c (April 1, 1964).

(4) Stephenson, R. L., "Comparative Creep-Rupture Properties of D-43 and B-66 Alloys", Report ORNL-TM-944, Union Carbide Corporation, Oak Ridge National Laboratory, Oak Ridge, Tennessee, under Contract W-7405-eng-26 (November, 1964).

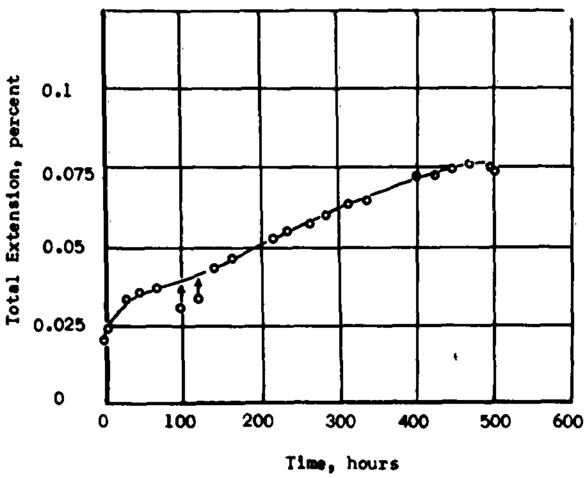

FIGURE 1. CREEP DATA FOR COLUMBIUM ALLOY FS-85, 2000 F, 7000 PSI, <10^{-8} TORR, 1 HOUR PRIOR ANNEAL AT 2600 F

TABLE 1. CREEP PROPERTIES OF SOME COLUMBIUM ALLOYS (NASA-LEWIS DATA)

Alloy	Condition	Stress, psi	Temp, F	Time, hours	Total Elongation, percent
FS-85	Recrystallized	10,000	2000	1400	2
FS-85	Recrystallized	4,000	2200	300	0.26
FS-85	Recrystallized	4,000	2400	300	3.35
D-43	Stress relieved	10,000	2000	230	2
D-43	Recrystallized(a)	10,000	2000	510	2
D-43	Recrystallized	4,000	2200	300	0.30
D-43	Recrystallized	4,000	2400	300	1.26
Cb-1Zr	Recrystallized	10,000	2000	7	2
Cb-1Zr	Recrystallized	4,000	2000	300	1.10
Cb-1Zr	Recrystallized	4,000	2200	235	15.11
Cb-752	Recrystallized	4,000	2000	300	0.53
Cb-752	Recrystallized	4,000	2200	300	3.57
Cb-752	Recrystallized	4,000	2400	300	12.00
B-66	Recrystallized	4,000	2000	300	0.18
B-66	Recrystallized	4,000	2200	300	1.42
B-66	Recrystallized	4,000	2400	124	12.33
C-129	Recrystallized	4,000	2000	300	1.05
C-129	Recrystallized	4,000	2200	300	3.64
AS-55	Recrystallized	4,000	2000	300	0.31
AS-55	Recrystallized	4,000	2200	300	3.82
D-14	Recrystallized	4,000	2000	300	3.05
D-14	Recrystallized	4,000	2200	72	8.40
B-33	Recrystallized	4,000	2000	300	8.60
B-33	Recrystallized	4,000	2200	255	33.9

(a) Slightly deformed grains remained. This material probably supplied in Du Pont's "optimum" condition, i.e., solution treated at 3000 F, cold worked 25 percent, and aged at 2600 F.

TABLE 2. DIMENSION CHANGES FOR FS-85 AT 2000 F AND 4000 PSI

Time, hr	Reference Length, in.	Creep, %
0-cold	2.00163(a)	--
0-hot	2.01925(a)	--
0-loaded	2.01994	--
96	2.02004	0.005
161	2.02012	0.009
190	2.02016	0.011
304	2.02051	0.029

(a)

Parameter	Value Calculated From Above Data	Previously Reported Value
Thermal expansion coefficient	4.04×10^{-6}/F	4.8×10^{-6}/F
Elastic modulus	10×10^{6} psi	18×10^{6} psi

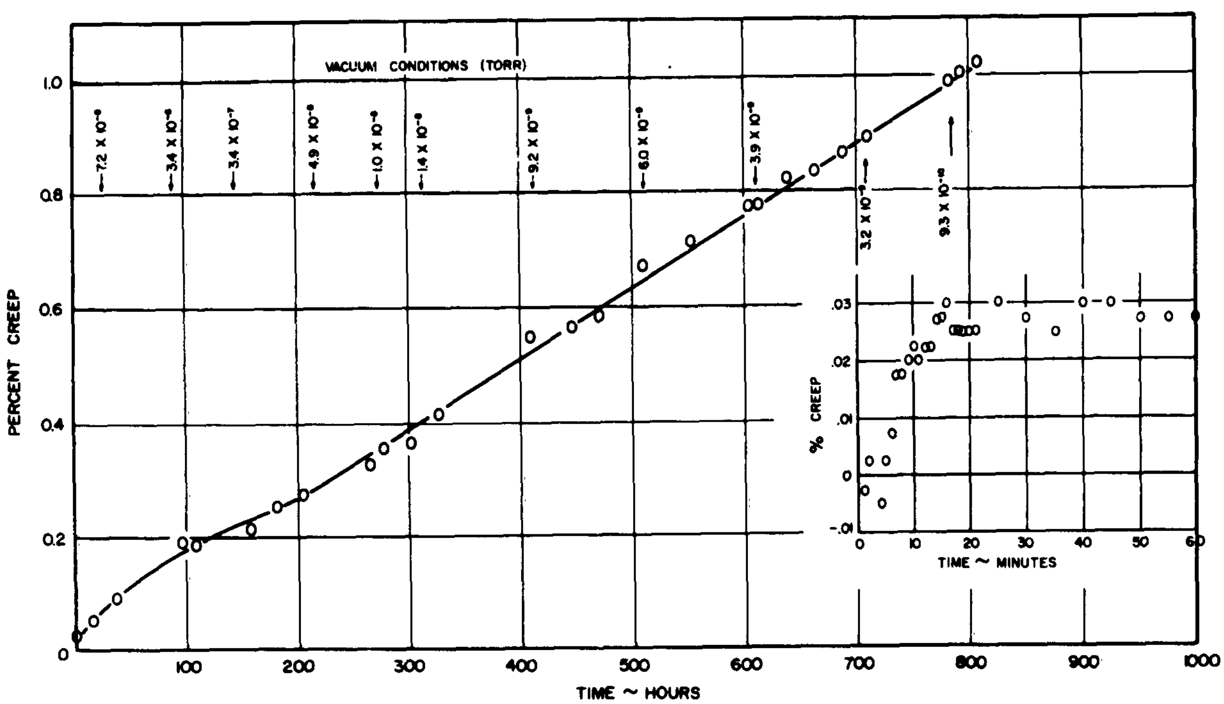

FIGURE 2. CREEP OF STRESS-RELIEVED AS-30 TESTED AT 2000 F; 12,000 PSI

TABLE 3. CREEP-TEST RESULTS FOR PFAUDLER SILICIDE AND TRW Cr-Ti-Si COATED D43

		Pfaudler Coating		TRW Coating	
Test Temp, F	Stress, psi	Test Duration, hours	Elongation in 1 Inch, percent	Test Duration, hours	Elongation in 1 Inch, percent
1600	10,000	16.5	0	—	—
		18.6	Fractured	—	—
	15,000	27.6	Fractured	—	—
	20,000	17.2	Fractured	—	—
	21,400	24.0	0	—	—
	25,000	16.8	0	—	—
		20.1	Fractured	—	—
	27,000	—	—	24.0	0.9
		—	—	49.4	1.3
		—	—	61.1	Fractured
		—	—	70.8	Fractured
	27,500	8.1	Fractured	—	—
		12.1	0.1	—	—
		16.0	0.3	—	—
	29,350	4.9	Fractured	8.0	1.1
		7.0	0.3	24.0	2.6
		8.4	Fractured	52.0	Fractured
		11.9	Fractured	60.5	Fractured
		—	—	64.7	Fractured
		—	—	65.2	2.7
	31,000	—	—	2.0	0.7
		—	—	6.0	1.8
		—	—	24.0	2.7
		—	—	48.1	3.8
		—	—	73.3	5.2
	31,750	—	—	4.0	0.9
		—	—	5.0	2.0
		—	—	6.0	0.8
		—	—	9.2	Fractured
		—	—	12.0	3.2
		—	—	24.0	3.3
		—	—	46.8	Fractured
		—	—	55.2	Fractured
	32,500	—	—	0	Fractured
	34,700	2.6	Fractured	—	—
		3.0	0.5	—	—
		3.2	Fractured	—	—
		5.0	0.7	—	—
		6.3	Fractured	—	—
		9.0	0.6	—	—
	37,000	1.2	0.4	—	—
		1.6	Fractured	—	—
		2.0	1.7	—	—
		4.1	0.7	—	—
		6.0	0.5	—	—
2000	10,000	—	—	65.6	1.2
	11,000	—	—	94.6	1.5
	11,700	—	—	16.0	1.7
		—	—	24.0	1.6
		—	—	32.0	1.2
		—	—	48.1	3.3
		—	—	65.0	2.0
		—	—	89.4	3.8
		—	—	100.0	5.2
	12,500	40.1	0.6	6.5	0.5
		—	—	16.4	1.2
		—	—	24.3	1.6
		—	—	40.4	4.1
		—	—	48.1	3.0
		—	—	65.0	5.4

TABLE 3. (Continued)

		Pfaudler Coating		TRW Coating	
Test Temp, F	Stress, psi	Test Duration, hours	Elongation in 1 Inch, percent	Test Duration, hours	Elongation in 1 Inch, percent
2000	15,000	24.3	0.4	2.0	0.4
		48.0	1.2	4.0	1.3
		72.0	3.8	6.0	1.4
		75.7	4.8	10.0	3.1
		94.8	3.1	16.4	5.0
	17,500	12.1	0.5	1.2	0.7
		24.1	1.3	3.0	1.6
		32.0	2.1	4.0	1.8
		48.4	1.9	5.0	3.9
		52.1	1.9	6.0	2.3
		65.0	6.0	6.4	2.8
		—	—	8.0	7.8
	20,000	6.1	0.6	—	—
		16.0	1.0	—	—
		16.0	2.1	—	—
		22.1	1.6	—	—
		24.1	1.4	—	—
		40.0	5.7	—	—
	22,500	1.8	0.8	—	—
		3.0	0.6	—	—
		3.0	5.8	—	—
		4.1	4.1	—	—
		5.1	1.0	—	—
	25,000	4.3	14.9	—	—
2300	3,000	—	—	65.0	2.4
	4,000	52.1	Fractured	16.0	0.9
		—	—	24.0	1.7
		—	—	40.0	2.7
		—	—	47.0	3.4
		—	—	65.0	3.7
		—	—	82.0	4.7
		—	—	100.0	6.9
	5,000	13.7	0.6	8.0	0.5
		24.0	1.2	20.0	1.9
		32.0	2.0	24.0	2.4
		40.1	Fractured	32.0	2.5
		48.2	6.0	65.0	5.2
		49.7	Fractured	—	—
	7,500	6.0	0.5	2.0	1.0
		16.0	1.5	4.0	1.9
		17.1	1.6	6.0	1.5
		24.0	2.8	6.0	2.5
		24.2	3.8	8.0	2.7
		42.1	6.2	16.0	3.9
		—	—	20.0	5.4
	10,000	2.0	0.7	0.5	1.7
		4.0	1.6	1.0	1.9
		6.0	2.2	2.0	2.9
		8.0	3.0	3.0	3.6
		12.0	3.8	4.0	4.7
		13.5	5.0	—	—
		18.8	5.9	—	—
		24.0	6.8	—	—
	12,000	1.0	0.2	—	—
		2.0	0.9	—	—
		3.0	1.7	—	—
		4.0	2.2	—	—
		5.0	4.4	—	—
		6.0	5.0	—	—

TABLE 4. CREEP-TEST RESULTS FOR PFAUDLER SILICIDE AND TRW Cr-Ti-Si COATED B66

		Pfaudler Coating		TRW Coating	
Test Temp, F	Stress, psi	Test Duration, hours	Elongation in 1 Inch, percent	Test Duration, hours	Elongation in 1 Inch, percent
1600	30,000	6.0	0.1	—	—
		24.0	0.2	—	—
		44.9	Fractured	—	—
		66.0	0.2	—	—
		100.2	0.1	—	—
	34,000	0	Fractured	—	—
		8.8	Fractured	—	—
		16.1	0.1	—	—
		20.4	Fractured	—	—
		24.3	Fractured	—	—
		24.4	0.5	—	—
		40.3	0.2	—	—
	35,500	16.0	0.2	—	—
		24.0	0.1	—	—
		47.5	0.1	—	—
		48.0	0.5	—	—
	37,400	3.0	0.3	65.2	0.5
		3.3	Fractured	—	—
		4.7	Fractured	—	—
	41,350	1.1	0.3	1.0	0.4
		2.2	0.2	6.0	0.6
		3.0	0.3	24.0	1.1
		4.2	0.2	69.4	1.3
		4.7	Fractured	94.0	1.3
		6.0	0.1	—	—
		12.0	0.2	—	—
	45,000	—	—	2.0	0.9
		—	—	6.0	1.0
		—	—	24.3	1.8
		—	—	48.3	2.5
		—	—	72.2	2.3
	46,700	—	—	0.5	0.6
		—	—	2.0	1.1
		—	—	6.0	1.6
		—	—	22.7	2.2
		—	—	24.0	2.9
		—	—	26.0	1.7
		—	—	34.5	Fractured
	48,000	—	—	0.5	1.0
		—	—	1.0	1.4
		—	—	1.7	Fractured
		—	—	2.0	1.0
		—	—	2.2	Fractured
		—	—	3.1	Fractured
		—	—	6.2	1.9
2000	10,000	—	—	67.0	0.8
	12,500	—	—	65.3	2.6
		—	—	112.7	3.7
	13,500	—	—	16.2	0.6
		—	—	40.0	1.0
		—	—	65.3	3.0
		—	—	94.3	4.2
	15,000	23.8	0.6	16.0	1.1

TABLE 4. (Continued)

		Pfaudler Coating		TRW Coating	
Test Temp, F	Stress, psi	Test Duration, hours	Elongation in 1 Inch, percent	Test Duration, hours	Elongation in 1 Inch, percent
2000	15,000	43.2	1.1	24.0	1.7
		53.0	1.5	40.3	3.8
		68.2	3.2	50.9	4.4
		96.1	4.1	66.5	4.6
		—	—	76.0	5.0
	17,500	12.1	0.8	4.1	0.5
		24.2	1.5	8.5	1.1
		32.3	2.2	16.0	1.8
		40.5	3.3	18.0	2.7
		52.0	6.8	20.1	3.4
		—	—	25.1	4.8
	20,000	6.0	0.8	6.0	1.7
		12.4	2.2	—	—
		16.0	3.7	—	—
		23.0	4.0	—	—
		32.0	7.5	—	—
	22,500	4.2	0.9	2.1	0.5
		8.0	2.2	4.2	2.0
		10.0	2.9	6.0	3.5
		13.0	6.7	6.1	3.8
		16.6	6.1	8.0	4.2
2300	3,000	—	—	65.0	2.3
	4,000	—	—	16.2	0.7
		—	—	28.1	0.7
		—	—	40.0	3.3
		—	—	47.0	3.7
		—	—	56.0	2.7
		—	—	65.3	4.4
	5,000	6.0	0.7	8.0	0.7
		8.0	1.2	16.0	1.7
		16.0	1.3	20.0	2.4
		19.5	1.7	25.6	3.4
		25.5	3.2	32.0	4.7
		30.0	3.5	65.0	7.3
	6,000	4.2	0	—	—
		6.0	0.4	—	—
		12.0	2.2	—	—
		13.0	1.9	—	—
		16.2	2.4	—	—
		20.0	4.5	—	—
		24.0	6.0	—	—
	7,500	4.0	1.4	3.0	0.7
		6.0	2.5	4.0	1.4
		8.0	3.1	5.0	2.0
		8.0	3.5	6.0	3.1
		12.0	5.3	7.0	4.3
		16.0	5.7	8.0	5.1
	10,000	2.0	0.9	0.5	0.1
		3.0	2.2	1.0	1.4
		3.5	2.7	1.5	1.4
		4.0	4.3	2.0	3.8
		5.1	6.2	2.5	5.0
		—	—	3.0	6.5
		—	—	4.0	7.0

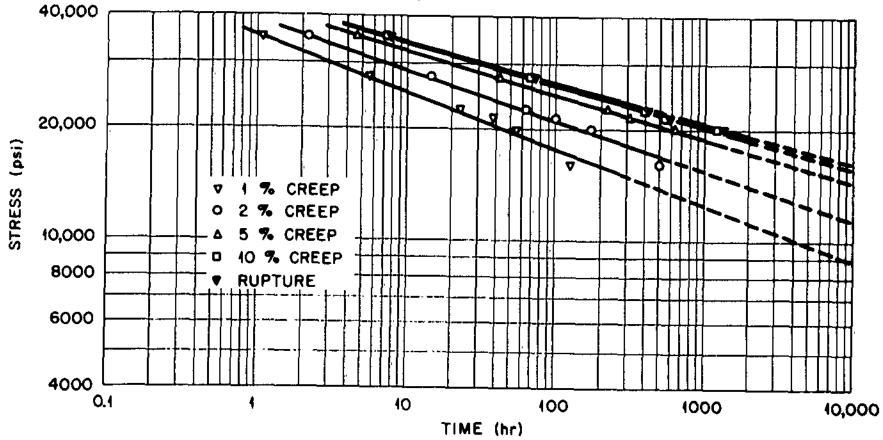

FIGURE 3. CREEP-RUPTURE PROPERTIES OF D-43 AT 1800 F

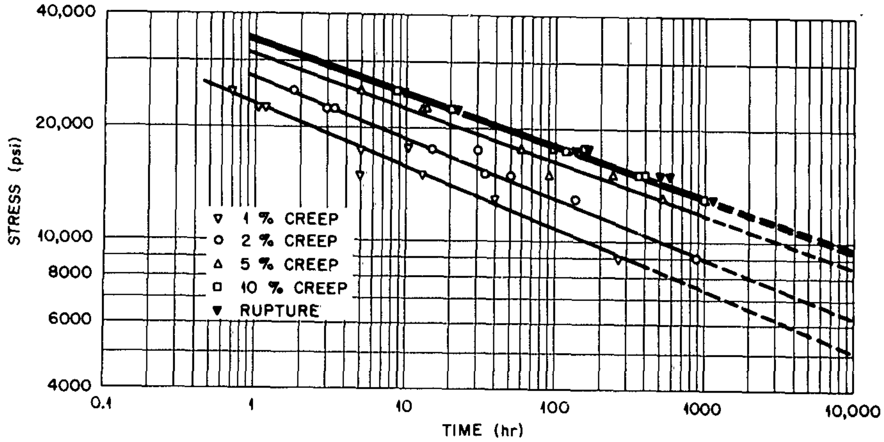

FIGURE 4. CREEP-RUPTURE PROPERTIES OF D-43 AT 2000 F

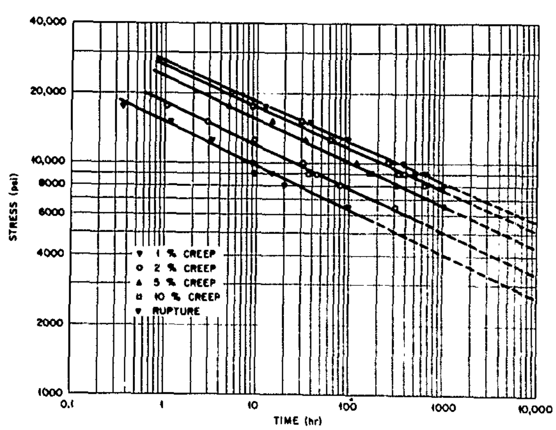

FIGURE 5. CREEP-RUPTURE PROPERTIES OF D-43 AT 2200 F

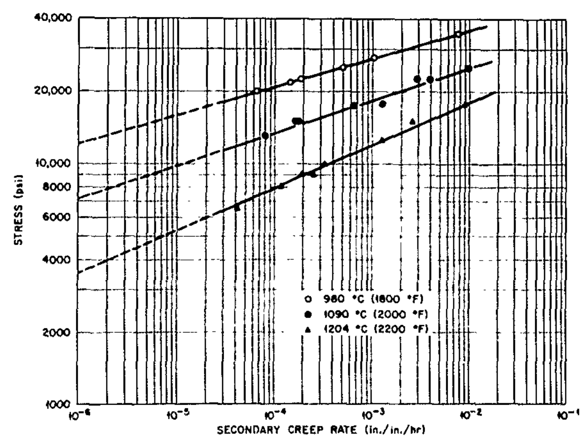

FIGURE 6. SECONDARY CREEP RATE VERSUS STRESS FOR D-43

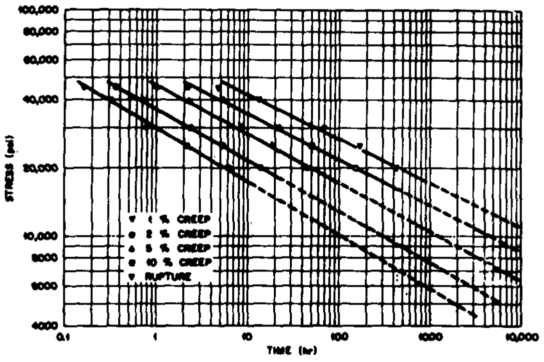

FIGURE 7. CREEP-RUPTURE PROPERTIES OF B-66 AT 1800 F

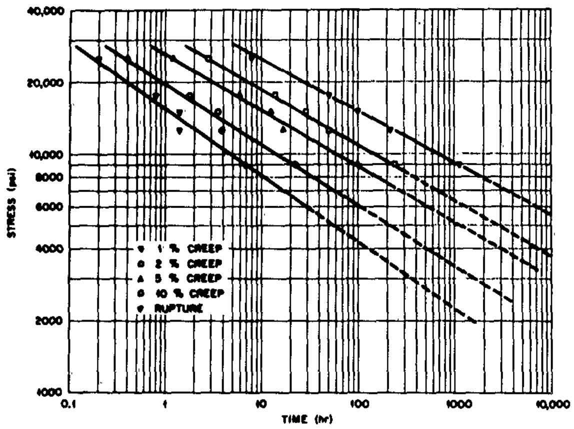

FIGURE 8. CREEP-RUPTURE PROPERTIES OF B-66 AT 2000 F

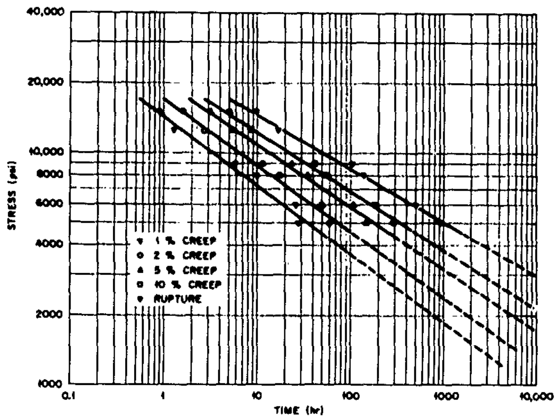

FIGURE 9. CREEP-RUPTURE PROPERTIES OF B-66 AT 2200 F

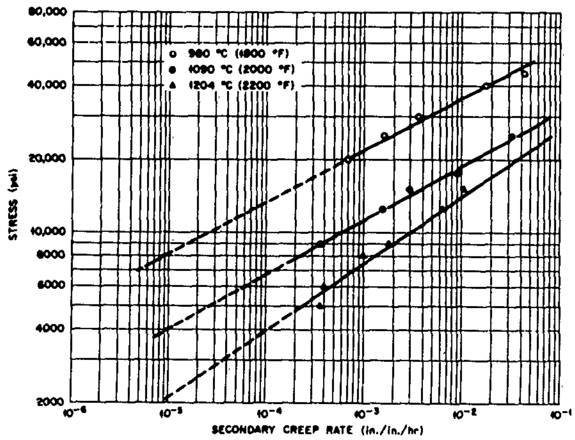

FIGURE 10. SECONDARY CREEP RATE VS STRESS FOR B-66

Unclassified

Security Classification

DOCUMENT CONTROL DATA - R&D

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13. ABSTRACT

This memorandum summarizes recent information relating to long-time creep parameters for columbium alloys. The available creep data for columbium alloys through mid-1963 were previously reported in DMIC Memorandum 170. Long-time-creep test programs are being conducted by NASA-Lewis Research Center, Thompson Ramo Wooldridge, and Oak Ridge National Laboratory. Data from these and other selected sources for times greater than 10 hours are compiled in this memorandum.

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14. KEY WORDS	LINK A ROLE	LINK A WT	LINK B ROLE	LINK B WT	LINK C ROLE	LINK C WT
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Columbium alloys	9	3				

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