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HIGH GAS VELOCITY OXIDATION AND HOT CORROSION TESTING OF OXIDE DISPERSION-STRENGTHENED NICKEL-BASE ALLOYS

by Daniel L. Deadmore, Carl E. Lowell, and Gilbert J. Santoro Lewis Research Center Cleveland, Ohio 44135 November, 1975

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Several oxide dispersion strengthened (ODS) nickel-base alloys were tested in high velocity gases for cyclic oxidation resistance at temperatures to 1200° C and times to 500 hours and for hot corrosion resistance at 900° C for 200 hours. Nickel-chromium-aluminum ODS alloys were found to have superior resistance to oxidation and hot corrosion when compared to bare and coated nickel-chromium ODS alloys. The best of the alloys tested had compositions of nickel - 15.5 to 16 weight percent chromium with aluminum weight percents between 4.5 and 5.0. How- ever, all of the nickel-chromium-aluminum ODS materials experienced small weight losses (<16 mg/cm ²).						
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DISPERSION-STRENGTHENED NICKEL-BASE ALLOYS

by Daniel L. Deadmore, Carl E. Lowell, and Gilbert J. Santoro

Lewis Research Center

SUMMARY

Several thorium and yttrium oxide dispersion strengthened (ODS) nickel-chromium-aluminum alloys were tested in Mach 0.3 and Mach 1 gases at 1100° to 1200° C for up to 500 one-hour cycles. Some of the alloys were also tested at 900° C in high temperature Mach 1 gases containing 5 ppm synchetic sea salt. Thoriated nickel-chromium-aluminum (TD-NiCrAl) was found to be less attacked, under all conditions, than either of two bare or coated Ni-Cr ODS alloys, IN-MA-754 and DS-NiCr.

A TD-NiCrAl alloy was also compared to yttria dispersion strengthened alloys (YD-NiCrAl) with two yttria and various aluminum levels in Mach 1 gases. All of the alloys were found to have small (<16 mg/cm²) weight losses after 500 one-hour cycles at 1100° C. As the aluminum content was increased, the final weight loss decreased, although no effect of the yttria level (0.86 to 1.15 weight percent) could be detected. Thermal fatigue cracking increased with increasing aluminum content. The best compromise of high velocity cyclic oxidation resistance and thermal fatigue resistance seemed to be an alloy of less than five but greater than four and one-half weight percent aluminum. However, after 400 cycles to 1100° C, the rate of weight loss for all nickel-chromium-aluminum ODS alloys increased markedly, indicating lifetimes not greatly exceeding 500 cycles under these test conditions.

INTRODUCTION

Oride dispersion strengthened (ODS) nickel-base alloys are being considered for application as high temperature (1100° C) vane materials in advanced gas turbine engines. Projected service life requirements range from 500 to 3000 hours or more, depending on application ODS alloys appear promising for vane service on the basis of strength. However, there is some question as to their oridation, hot corrosion, and thermal fatigue resistances. One of the first ODS alloys evaluated was thoriated nickel 20-chromium (TD-NiCr). While in static tests this alloy was very oxidation resistant, high velocity testing resulted in unacceptably high oxidation attack (ref. 1). The loss of oxidation resistance in high velocity gases is due to the fact that the oxidation resistance depends on the formation of a protective layer of chromium sesquioxide (Cr_2O_3) which reacts with oxygen to form the highly volatile oxide CrO_3 . Under static test conditions the rate of CrO_3 loss is low because vaporization is, to some extent, balanced by condensation. However, in a high velocity gas condensation is minimized and the effective loss rate increases by several orders of magnitude.

In an effort to improve the resistance of NiCr ODS alloys, a class of alloys has been developed based on the nickel-chromium-aluminum system commonly called NiCrAls (ref. 2). These alloys derive their oxidation resistance from the formation of aluminum sesquioxide (Al₂O₃) which is significantly more stable than Cr_2O_3 in high velocity gas streams. The first of these alloys used thorium dioxide (ThO₂) as the dispersoid, but more recent alloys have used yttria (Y₂U₃) in order to avoid the use of a radioactive metal (Th).

The object of the work reported here was to evaluate the environmental resistance of potential ODS vane alloys in high velocity, cyclic oxidation at 1100° C, the expected material use temperature; and in some cases, at 1200° C to provide some data concerning overtemperature capabilities. A few of the alloys were also tested at 900° C with 5 ppm synthetic sea salt injected in the fuel to examine hot corrosion resistance. While most of the testing was done at Mach 1, a few alloys were also tested at Mach 0.3. The material's environmental resistance was judged on the basis of specific weight change, microstructural changes, and thermal fatigue cracking.

MATERIALS AND SAMPLE PREPARATION

Four alloy types and two coatings were used in this program: thoria dispersion strengthened NiCrAls (TD-NiCrAl); an Y_2O_3 dispersion strengthened Ni-Cr alloy (IN-MA-754); a thoria dispersion strengthened Ni-Cr alloy (DS-NiCr); and several yttria dispersion strengthened NiCrAls (Cabot-Stellite experimental alloys A-1 through A-6 and B-1 through B-6). The compositions of the alloys evaluated are shown in table I. Their extrusion conditions and heat treatments (where known) are listed in table II. In addition, samples of DS-NiCr and IN-MA-754 were obtained with vendor supplied NiCrAl experimental coatings.

The TD-NiCrAls were produced under NASA contract NAS3-13490 by Fansteel. The compositions of the alloys were essentially Ni-19 Cr-3.9 Al-2 ThO₂ (3931) and Ni-16 Cr-4.6 Al-2 ThO₂ (3933). These alloys were tested mostly in a fine grained condition. However, a few samples of 3933 with elongated grains were produced by hot rolling and given limited evaluation. These alloys are designated 3933 HR. All of these alloys were solid solutions with some Ni₃Al (γ ') as can be seen from table III.

The IN-MA-754 and DS-NiCr alloys were obtained by courtesy of the General Electric Company. These are commercial ODS alloys that are essentially Ni-20 Cr, although the MA 754 contains small amounts of Al, Ti (titanium) and Fe (iron) (table I). DS-NiCr is strengthened with ThO₂ while IN-MA-754 is strengthened with Y₂O₃. The vendor supplied Ni-Cr-Al coatings on these alloys were intended to be identical. However, while both coatings contained γ' and β (NiAL), the x-ray diffraction pattern from the coatings on MA 754 contained substantially less intense lines of β than did the pattern from the DS-NiCr (table III).

The Cabot Corporation supplied the YD-NiCrAl's under NASA contract NAS3-17806. They were meant to be Ni-15Cr with 4, 5, and 6 percent Al, and with two levels of yttria - nominally 0.8 and 1.2 weight percent. The yttria levels were obtained, but the aluminum contents proved difficult to control as can be seen in table I. However, the desired range of aluminum contents (i.e., from about 4 to 6 percent) was produced and each alloy sample was individually analyzed for Al, Y, and C (carbon). The metallic phases present in these alloys (table III) were similar to those found in the TD-NiCrAl, although γ' was only found in those alloys with greater than 5 percent Al. Unlike the TD-NiCrAls, no diffraction pattern for the dispersed oxide phase was found in these alloys.

All samples were machined into 4 foot by 1 foot by 1/4 inch test bars and oxidation/hot corrosion tested as machined (see refs. 3 and 4 for details of sample shapes). Prior to testing, all samples were weighed to 0.1 milligram.

FROCEDURE

Three series of tests were run. The first were conducted in a Mach 1 test facility for 200 one-hour cycles (see fig. 1 and ref. 3). In this test eight samples at a time are heated in a Mach 1 stream of combustion products of either natural gas or A-1 jet fuel. The specimen holder is rotated to minimize temperature differences among specimens. After an hour of heating, the specimen holder is lowered into a Mach 1 cold air stream to cool for three minutes. The cycle is then repeated. At approximately 15 cycle intervals the samples are removed, weighed, and put back in test. The samples used in this series were the TD-NiCrAls; IN-MA-754, bare and coated; and DS-NiCr, bare and coated. Three sets of tests in this series were completed: 1200° C oxidation using natural gas as the fuel, 1100° C oxidation using natural gas, and 900° C hot corrosion using A-1 fuel containing 5 parts per million synthetic sea salt (ATSM D-1141).

The second series of tests, also for 200 hours, were run in a Mach 0.3 stream of the combustion products of A-1 jet fuel (ref. 4). Both 1100° and 1200° C data were obtained. Only TD-NiCrAl samples were used including hot rolled samples of 3933, i.e., 3933 hours. Data was obtained the same way as in the first series.

The final test series was at 1100° C in the Mach 1 test facility for 500, one-hour cycles. The alloys tested were those obtained from Cabot-

Stellite with 3933 also in the test for comparison with earlier data. Two runs were made: one in A-1 fuel and one in natural gas. Once again samples were weighed approximately every 15 cycles. At the conclusion of each test, the samples were weighed and photographed. Each sample was then mounted in epoxy, transversely section in the hot zone, and examined metallographically.

RESULTS AND DISCUSSION

Mach 1, 200 Hours

The specific weight change results of this series are shown in table IV-A and figures 3 and 4. At 1100° C (fig. 3) the TD-NiCrAls (3931 and 3933) are substantially superior to coated and bare MA 754 and bare DS-NiCr. Only coated Ds-NiCr was comparable to the TD-NiCrAls and it appeared to be starting to lose weight slightly when it had to be removed from test at 169 cycles due to a holder failure. At 1200° C (fig. 4, the differences are greatly accentuated. Even the coated DS-NiCr has lost considerably more weight than the TD-NiCrAls.

The source of the superiority of the TD-NiCrAl alloys over bare DS-NiCr and MA 754 is that the former are protected by nonvolatile Al₂O₂ while the latter rely on Cr₂O₃ which is known to form a volatile, nonprotective oxide in high velocity applications (refs. 1 and 5). The coated alloys also start out with Al₂O₃ as the protective oxide, but a combination of coating-substrate interdiffusion and spalling result in a loss of protection with time (ref. 6). This can be seen in figs. 5 and 6. The microstructures of the coated samples reveal that after 200 cycles at 1200° C, the coatings on both alloys are consumed. At 1100° C, one side of the coated MA 754 has been severely degraded. Even where the coatings have not been severely attacked, considerable porosity exists at the coating substrate interface. There is also porosity present in the TD-NiCrAlS. This was assumed to be Kirkendhal porosity as a result of Al diffusion. Microprobe scans of the samples revealed an aluminum gradient, reinforcing this assumption. This phenomenon can be seen in figure 7. The pores are near the surface and widely scattered. What the effect of these pores is on the mechanical properties is not known, but since the load bearing area is decreased, the pores can be expected to lower such properties.

Exposure to combustion gases containing 5 ppm synthetic sea salt resulted in hot corrosion which was significantly less severe in the case of the TD-NiCrAl than for the other alloys listed in table IV-A (figs. 8 and 9). The coatings were of little help. As can be seen in figure 8(a), the coating on DS-NiCr has almost disappeared. While the coating on MA 754 (fig. 9) is still partially retained, corrosive attack has penetrated the coating and has proceeded past the coating-substrate interface.

Mach 0.3, 200 Hours

The results of these tests are shown in table IV(a) and figures 10 and 11. For comparison, Mach 1.C data on these alloys are replotted on these figures. At both 1100° and 1200° C there is less weight loss during Mach 0.3 testing than during Mach 1.0 testing. However, these differences are slight and may be related to the less severe cooling rate of the Mach 0.3 tests. The effect of grain size as shown by comparing the 3933 (fine grain) data with that of 3933 HR (coarse grain) was negligible. At both temperatures, but especially 1200° C, the differences are little more than normal scatter in the data.

Mach 1.0, 500 Hours

The specific weight change results of the 500-hour tests at 1100° C conducted using natural gas are summarized in table IV(b) and figure 12. While the trend of increasing weight loss with decreasing Al content is apparent, no effect of yttria content could be detected when a multiple regression analysis of the data (ref. 7) was conducted. None of the total weight losses are excessive in 500 hours (less than 6 mg/cm²). However, there is a definite trend toward increased weight loss after 400 hours which indicates that, at least as judged by material loss, lives greatly in excess of 500 hours should not be anticipated. The microstructures, as in the case of the 200-hour samples of TD-NiCrAl, showed Kirkendhal porosity on all samples.

The effect of utilizing A-1 fuel rather than natural gas appeared to be to accelerate the attack, especially in the early stages and after about 400 cycles, but the same Al and Y effects can be seen, table IV(b)and figure 13. It does appear as though the TD samples need less Al to achieve the same oxidation resistance as YD alloys. While this may be due to the slightly higher Cr content of the TD alloys (16 against 15.5), it seems unlikely. As in the natural gas test there is a definite increase in negative slope toward the end of the test. Indeed it is more pronounced and starts earlier (about 300 hr) in the A-1 fuel tests.

While there were differences in weight loss between the two fuel tests, the thermal fatigue cracking was the same. Table V and figure 14 summarize the thermal fatigue data while figure 15 shows the appearance of the samples after 500 one-hour cycles in the combustion products of A-1 fuel. (The samples tested in natural gas are similar.)

In general, alloys with less than 5 percent aluminum formed thermal fatigue cracks after more cycles than those alloys with more than 5 percent aluminum. Also, the cracks formed on the alloys with less than 5 weight percent aluminum were shotter and more numerous than those on the alloys with the higher aluminum contents. It was noted that the thermal fatigue cracks of the TD-NiCrAl samples were less severe than any of the YD-NiCrAls. This may be due mainly to the fine grain size and resultant low modulus and strength of the TD alloy.

CONCLUSIONS

Based on burner rig oxidation testing at 1100° and 1200° C and on hot corrosion testing at 900° C of coated and bare ODS NiCr alloys and bare ODS NiCrAl alloys, the following conclusions were reached:

1. For 500 hour oxidation resistance at 1100° C in high velocity gases, ODS NiCrAl alloys can be produced which yield minimal weight losses and are significantly superior to bare or coated ODS NiCr alloys.

2. The ODS NiCrAl alloys are significantly superior in hot corrosion resistance to the ODS NiCr alloys, bare or with the experimental coatings evaluated.

3. All ODS NiCrAls suffer from thermal fatigue cracking, but those with less than 5 percent Al suffer substantially less.

4. After 300 to 400, one-hour cycles at 1100° C weight loss rates of ODS NiCrAL begin to accelerate making lives greatly in excess of 500 hours unlikely without further protection.

5. Changing the yttria level from 0.9 to 1.1 percent has no apparent effect on either oxidation or thermal fatigue properties of YD-NiCrAl alloys.

6. Probably the best compromise between oxidation and thermal fatigue resistance in a YD-NiCrAl alloy would be achieved with an aluminum content of less "han 5.0 percent and greater than 4.5 percent.

7. Mach 1 rig tests are slightly more severe than Mach 0.3 tests at both 1100° and 1200° C.

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TABLE 1. - COMPOSITION OF ALLOYS

Т	- 1		Т			-				5 .		1	
		1		4.J	5.7 X.A.	do. 3-0.6	M.A.	.87	2.5	40.38-0.4	4.02 Fel		
		Ĩ	:	15.5	5.5	60.3 -0.6	N.A.	0.90	S0. 1	40.36-0.49	4,02		
	1			25.51	5.6	40.3-0.6	И.А.	0.00	5	40 18-0.2	d.02	141.	
	mj T-V .			15.5	9.4	do. 1-0.6	N.A.	1.12	.05	4×.002	4.02	Bal.	•••
STOTE STOTE			B-2		.	do 1.0.6	N.A.	1.10	20	de. 002		341.	
C-SCELLTE &			B-1		. . .	40 2 0 E		0.86	89.0	d<.002	0.38-0.49	Pel.	
Hental Cebo			9-4 4		5.7	N.A.		1.12		d<,002	60.38-0.49	bal.	
Expel			A-5		15.6	М.А.	0.3-0.6	0.96	.76	d 202	40.36-0.49	Bel.	
			44		5.5	Y N	40.3-0.5	0.95	27	d, .002	do.38-0.49	1	
			E-A		15.4	K.A.	40.3-0.6	N.A.	6.	d<.002	64.0-86.0b		
			<u></u>		15.4	M.A.	d0.3-0.6	N.A.	8	50°.9	40.38-0.49		
					5.21	1. V. N	do. 3-0.6	N.A.		20. 20. 20.	40.38-0.49	20.02	
		3			2			~ ~					i
		9			8	.		,	• •	~			
	ICCAL	1011			16.22	4.63		2.03	N.N. X.A.	0.039	100. 0/1. 0/1.	N.A.	
		10.00			19.13	88.5		5.00	X.A.	0.035	. 376	N.A.	
	Constit-	Ĭ			5	4	= :	2 1 1 1	1 ⁵ 0 ³	• 0	v 0	×	<u>,</u>

Not malyzed.

^bAverage of three **maiyees**. Filosiaal compositios.

^d7rom starting powders.

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Alloy	Extru	Extrusion		atment	Hot rol	ling	Final	heat
1	Ratio	Ratio T.		Hrs T.		T.	treatm	ent
		°ć	°Č		°C percent °C		Hrs	T, °C
3931 3933 3933 HR	12:1	1040		1360	30	1090	2	1360
TN_MA 754	Unknown	Unknown	Unknown	Unknown	Unknown	Unknown	Unknown	Unknown
Cabot A-1	9:1	1040	1	1340	UIKIIOWII	UIKHOWH	Olikilowi	UIIKIIOWII
2 3 4 5 6 B-1 2 3 4				1310 1310 1340				
5				1310	1			1
6				1310				ł

TABLE II. - FORMING AND HEAT TREATMENT

TABLE III. - ALLOY PHASES PRESENT AS RECEIVED AS

Alloy	w/o Al	Nickel solid	Ni ₃ A1,	NIA1,	Th02	¥2 ⁰ 3
		Ŷ	γ'	β		
3931	3.88	Sa	Wp	N.D.C	Mq	N.D.
3933 and	4.63	S	W	N.D.	M	N.D.
3933 HR						
DS-NiCr		S	N.D.	N.D.	M	N.D.
Coatad		N.D. ^e	S	S	N.D.	N.D.
DS-N1Cr						
IN-MA-754	0.6	S	N.D.	N.D.	N.D.	N.D.
Coated		N.D. ^e	S	M	N.D.	N.D.
MA 754						
Cabot	5.0	S	N.D.	N.D.	N.D.	N.D.
	>~5.0	S	W	N.D.	N.D.	N.D.

DETERMINED BY X-RAY DIFFRACTION

^aStrong,

^bWeak.

^CNot detected.

CONTRACTOR AND A CONTRACTOR OF A CONTRACTOR OF

^dMedium.

^eMay be obscured by y' pattern.

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TABLE IV(A). - WEIGHT CHANGE MG/CM⁻² AFTER 200 ONE-HOUR CYCLES

Alloy		A-1 fuel,			
	1100 ⁰ C Mach 1	1100 ⁰ C Mach 0.3	1200 ⁰ C Mach 1	1200 ⁰ C Mach 0.3	9C) ^O C, +5 ppm sea salt
^a 3931	-0.93	-0.51	-4.3	-2.1	-1.1
^a 3933	51	.75	-4.1	93	-1.2
^a 3933 HR		. 49		97	
DS-NiCr	-46.6		-352		-9.2
Coated DS-NiCr	^b 68		-193		– 5.0
MA 754	-43.7		-290		-8.6
Coated MA 754	-13.2		-236		-2.8

^aAcerage of two.

^b2.59 lycles only (holder failure).

TABLE IV(B). - WEIGHT CHANGE AFTER 500

ONE-HOUR CYCLES AT 1100° C

Alloy	w/o Cr	w/o Al	w/o Y	Fuel	Specific weight change, mg/cm ⁻²
A-1 2 3 4 5 6 3933 B-1 2 3 4 5 6 3933	15.5 15.4 15.4 5.5 15.6 15.4 16.22 15.5 15.4 15.5 15.5 15.5 15.5 15.4 16.22	4.1 4.7 4.9 6.9 5.7 4.63 4.1 4.2 4.8 5.6 5.5 5.7 4.63	0.68 .88 .90 .75 .76 .88 2.03 ThO ₂ .68 .87 .88 .71 .71 .87 2.03 ThO ₂	Natural gas	$\begin{array}{r} -3.3 \\ -5.2 \\ -1.8 \\ -2.5 \\ -2.5 \\ -2.4 \\ -3.1 \\ -14.5 \\ -15.5 \\ -8.4 \\ -4.5 \\ -3.2 \\ -2.8 \\ a_{-2.6} \end{array}$

^aAverage of two.

TABLE V. - THERMAL FATIGUE CRACKING BEHAVIOR IN

MACH 1, 500 ONE-HOUR CYCLIC OXIDATION

Alloy	Fuel	w/o Al	Cycles to first crack	Final crack length, mm
3933	Natural gas	4.63	154	2.0
3933	A-1	4.63	154	.5
A-1	Natural gas	4.1	48	2.5
2		4.1	48	4.0
3		4.7	48	2.5
4		4.9	74	3.0
5		4.9	74	2.0
6		5.7	23	9.5
B-1	A-1	4.1	74	2.5
2	1	4.2	74	2.0
3		4.8	74	1.5
4		5.6	48	10.5
5		5.5	48	10.5
6		5.7	23	9.0

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TESTS AT 1100° C



(a) A second se second sec

(a) Overall view.

Figure 1. - Mach 1 oxidation surface.

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(a) Overall view.

Figure 1. - Mach 1 oxidation surface.

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Figure 2. - Mach 0.3 oxidation apparatus.

FIGURE 3. Mach. 1 Oxidation of Several Dispersion Strengthened Alloys at 1100° C. One Hour Cycles.

FIGURE 4. Mach 1 Oxidation of Several Dispersion Strengthened Allovs at 1200° C. One Hour Cycles.

A 1100°C, 169-

B. MOOR , 200 -

FIGURE 6. THE EFFECT OF MACH 1 OXIDATION ON THE MICROSTRUCTURE OF COATED DS-NICT. 250X

B, 1200°C , 200-1Hour Cycles.

FIGURE 6. THE EFFECT OF HACHI OXIDATION ON THE MICROSTRUCTURE OF COATED IN-MA754. 250 X

BJ200°C , 200-1 HOUR CYCLES

FIGURE 7, THE EFFECT OF MACH 1 ONDERNON ON THE MICROSTRUCTURE OF TONICRAL (3933). 250%

& COATED

FIGURES. HOT CORRESION OF DS-NICE AT 900°C WITH 5 PPM SYNTHETIC SEA SALT. MACH1. 200 - 1HOUR CYCLES. 250X

FIGURE 9. HOT CORROSION OF COATED IN-MATSY AT 900°C WITH 5 PPM SYNTHETIC SEA SALT. MACH 1, 200 - I HOUR CYCLES. 250%

FIGURE 12. Much 1 Oxidation of Dispersion Strengthened NiCrAl Alloys at 1100° C. Natural Gas. One Hour Cycles.

Fuel. One Hour Cycles.

FIGURE 14. EFFECT OF ALUMINUM CONTENT ON THE MAXIMUM THERMAL FATIGUE CRACK LENGTH OF SME YD-NICHAI ALLOYS, EOD - ONE HOUR CYCLES AT 1100°C IN MACH 2 GASSES

4.1 AL 4.2 AL 114203 . St 12 42

4.8A1 1.14203

S.GAI AOTIOS

S.SAI 5.7AI 904203 LIY205

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4.6 AL 2.0 THOL

FIGURE IS. THERMAL FATIGUE CRACKS IN SOME DISPERSION STRENGTHENED NICAL ALLOYS. 500-IHOUR CYCLES AT