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# The Cyclic Oxidation Resistance at 1200 °C of $\beta$ -NiAl, FeAl, and CoAl Alloys With Selected Third Element Additions

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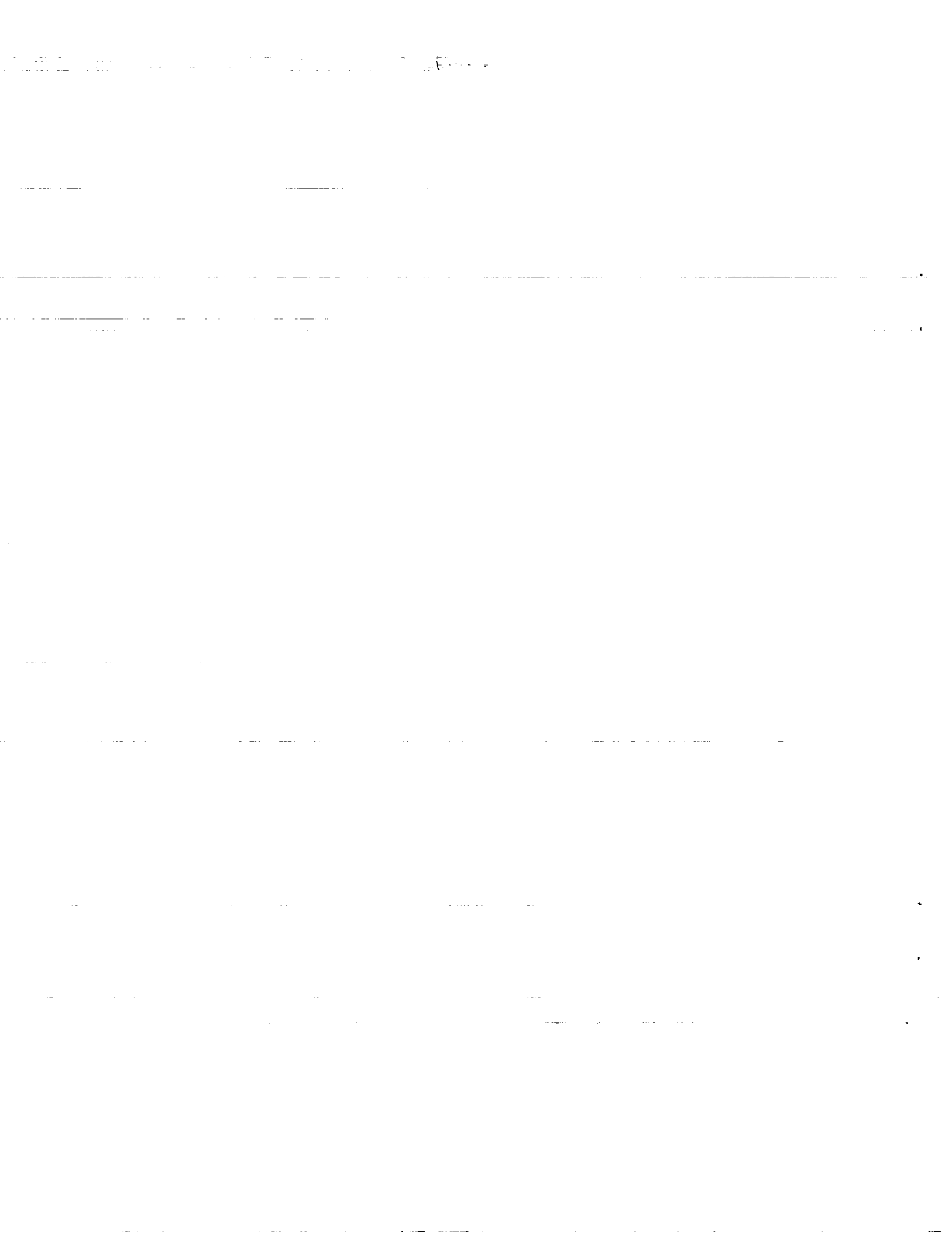
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ALLOYS WITH SELECTED THIRD ELEMENT ADDITIONS

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SUMMARY

The intermetallic compounds  $\beta$ -NiAl, FeAl, and CoAl were tested in cyclic oxidation with selected third element alloy additions. Tests in static air for 200 1-hr cycles at 1200 °C indicated by specific weight change/time data and x-ray diffraction analysis that the 5 at % alloy additions did not significantly improve the oxidation resistance over the alumina forming baseline alloys without the additions. Many of the alloy additions were actually deleterious. Ta and Nb were the only alloy additions that actually altered the nature of the oxide(s) formed and still maintained the oxidation resistance of the protective alumina scale.

INTRODUCTION

The intermetallic compounds  $\beta$ -NiAl, FeAl, and CoAl are potentially attractive materials for high temperature use. The ordered structure provides the high temperature strength while the Al present would tend to form  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> to provide oxidation resistance. Other investigations at this laboratory have focused on third element additions to these alloys and their effect on various high temperature strength properties (refs. 1 and 2). This investigation will study the cyclic oxidation resistance of a similar series of ternary alloys at 1200 °C in static air. The relative oxidation resistance among the alloys will be evaluated and compared with the binary alloys used here and in other studies.

PROCEDURES

The aluminides of Ni-, Fe-, and Co- with third element additions tested along with the basic binary alloy(s) are listed in table I. All these binary and ternary alloys were made by hot extrusion of blended binary powders with or without the third element powder additions. The Ta, Cr, etc., tend to be present as large inclusions in a lean aluminide matrix (i.e., these alloys tend to be nonhomogeneous). These ternary additions were all at the 5 atomic percent (at %) level. The microstructures are described in reference 3.

These extruded bars were machined into small disk samples approximately 10 mm in diameter by 2 mm in thickness with a 1.5 mm diameter hanger hole to suspend the sample(s) during cyclic testing. The samples with the as ground surface(s) with a 32 rms finish are suspended from Pt wire hanger hooks into a standard NASA vertical multitube cyclic oxidation furnace rig (ref. 4). The individual alloy samples were tested in groups of six for 200 1-hr cycles at 1200 °C. The samples were removed for weighing at selected intervals to generate specific weight change/time curves. The sample surfaces were analyzed by x-ray diffraction after 1, 100, and 200 cycles (hr).

## RESULTS AND DISCUSSION OF RESULTS

The specific weight change/time data were divided into three oxidation groupings (classes A, B, and C, respectively). Class A data which includes the three binary alloys  $\beta$ -NiAl, FeAl, and CoAl and their ternary alloys, which behave and appear similar to their basic binary alloys are listed in table II. The class B alloys detailed in table III exhibited poorer cyclic oxidation resistances than their basic binary alloys but lasted 200 1-hr cycles without appreciable sample degradation. Table IV summarizes class C alloys which show catastrophic cyclic oxidation behavior either by sample cracking and breakage, severe oxide spalling, large sample growth where the metal actually appeared to grow along with the scale or some combination of all three failure modes. In most cases the sample failed prematurely some time during, or after, the first 1-hr cycle.

Most of the class A alloys appear similar after the 200 1-hr cycle exposure. All had a uniform dark gray scale with a slight powdery texture. Two of the alloys (Fe-46Al-5Cr and Co-50Al) showed minor edge thinning due to scale spalling. The (Co-49Al-5Ni) alloy had a glazed cracked gray scale. All of the alloys in this grouping formed  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> as the primary oxide as determined by x-ray analysis. It is basically a thin protective scale which tends to spall slightly upon cooling. X-ray diffraction also detected the underlying aluminide alloy. In a few cases the aluminate spinel ( $a_0 = 8.05$  to  $8.10$  Å) was also observed. The more interesting results with respect to cyclic oxidation resistance are for the alloys that contain Nb or Ta, in that they form the refractory metal oxides, AlNbO<sub>4</sub> or AlTaO<sub>4</sub>, respectively, even though free metallic Nb or Ta was observed. In addition, the Ni-50Al-5Ta alloy forms the tri-rutile oxide, shown to be protective with Al<sub>2</sub>O<sub>3</sub>/aluminate forming superalloys (refs. 5 to 7). For this reason it is believed that the Ta alloy addition to binary aluminide alloys, particularly stoichiometric  $\beta$ -NiAl, is promising for further evaluation but at a lower Ta level, say in the 1 to 3 at % range.

The kinetic behavior of the class A alloys are quite similar. Of the 24 alloys listed in table II, 20 behave in a classic parabolic manner usually observed in cyclic oxidation as indicated by a maximum specific weight change,  $\Delta W/A$ , followed by a steady negative specific weight change finally giving negative values. This is typical of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> protective scale growth but with significant oxide spalling as well. The baseline binary  $\beta$ -NiAl alloys tested are plotted in figure 1 indicating the range of behavior. Figure 2 shows the  $\Delta W/A$  versus time curves for the five third-element (Si, Co, Nb, Re, and Ta) additions showing similar behavior to the binary  $\beta$ -NiAl alloys. A typical  $\beta$ -NiAl alloy from figure 1 is also shown on this plot. Both Ta and Nb show high positive initial  $\Delta W/A$  values due to the refractory metal oxides being formed along with the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>.

The binary FeAl alloys shown in figure 3 behave similarly to the  $\beta$ -NiAl alloys. The FeAl specific weight change curves tend to flatten out slightly and in the case of the Fe-39.8 percent alloy tended to increase in weight after 100 cycles.

Alloy additions of Si, Ti, Ni, Cr, Re, and Nb to the FeAl system had little effect on the oxidation behavior of the binary material as indicated by the similar net change data plotted in figure 4. Again a typical FeAl binary alloy curve from figure 3 is plotted for comparison. In the FeAl system Nb appeared to change the nature of the oxide growth/spalling process as Nb and Ta did in the  $\beta$ -NiAl alloys. This was confirmed by the x-ray identification of niobates or tantalates.

Cyclic oxidation studies of the CoAl alloys indicate that only the Ni-third element CoAl alloy could be considered as a class A alloy based on specific weight changes. Still the scale appeared cracked and glazed and therefore would not be expected to be protective. Class B alloys listed in table III lasted the full 200 1-hr cycles and were mainly Al<sub>2</sub>O<sub>3</sub> formers but spalled excessively to give large negative specific weight change values. The oxidation behavior was significantly poorer than their binary alloy standards and showed little promise.

The class C alloys listed in table IV showed catastrophic oxidation behavior in cyclic testing at 1200 °C. Roughly one-third of the alloys were so brittle they cracked and broke apart after the first cycle. The rest tended to shatter well under 200 cycles. Some samples actually showed massive growth with large linear specific weight change increases. This is particularly true with the 5 percent Zr additions which in the low percentage range (~0.1 to 0.2 wt %) is so beneficial in conferring oxidation resistance in the  $\beta$ -NiAl system (refs. 8 to 10).

### CONCLUDING REMARKS

Additions at the nominal 5 at % were selected from a mechanical property viewpoint, however they tended to be extremely brittle particularly when tested in cyclic oxidation. To optimize the oxidative properties of these materials, third element additions of less than 1 at % would have been a better choice. The most dramatic case is Zr, which at the 5 percent level leads to catastrophic oxidation, while in the range of 0.03 to 0.1 at % results in the  $\beta$ -NiAl having the best cyclic oxidation resistance of all the alumina or chromia formers in the 1200 to 1300 °C range.

The 5 at % alloy additions that effectively maintained the cyclic oxidation resistance of the baseline binary alloys were Si, Co, Nb, Ta, and Re in the  $\beta$ -NiAl; Si, Ti, Ni, Nb, and Re in FeAl; and Ni in CoAl. Nb and Ta actually altered the nature of the external scale and would be of interest perhaps in the 1 to 2 at % range. From other studies at this laboratory, a 0.1 at % fourth element addition of Zr should significantly improve the cyclic oxidation resistance as well.

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TABLE I.—NOMINAL ALLOY  
 CONTENT OF M-AL AND  
 M-AL-X ALLOYS TESTED  
 IN CYCLIC OXIDATION (at %)

<b>Nickel base</b>	
Ni-43.9Al	Ni-42Al-5Hf
-50Al	-42Al-5Si
-52.7Al	-42Al-5Zr
	-42Al-5Mn
	-50Al-5Co
	-50Al-5Cr
	-50Al-5Co
	-50Al-5Fe
	-50Al-5Nb
	-50Al-5Re
	-50Al-5Ta
<b>Iron base</b>	
Fe-39.8Al	Fe-39Al-5Ni
-41.7Al	-39Al-5Si
-48.7Al	-39Al-5Ti
-50Al	-39Al-5Zr
	-46Al-5Co
	-46Al-5Cr
	-46Al-5Nb
	-46Al-5Ni
	-46Al-5Mo
	-46Al-5Re
	-46Al-5Ta
	-46Al-5W
	-47Al-5Zr
<b>Cobalt base</b>	
Co-50Al	Co-49Al-5Fe
	-49Al-5Mo
	-49Al-5Ni
	-49Al-5Ta
	-49Al-5W

TABLE II.—CLASSIFICATION OF CYCLIC OXIDATION BEHAVIOR OF M-AL AND M-AL-X ALLOYS  
 TESTED FOR UP TO 200 ONE-HOUR CYCLES AT 1200 °C IN STATIC AIR—CLASS A  
 BEHAVIOR—THE TERNARY ALLOY BEHAVIOR SIMILAR TO  
 THE BINARY BASE ALLOY

Alloy	Run number <sup>a</sup>	Final		XRD phases at 1, 100, and 200 hr	Final sample description
		$\Delta W/A$	Time		
Ni-50Al	628-1	-8.43	200	$Al_2O_3$ ; $\beta NiAl$	Uniform dark grayish speckled scale ↓
-50Al	628-2	-8.66	↓	$Al_2O_3$ ; $\beta NiAl$	
-52.7Al	630-1	-4.48		$Al_2O_3$ ; aluminate spinel; $\beta NiAl$	
-50Al	660-5	-10.54		$Al_2O_3$ ; $\beta NiAl$	
-43.9Al	630-2	-7.69		$Al_2O_3$ ; $\beta NiAl$	
Ni-42Al-5Si	628-3	-7.98	200	$Al_2O_3$ ; $\beta NiAl$	Uniform dark grayish speckled scale ↓
-50Al-5Co	629-1	-9.47	↓	$Al_2O_3$ ; $\beta NiAl$ ; aluminate spinel at 1 hr	
-50Al-5Nb	629-2	-9.50		$Al_2O_3$ ; $AlNbO_4$ ; $\beta NiAl$ <sup>b</sup>	
-50Al-5Ta	629-4	.86		$Al_2O_3$ ; $AlTaO_4$ ; $\beta NiAl$ <sup>c</sup>	
-50Al-5Re	629-6	-7.35		$Al_2O_3$ ; $\beta NiAl$ <sup>d</sup>	
Fe-41.7Al	630-5	-7.39		↓	
-48.7Al	630-6	-2.30			$Al_2O_3$ ; FeAl
-39.8Al	631-1	-5.78	$Al_2O_3$ ; FeAl		
-50Al	631-2	-3.47	$Al_2O_3$ ; FeAl		
-50Al	660-6	-1.53	$Al_2O_3$ ; FeAl		
Fe-39Al-5Si	631-4	-15.04	↓		$Al_2O_3$ ; FeAl <sup>f</sup>
-39Al-5Ti	631-5	-10.42		$Al_2O_3$ ; FeAl	
-39Al-5Ni	631-6	-3.31		$Al_2O_3$ ; FeAl	
-46Al-5Cr	635-3	-1.44		$Al_2O_3$ ; FeAl	
-46Al-5Nb	635-5	6.13		$Al_2O_3$ <sup>g</sup>	
-46Al-5Re	636-3	-3.93		$Al_2O_3$ ; FeAl	
Co-50Al	636-4	-15.78	200	$Al_2O_3$ ; CoAl	Uniform dark grayish speckled scale ↓
-50Al	636-5	-8.27	200	$Al_2O_3$ ; CoAl	
Co-49Al-5Ni	636-6	-12.60	200	$Al_2O_3$ ; CoAl <sup>h</sup>	Glazed cracked gray scale

<sup>a</sup>Sequence run number with numbers 1 to 6 indicating each of six circumferential vertical tubes capable of holding one hanging test sample.

<sup>b</sup>1 hr  $NbO_2$  also detected; 200 hr  $NiNb_2O_4$  + aluminate spinel also detected.

<sup>c</sup>1 hr free Ta also detected; 200 hr tri-rutile,  $a_0 = 3.29 \text{ \AA}$  also detected.

<sup>d</sup>1 hr free Re also detected; 100 hr aluminate spinel also detected.

<sup>e</sup>1 hr aluminate spinel also detected.

<sup>f</sup>1 hr tri-rutile,  $a_0 = 3.23 \text{ \AA}$  also detected.

<sup>g</sup>1 + 100 hr  $AlNbO_4$  also detected; FeAl also detected at 200 hr.

<sup>h</sup>200 hr aluminate spinel also detected.

<sup>i</sup>Some edge thinning.



TABLE III.—CLASSIFICATION OF CYCLIC OXIDATION BEHAVIOR OF M-AL AND M-AL-X ALLOYS TESTED FOR UP TO 200 ONE-HOUR CYCLES AT 1200 °C IN STATIC AIR—CLASS B BEHAVIOR—  
THE TERNARY ALLOY SIGNIFICANTLY POORER THAN THE BINARY BASE ALLOY

Alloy	Run number <sup>a</sup>	Final		XRD phases at 1, 100, and 200 hr	Final sample description
		$\Delta W/A$	Time		
Ni-50Al-5Cr	628-5	-35.20	200	Al <sub>2</sub> O <sub>3</sub> ; aluminate spinel; Ni sol. sol. at 100 and 200 hr	Cracking and edge spall
-42Al-5Mn	630-3	-35.91	200	Al <sub>2</sub> O <sub>3</sub> ; $\beta$ NiAl; aluminate spinel	Spotted, edge spall, and spall to bare metal
Fe-46Al-5Ni	635-2	-33.14	200	Al <sub>2</sub> O <sub>3</sub> ; FeAl	Uniform dark grayish speckled scale
-46Al-5Co	635-4	-34.59	200	Al <sub>2</sub> O <sub>3</sub> ; FeAl	Uniform dark grayish speckled scale
Co-49Al-5Re	639-6	-44.22	200	Al <sub>2</sub> O <sub>3</sub> ; CoAl; aluminate spinel at 100 and 200 hr	Edge pest and spall to bare metal

<sup>a</sup>Sequence run number with numbers 1 to 6 indicating each of six circumferential vertical tubes capable of holding one hanging test sample.

TABLE IV.—CLASSIFICATION OF CYCLIC OXIDATION BEHAVIOR OF M-AL AND M-AL-X ALLOYS TESTED FOR UP TO 200 ONE-HOUR CYCLES AT 1200 °C IN STATIC AIR—CLASS C BEHAVIOR—  
THE TERNARY ALLOY BEHAVIOR CATASTROPHIC WHEN COMPARED TO  
THE BINARY BASE ALLOY

Alloy	Run number*	Final		XRD phases at 1, 100, and 200 hr	Final sample description
		$\Delta W/A$	Time		
Ni-42Al-5Zr	628-4	135.04	30	$Al_2O_3$ ; $ZrO_2$ ; aluminate spinel; Ni sol. sol. at 1 and 30 hr	Sample growth and severe cracks
-50Al-5Fe	628-6	.40	60	$Al_2O_3$ ; $\beta NiAl$	Cracked and broke
-50Al-5Mo	629-3	-145.11	15	$Al_2O_3$ ; $\beta NiAl$ ; aluminate spinel and free Mo at 1 hr	Two large pieces broke off, liquid spots after 1 hr
-50Al-5W	629-5	-2.99	1	$NiWO_4$ ; $Al_2O_3$ ; $VNiAl$ ; free W	Liquid spots and pieces broke off
-42Al-5Hf	630-4	5.93	60	$HfO_2$ ; $\beta NiAl$ ; aluminate spinel at 1 hr; $Al_2O_3$ + Ni sol. sol. at 60 hr	Sample cracked and broke
Fe-47Al-5Zr	631-3	209.4	200	$Al_2O_3$ ; FeAl at 1 hr; $Fe_2O_3$ and $ZrO_2$ at 100 hr; $Fe_2O_3$ at 200 hr	Large uniform growth
-39Al-5Zr	635-1	99.40	75	$ZrO_2$ ; $Al_2O_3$ ; FeAl	Large growth and radial cracks
-46Al-5Mo	635-6	-3.74	30	$Al_2O_3$ ; FeAl; free Mo at 1 hr	Large crack 1 hr and sample broke in two
-46Al-5Ta	636-1	.84	1	$AlTaO_4$ ; $Al_2O_3$ ; FeAl	Sample broke
-46Al-5W	636-2	-60.94	1	$Al_2O_3$	Large piece broke off
Co-49Al-5Fe	639-1	-10.28	130	$Al_2O_3$ ; CoAl	Large crack to hanger hole
-49Al-5Nb	639-2	188.5	200	CoAl; $AlNbO_4$ ; $Al_2O_3$ at 1 hr; aluminate spinel; $CoNb_2O_6$ at 100 hr	Large uniform growth
-49Al-5Mo	639-3	-30.44	1	$Al_2O_3$ ; aluminate spinel; $CoMoO_4$ , CoAl	Piece broke off and severe "pest"
-49Al-5Ta	639-4	-38.72	100	Tri-rotile; $Al_2O_3$ ; CoAl; at 1 hr aluminate spinel; $AlTaO_4$ ; $CoTa_2O_6$ at 100 hr	One large crack and severe edge spall
-49Al-5W	639-5	116.6	75	$CoWO_4$ ; at 75 hr aluminate spinel; CoAl	Large growth and radial cracks

\*Sequence run number with numbers 1 to 6 indicating each of six circumferential vertical tubes capable of holding one suspending test sample.

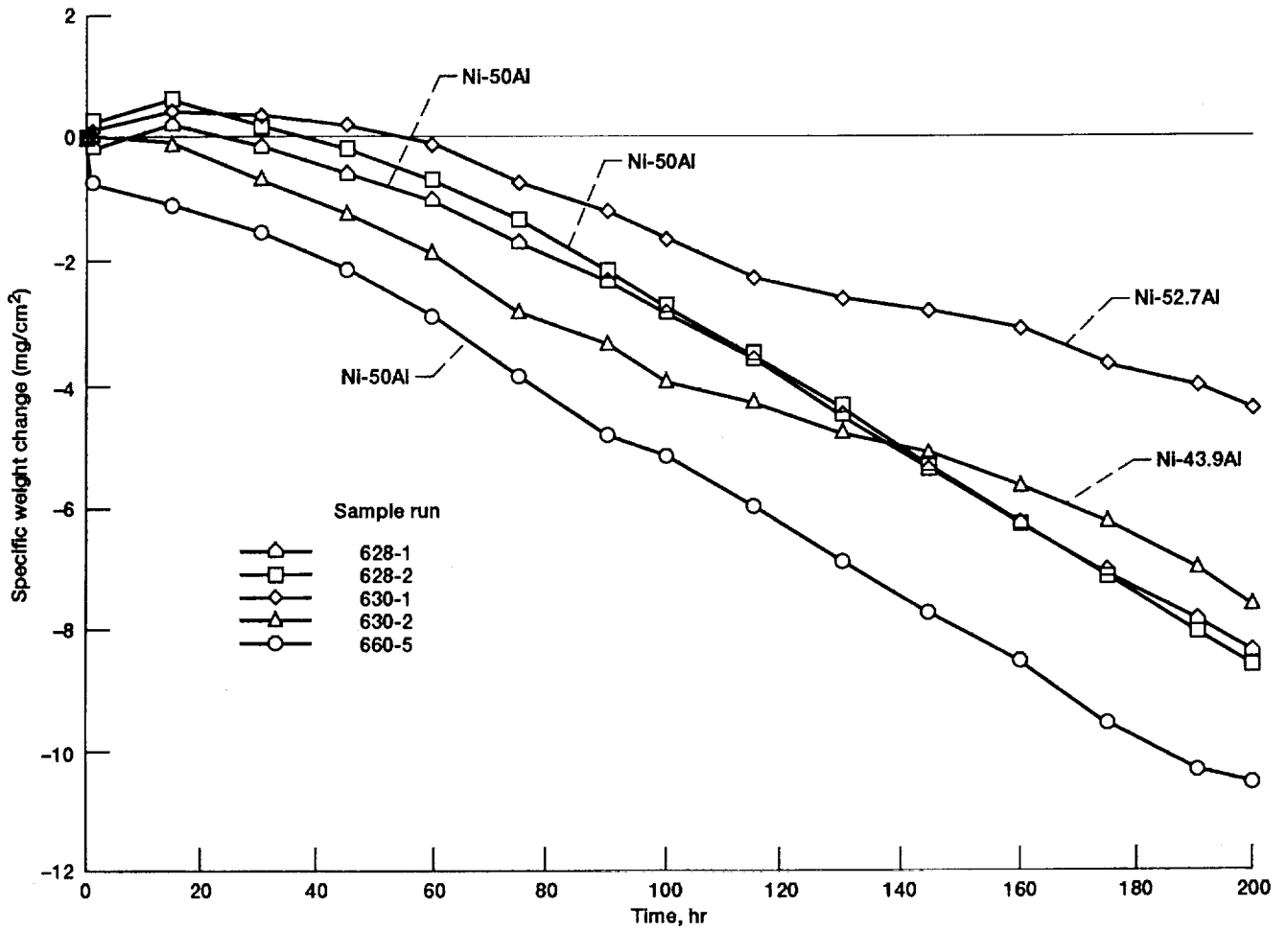


Figure 1.—Specific weight change versus time curve for  $\beta$  NiAl binary alloys with varying Al content. One hour exposure cycles in 1200 °C static air.

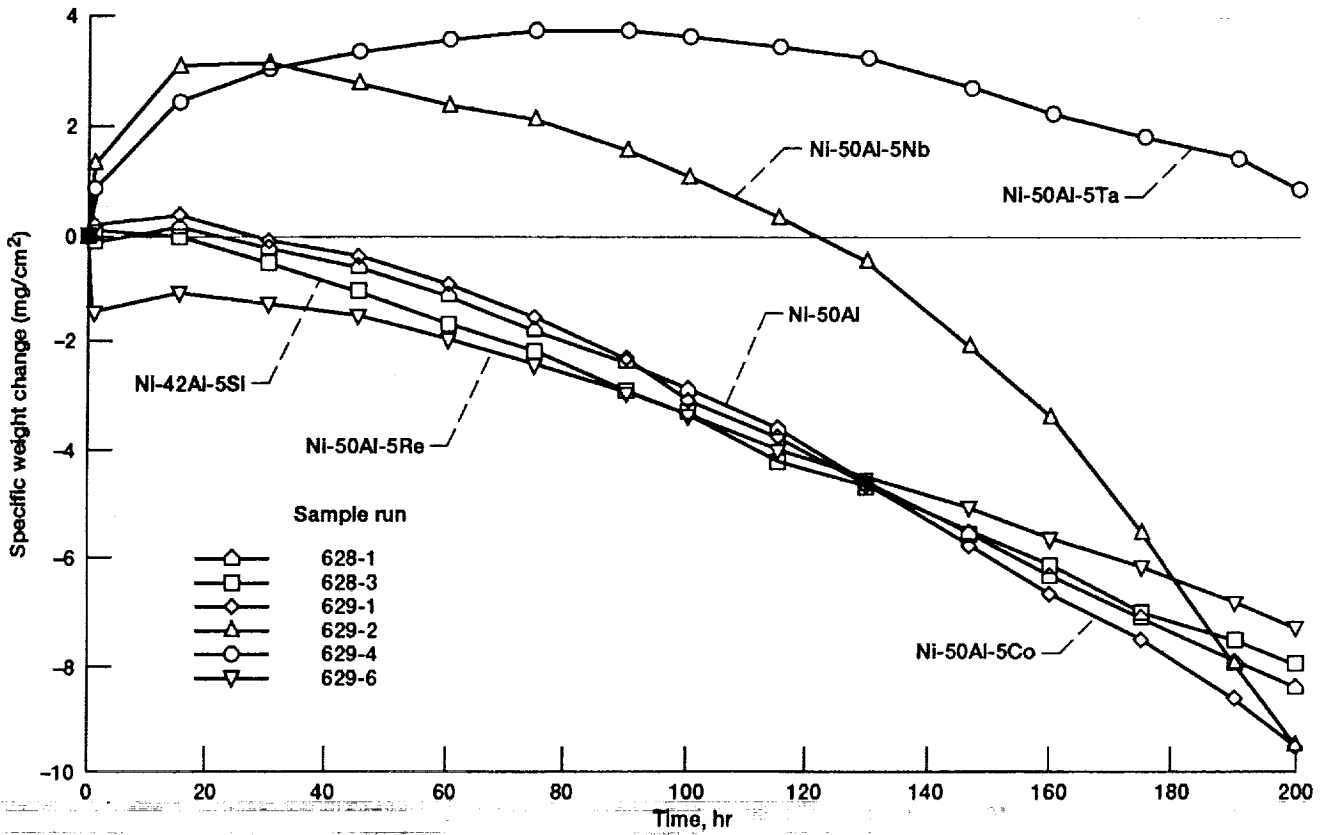


Figure 2.—Specific weight change versus time curve for class A behaving Ni-Al-X alloys compared to a  $\beta$  NiAl alloy from figure 1. One hour exposure cycles in 1200 °C static air.

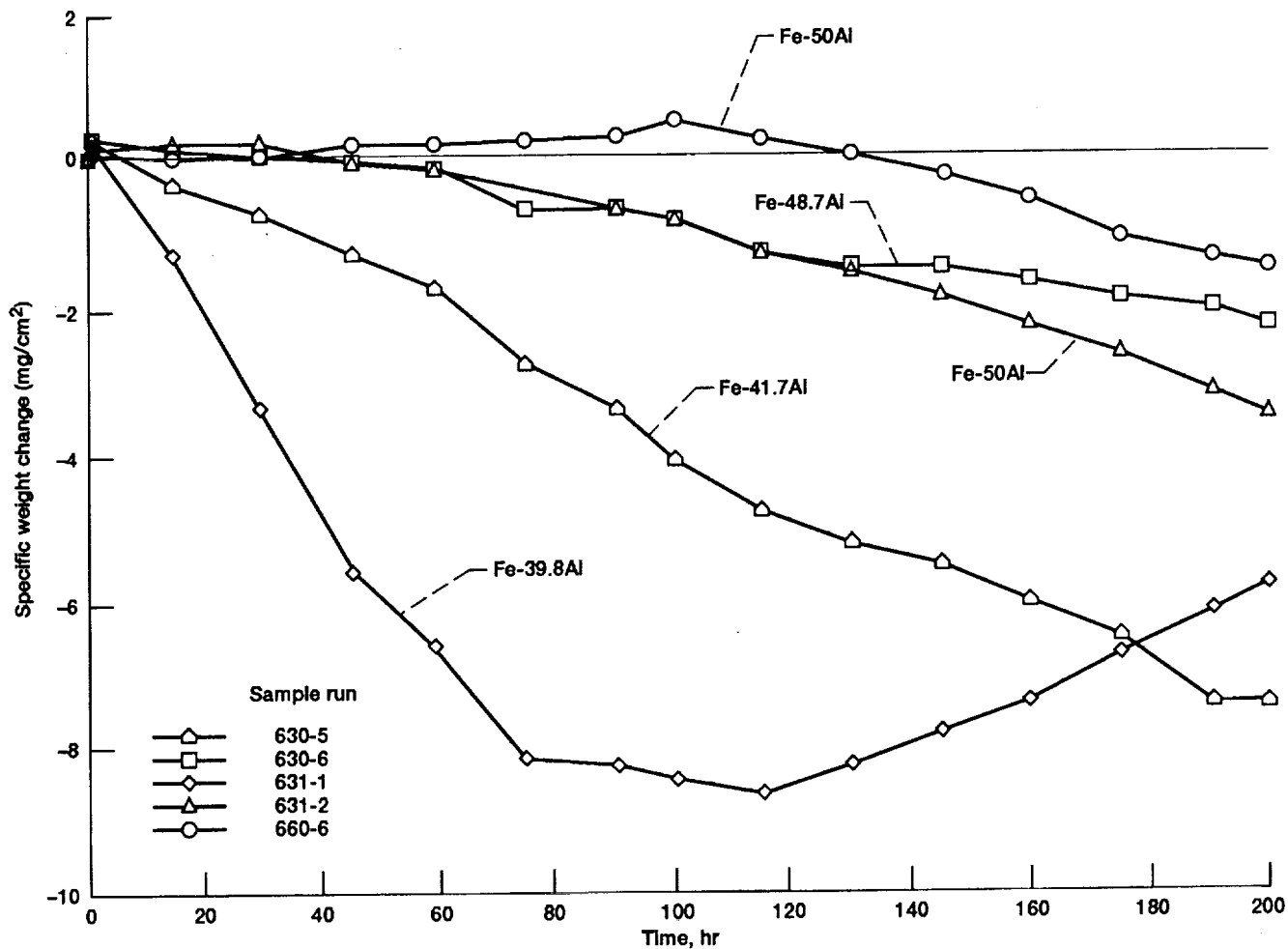


Figure 3.—Specific weight change versus time curve for FeAl binary alloys with varying Al content. One hour exposure cycles in 1200 °C static air.

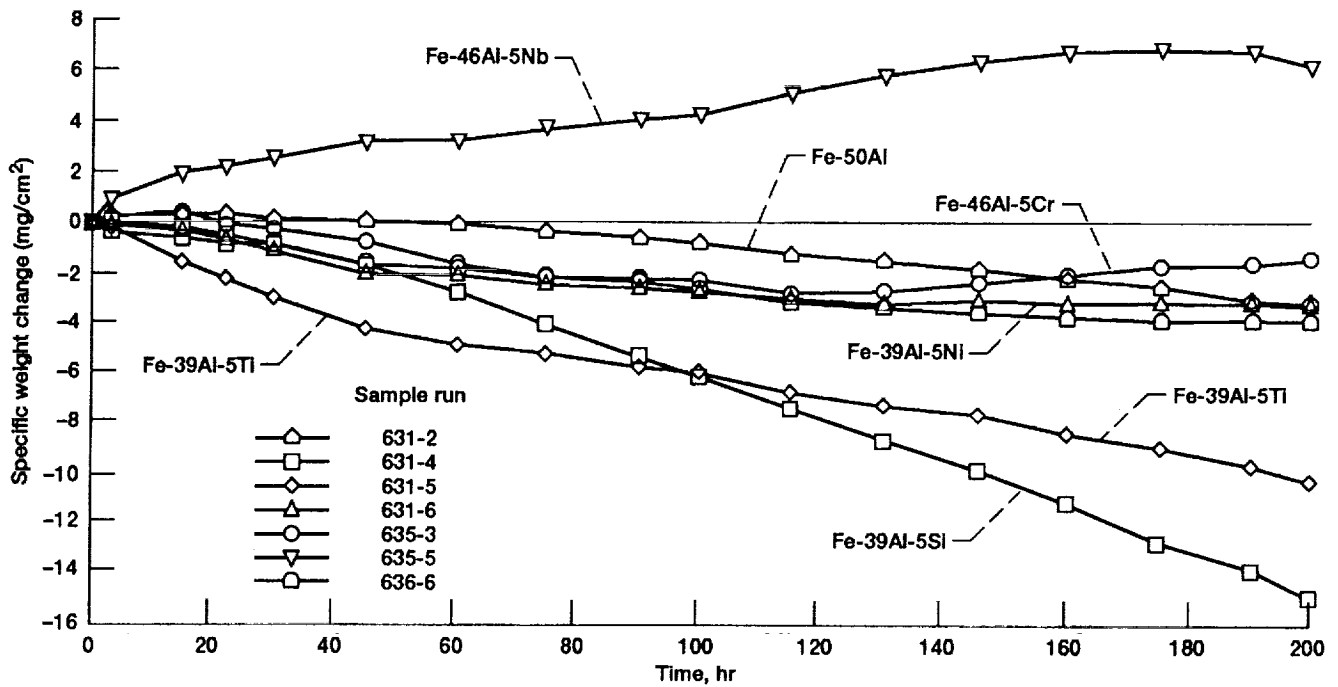


Figure 4.—Specific weight change versus time curve for class A behaving Fe-Al-X alloys compared to an Fe-Al alloy from figure 3. One hour exposure cycles in 1200 °C static air.

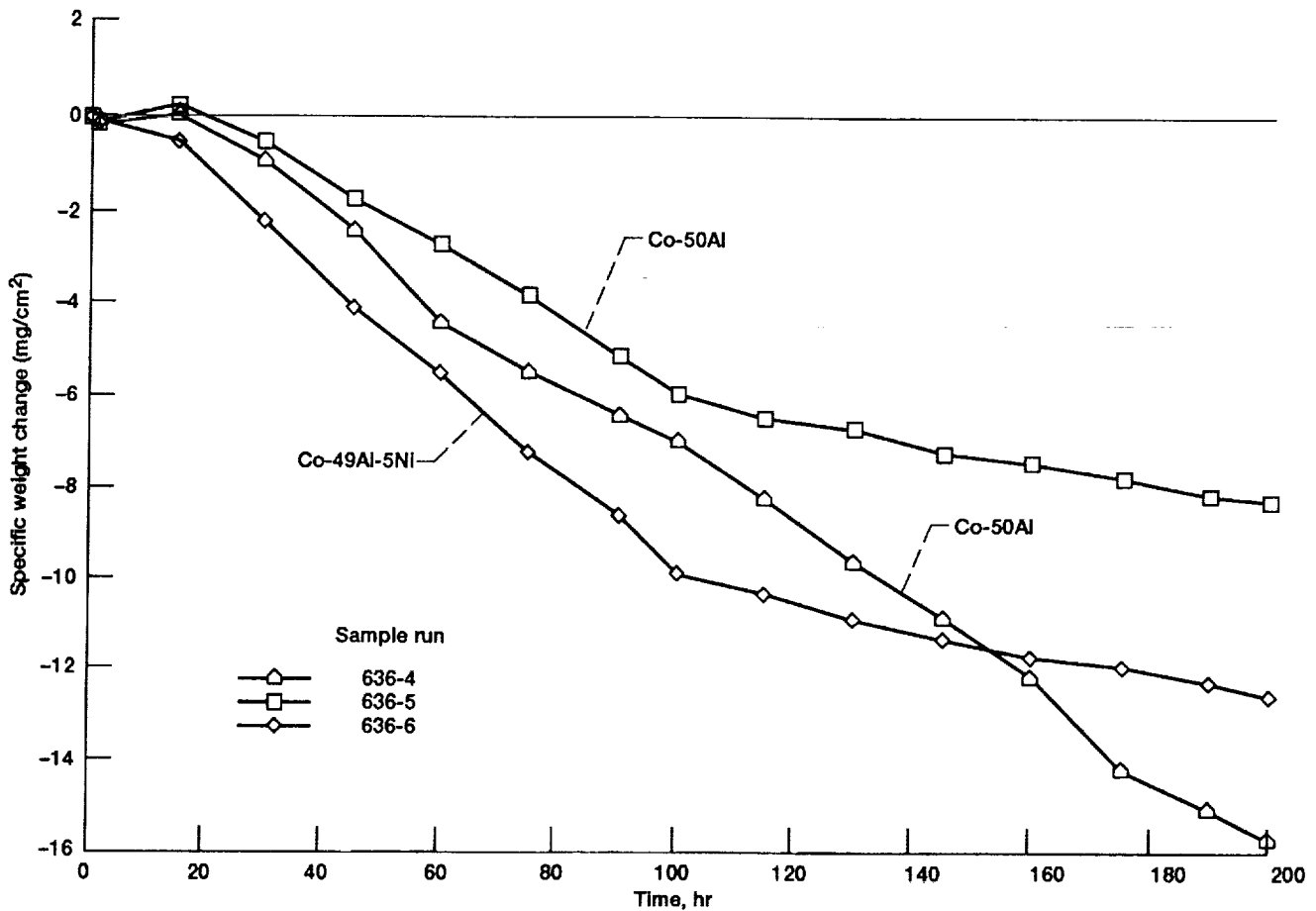


Figure 5.—Specific weight change versus time curve for class A behaving Co-Al-X alloys compared to an Co-Al binary alloys. One hour exposure cycles in 1200 °C static air.



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**13. ABSTRACT** (Maximum 200 words)

The intermetallic compounds  $\beta$ -NiAl, FeAl, and CoAl were tested in cyclic oxidation with selected third element alloy additions. Tests in static air for 200 1-hr cycles at 1200 °C indicated by specific weight change/time data and x-ray diffraction analysis that the 5 at % alloy additions did not significantly improve the oxidation resistance over the alumina forming baseline alloys without the additions. Many of the alloy additions were actually deleterious. Ta and Nb were the only alloy additions that actually altered the nature of the oxide(s) formed and still maintained the oxidation resistance of the protective alumina scale.

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