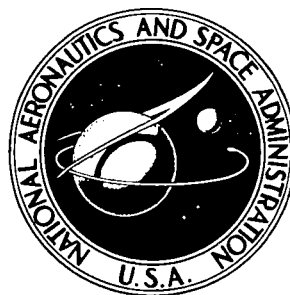


**NASA TECHNICAL
MEMORANDUM**



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**OXIDATION BEHAVIOR OF NICKEL-CHROMIUM-
ALUMINUM-YTTRIUM - MAGNESIUM OXIDE
AND NICKEL-CHROMIUM-ALUMINUM-YTTRIUM -
ZIRCONATE TYPE OF CERMETS**

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16. Abstract <p>The 1100^o and 1200^o C cyclic oxidation resistance of dense Ni-Cr-Al-Y - MgO, Ni-Cr-Al-Y - CaZrO₃, Ni-Cr-Al-Y - SrZrO₃, Ni-Cr-Al-Y - MgZrO₃ cermets and an ≈70 percent dense Ni-Cr-Al-Y developmental material was determined. The cermets contained 60 and 50 volume percent of Ni-Cr-Al-Y which formed a matrix with the oxide particles imbedded in it. The cermets containing MgO were superior to cermets based on zirconates and to the porous Ni-Cr-Al-Y material.</p>			
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MAGNESIUM OXIDE AND NICKEL-CHROMIUM-ALUMINUM-
YTTRIUM - ZIRCONATE TYPE OF CERMETS

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SUMMARY

The cyclic oxidation resistance of two groups of Ni-Cr-Al-Y - MgO, Ni-Cr-Al-Y - CaZrO₃, Ni-Cr-Al-Y - SrZrO₃ and Ni-Cr-Al-Y - MgZrO₃ cermets was studied at 1100° and 1200° C in still air using a thermogravimetric method supplemented by X-ray diffraction analysis and metallography. One group of cermets contained 60 percent by volume of Ni-Cr-Al-Y and 40 percent of refractory oxides, and the second group contained 50 percent Ni-Cr-Al-Y and 50 percent of refractory oxides. In both cases, Ni-Cr-Al-Y formed the matrix, with the oxide particles imbedded in it. For comparison, a similar study was made on an approximately 70 percent dense Ni-Cr-Al-Y developmental material.

This study showed that the oxidation resistance of the cermets containing MgO was superior to those cermets containing zirconates and to the 70 percent dense Ni-Cr-Al-Y. No evidence of oxidation along interphase boundaries or reaction between MgO and Ni-Cr-Al-Y were observed. However, oxidation of the zirconate type of cermets was significantly more severe and was accompanied by swelling and cracking of the specimens. In addition, all of the zirconates reacted with the Ni-Cr-Al-Y matrix. The 70 percent dense Ni-Cr-Al-Y, due to its porous nature, exhibited intermediate oxidation weight change behavior and some dimensional growth.

INTRODUCTION

The present study is a continuation of a program to develop materials for use as turbine-blade-tip seals in advanced aircraft gas-turbine engines. Better seal materials will allow these engines to operate at higher temperatures, higher thrust, and lower specific fuel consumption. Improved abradable seals could be made of cermets (refs. 1

to 4). The initial stage of this program (ref. 5) was concerned with the oxidation behavior of several superalloy-based cermets containing variable amounts of magnesium oxide (MgO). That study led to the conclusion that the oxidation resistance of low aluminum content (~5% Al) Ni-Cr-Al-Y cermets was superior to that of cermets based on any of the other superalloys studied. The present interest in the applications of M-Cr-Al-Y alloys (ref. 6) and of graded stabilized zirconia (refs. 7 and 8) as blade-tip-seal materials prompted this study of the cyclic oxidation resistance and thermal stability of higher aluminum content Ni-Cr-Al-Y (~17% Al) based cermets. The oxides selected were calcium zirconate, strontium zirconate, magnesium zirconate, and magnesia, and these were added to the Ni-Cr-Al-Y in amounts of 40 and 50 percent by volume. The zirconates were chosen because they offered higher thermal coefficients of expansions, and thus a good match with the matrix. Oxidation experiments were performed in still air at 1100^o and 1200^o C by a thermogravimetric method and were supplemented by metallographic and X-ray diffraction analysis.

EXPERIMENTAL PROCEDURES

Sample Preparation

The alloy used in this investigation was an aluminum rich Ni-Cr-Al-Y of the following composition (chemical analysis determined by supplier):

Element	Content, wt. %
Aluminum	17.05
Chromium	19.61
Yttrium	.97
Nickel	Bal.

It was purchased in powder form. According to the equilibrium diagram for the Ni-Cr-Al system at 1000^o C (ref. 9) the alloy should be composed of a β matrix containing γ precipitates.

The MgO, CaZrO₃, SrZrO₃, and MgZrO₃ refractory oxides were also obtained in the form of powders with graded particle sizes. Their analyses are shown in table I.

The metallic powder (-325 mesh or 44 μ m particle size) was blended with refractory oxide powders (-100 to 200 mesh or 149 to 74 μ m particle size) in such ratios as to obtain two types of mixtures. One contained 60 percent by volume of metallic powder and 40 percent by volume of refractory oxide powder (60-40), and the other equal volume

amounts of metallic and oxide powders (50-50). The choice of powder particle sizes was such that, on blending and hot pressing, the metallic phase would form a continuous matrix with refractory oxide grains imbedded in it. The microstructure of hot-pressed matrix material alone (gray β containing white γ precipitates) is shown in figure 1. Typical microstructures of the cermets are shown in parts (a) of figures 2 to 5. What appears to be a second phase in the grains of MgZrO_3 (fig. 5(a)) was metallographically identified as etch pits. The methods of hot pressing and of specimen preparation were described in reference 5. The 70 percent dense Ni-Cr-Al-Y development material was obtained from an outside source.

Cyclic Oxidation

Cyclic oxidation was carried out at 1100°C for 90 1-hour cycles and at 1200°C for 72 1-hour cycles, or less. Only the 70 percent dense Ni-Cr-Al-Y was given a 90-cycle exposure at both temperatures. Each exposure cycle was followed by a 20-minute cooling period in ambient air. It took about 15 minutes for the specimens to reach the test temperature after they were inserted into the furnace. After they were withdrawn from the furnace, the specimens cooled to room temperature in 7 minutes. The specimens were held in platinum baskets which were suspended in the individual ceramic tubes of a vertical tube furnace. The details of the furnace design and its operation were also described in reference 5.

The specimens were weighed after 5, 18, 36, 54, 72, and 90 cycles. The 50-50 Ni-Cr-Al-Y - MgO specimens were subjected to a furnace malfunction during oxidation at 1200°C and those test results will not be reported. After oxidation, the specimens were X-rayed on a diffractometer to determine the phase composition of the retained oxide scales or of the specimen surfaces. The spalled oxide scales were not analyzed. While cross sections of the specimens representing each cermet composition were examined metallographically, only representative ones were photographed and shown in this report.

RESULTS AND DISCUSSION

The net specific weight changes during cyclic oxidation of cermets are shown in figures 6 for the 60-40 cermets and 7 for the 50-50 cermets. Each point on these plots represents an average of two determinations. X-ray diffraction analysis of the surfaces of oxidized specimens was difficult because many diffraction lines could not be indexed. However, the phases that were identified on representative cermets are shown in table II.

Ni-Cr-Al-Y - MgO Cermets

At 1100^o C the 60-40 and 50-50 cermets showed very little weight change during 90 cycles of oxidation (figs. 6(a) and 7(a)). Moderate coarsening of the γ phase could be noted (fig. 2(b)). The spallation of oxide scale was insignificant. At 1200^o C for both types of cermets there was a slight weight increase followed by a decrease, due to spallation (figs. 6(b) and 7(b)). Significant coarsening of the γ phase was observed (fig. 2(c)). However, at both temperatures no oxidation along interphase boundaries, matrix cracking, or reaction between oxide and metallic phases was observed.

Ni-Cr-Al-Y - CaZrO₃ Cermets

At 1100^o C the 50-50 cermet oxidized twice as fast as the 60-40 cermet. Similar behavior was also observed at 1200^o C (figs. 6 and 7). The oxidation curves indicated that the effect of temperature was not as significant as that of the composition. After exposure at both temperatures, no formation of scale was observed. However, specimens experienced dimensional growth and cracking. The cracking was more pronounced at 1200^o C. The weight increase in the absence of oxide scale formation is an indication of internal oxidation or oxidation along interphase boundaries. Figures 3(b) and (c) show clearly that preferred oxidation along interphase boundaries has occurred. There is also evidence of interaction between calcium zirconate and the oxidized part of the matrix.

Ni-Cr-Al-Y - SrZrO₃ Cermets

In general, this group of cermets oxidized nearly twice as fast as the Ni-Cr-Al-Y - CaZrO₃ cermets (figs. 6 and 7). The effect of oxide content was less significant than temperature. Severe cracking and dimensional growth of the specimens characterized oxidation behavior of these cermets. The reaction of strontium zirconate with the oxidation products of the matrix was so severe that it caused the refractory oxide grains to disintegrate (figs. 4(b) and (c)). Also, a significant part of the metallic matrix was consumed by oxidation. No formation of oxide scale on the surface of the specimens was observed.

Ni-Cr-Al-Y - MgZrO₃ Cermets

The Ni-Cr-Al-Y - MgZrO₃ cermets performed much like the Ni-Cr-Al-Y - SrZrO₃

cermets except the degradation process was even more severe. Figures 5(b) and (c) illustrate the severity of the interaction of MgZrO_3 with the Ni-Cr-Al-Y matrix. Figures 6(a) and 7(a) show the net weight increases during cyclic oxidation at 1100°C of 60-40 and 50-50 cermets. The dimensional growth and cracking of the specimens at 1200°C were so pronounced that the 50-50 cermets disintegrated during testing and the oxidation of the 60-40 cermets had to be interrupted after only 31 cycles.

70 Percent Dense Ni-Cr-Al-Y

A sintered, ≈ 70 percent dense developmental Ni-Cr-Al-Y material was subjected to cyclic oxidation at 1100° and 1200°C . The results are shown in figure 6. At 1200°C it oxidized about twice as fast as at 1100°C . There was a slight dimensional growth of the specimens at 1200°C . No formation of oxide scale on external surfaces occurred and no cracking was observed. Figures 8(a) and (b) reveal clearly that oxidation occurred on the internal surfaces, which resulted in the destruction of bond areas between particles (fig. 8(b)) at 1200°C . As a result of this process, a continuous metallic network was transformed into a continuous oxide network containing porosity and metallic particles.

CONCLUDING REMARKS

Cyclic oxidation of the four cermet systems indicated that the Ni-Cr-Al-Y - MgO cermets were the most oxidation resistant. This resistance was due to compatibility between MgO and Ni-Cr-Al-Y, which was discussed in reference 5. However, there was considerable improvement over that reference's oxidation data for Ni-Cr-Al-Y cermets containing 40 percent MgO (ref. 5). This improvement may have been due to the much higher aluminum content of the Ni-Cr-Al-Y matrix in this present study and to the modification of the microstructure by decreasing the particle size of the Ni-Cr-Al-Y powder in relation to the MgO particle size.

The other three types of Ni-Cr-Al-Y - zirconate cermet did not perform well because of their thermal instability in oxidizing conditions. Their weight increases during oxidation were much more rapid. These weight increases were accompanied by pronounced dimensional growth and cracking. Internal oxidation of the specimens was the result of the rapid inward penetration of oxygen via interphase boundaries. This is indicative of poor bonding or lack of bonding between the matrix and zirconate particles. Note that in all tested cermets, the thermal expansion coefficient of Ni-Cr-Al-Y and the zirconates (ref. 10) were compatible and, therefore, could not be the cause of cracking and internal oxidation. It appears that cracking of the specimens was due to their

oxidation-produced swelling.

Disintegration of zirconate grains during oxidation was the result of their decomposition by Al_2O_3 and Cr_2O_3 (products of oxidation of Ni-Cr-Al-Y as shown in table II). According to reference 11, Al_2O_3 should react with SrZrO_3 to yield SrAl_2O_4 , $\text{Sr}_3\text{Al}_2\text{O}_6$, and other compounds, including $\text{Sr}_2\text{Al}_{12}\text{ZrO}_{22}$. These complex oxides could well be the origin of the diffraction lines that could not be indexed (table II). It is reasonable to assume that the disintegration of CaZrO_3 and MgZrO_3 is also due to their instability in the presence of Al_2O_3 . Similarly, it was established (ref. 12) that CaZrO_3 is decomposed by Cr_2O_3 . In this process chromia forms CaCrO_3 . It is probable that SrZrO_3 and MgZrO_3 are also unstable in the presence of Cr_2O_3 . Some of the compounds that are products of indicated reactions can have larger molar volumes than their component oxides and thus contribute to the dimensional growth of the specimens (in addition to the growth caused by internal oxidation of the matrix). A typical example of such a phenomenon would be the formation of MgAl_2O_4 spinel in Ni-Cr-Al-Y - MgZrO_3 cermets. The reaction $\text{MgO} + \text{Al}_2\text{O}_3 \rightarrow \text{MgAl}_2\text{O}_4$ is accompanied by a volume increase of about 10 percent.

It is obvious, therefore, that cermets made with CaZrO_3 , SrZrO_3 , or MgZrO_3 , and any superalloy containing aluminum or chromium would not be as thermally stable and oxidation resistant as MgO - Ni-Cr-Al-Y cermets. The suitability of the MgO - Ni-Cr-Al-Y material for potential turbine seal service, however, still requires evaluation of many factors including abrasibility.

SUMMARY OF RESULTS AND CONCLUSIONS

The cyclic oxidation study at 1100° and 1200° C of hot pressed Ni-Cr-Al-Y based cermets containing 40 and 50 volume percent of MgO, CaZrO_3 , SrZrO_3 , or MgZrO_3 and of a 70 percent dense Ni-Cr-Al-Y developmental material demonstrated the following:

1. Ni-Cr-Al-Y - MgO cermets were the most oxidation resistant. This was due to the thermal stability of this metal-oxide system.

2. Cermets with zirconates, listed in order of decreasing resistance, were CaZrO_3 - Ni-Cr-Al-Y, SrZrO_3 - Ni-Cr-Al-Y, and MgZrO_3 - Ni-Cr-Al-Y. These metal-oxide systems were not thermally stable as manifested by the reaction of zirconates with matrix oxidation products along interphase boundaries resulting in dimensional growth of the specimens and their subsequent cracking. Based on these observations the zirconate-Ni-Cr-Al-Y cermets appear to have little high temperature service potential despite the favorable match in coefficient of thermal expansion between zirconates and the superalloy matrix.

3. The 70 percent dense Ni-Cr-Al-Y developmental material, due to its porous nature, exhibited an intermediate oxidation weight change and some dimensional growth.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 10, 1976,
501-01.

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TABLE I. - CHEMICAL COMPOSITION OF
OXIDES USED IN THE PREPARATION
OF CERMETS

Simple oxide	Compound oxide			
	CaZrO ₃	MgZrO ₃	SrZrO ₃	MgO
	Simple oxide content, wt. %			
CaO	30.8	----	----	----
MgO	.3	24.0	0.2	97.0
SrO	----	----	44.0	----
SiO ₂	.7	.5	.4	1.7
Fe ₂ O ₃	.2	.2	.1	.2
TiO ₂	.2	.2	.1	.05
Al ₂ O ₃	.3	.3	.2	.1
ZrO ₂	bal.	bal.	bal.	----

TABLE II. - X-RAY DIFFRACTION ANALYSIS OF RETAINED
OXIDE SCALES OF SELECTED CERMETS

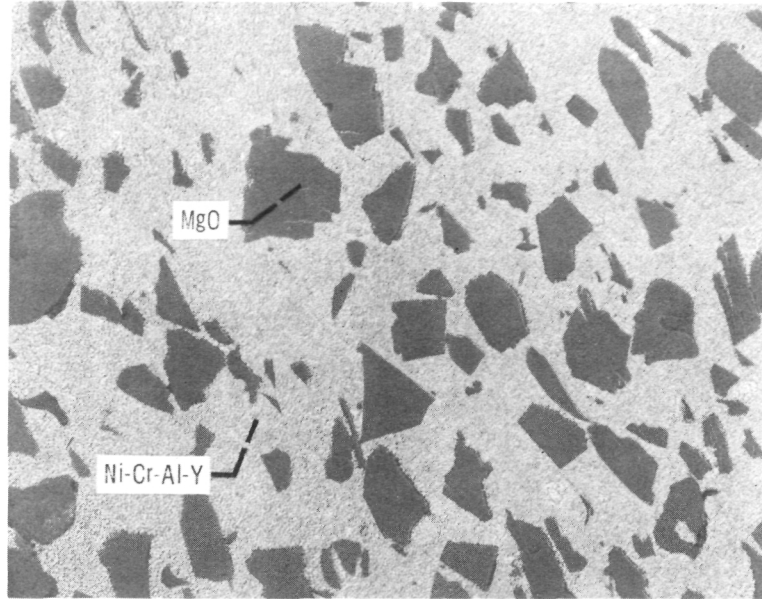
Cermets	Nominal composition, vol%	Oxidation temperature, °C	
		1100	1200
		Analysis results ^{a, b}	
Ni-Cr-Al-Y - MgO	60-40	MgO, VS α -Al ₂ O ₃ , M Spinel ($a_0 = 8.10 \text{ \AA}$), VW γ -phase, W X	MgO, VS α -Al ₂ O ₃ , VW Spinel, ($a_0 = 8.25 \text{ \AA}$), M γ -phase, VW X
Ni-Cr-Al-Y - CaZrO ₃	50-50	CaZrO ₃ , W α -Al ₂ O ₃ , S Spinel ($a_0 = 8.10 \text{ \AA}$), W AlNi, S γ -phase, W XX	γ -phase, S CaZrO ₃ , S Cr ₂ O ₃ , M AlNi, M XX
Ni-Cr-Al-Y - SrZrO ₃	60-40	γ -phase, VS α -Al ₂ O ₃ , M AlNi, M Spinel ($a_0 = 8.30 \text{ \AA}$), W ZrO ₂ , monoclinic ZrO, cubic	γ -phase, VS SrZrO ₃ , M Cr ₂ O ₃ , W XXX
Ni-Cr-Al-Y - MgZrO ₃	60-40	γ -phase, VS MgO, M α -Al ₂ O ₃ , M Cr ₂ O ₃ , W ZrO ₂ , cubic ZrO ₂ , monoclinic XXX	AlNi, M MgZrO ₃ , S α -Al ₂ O ₃ , M Cr ₂ O ₃ , W Spinel ($a_0 = 8.35 \text{ \AA}$), S XX
70% dense Ni-Cr-Al-Y	-----	α -Al ₂ O ₃ , S γ -phase ($a_0 = 3.57 \text{ \AA}$), VS XX	α -Al ₂ O ₃ , S γ -phase ($a_0 = 3.60 \text{ \AA}$), VS Cr ₂ O ₃ , M NiO, VW NiAl, VW

^aRelative pattern intensities: VS = very strong, S = strong, M = medium, W = weak, VW = very weak.

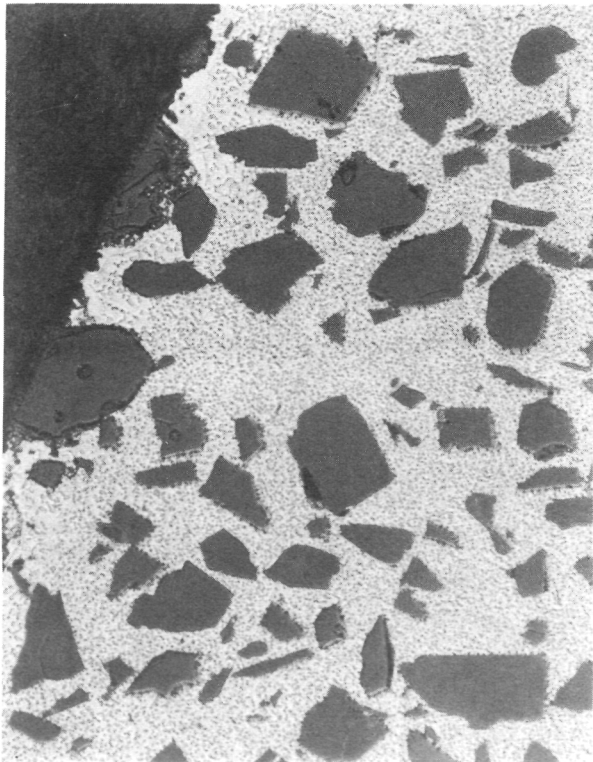
^bNumber of unidentified lines: X = one, XX = two to four, XXX = more than four.



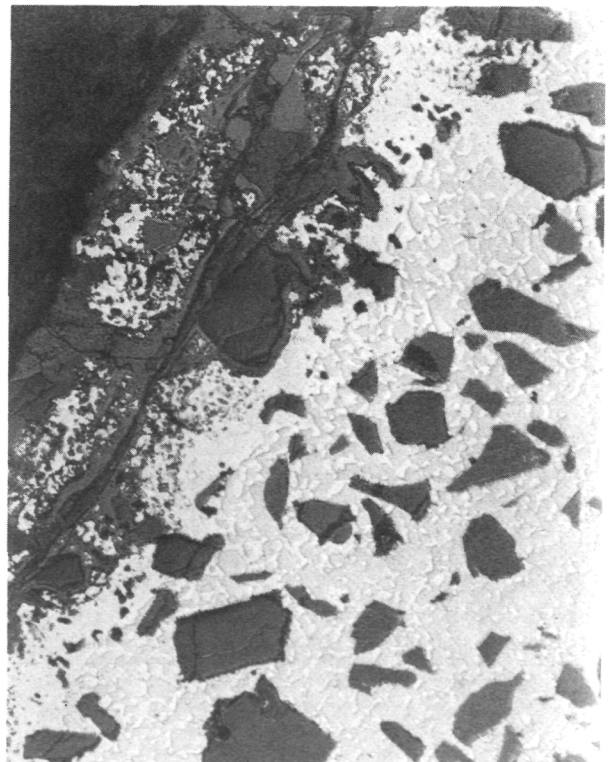
Figure 1. - Microstructure of hot pressed Ni-Cr-Al-Y powder. White phase is γ . Etched; X750.



(a) As fabricated.

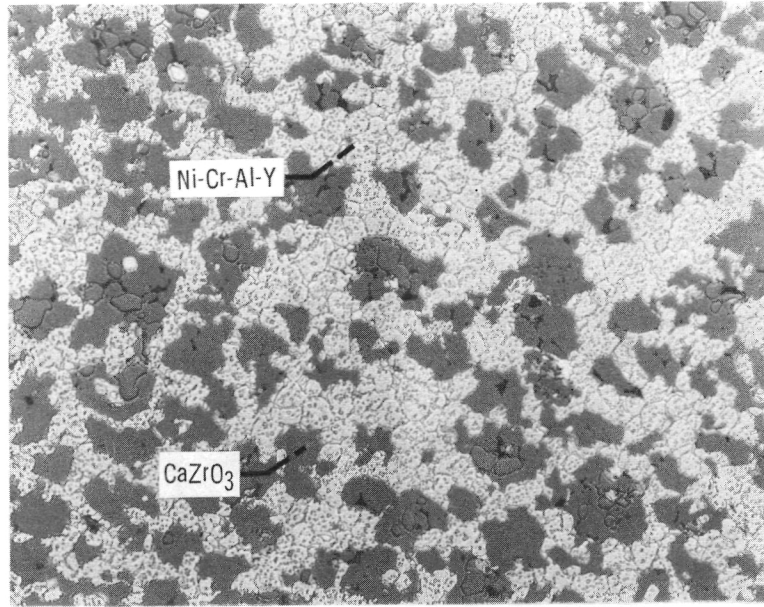


(b) After 90 cycles at 1100° C.

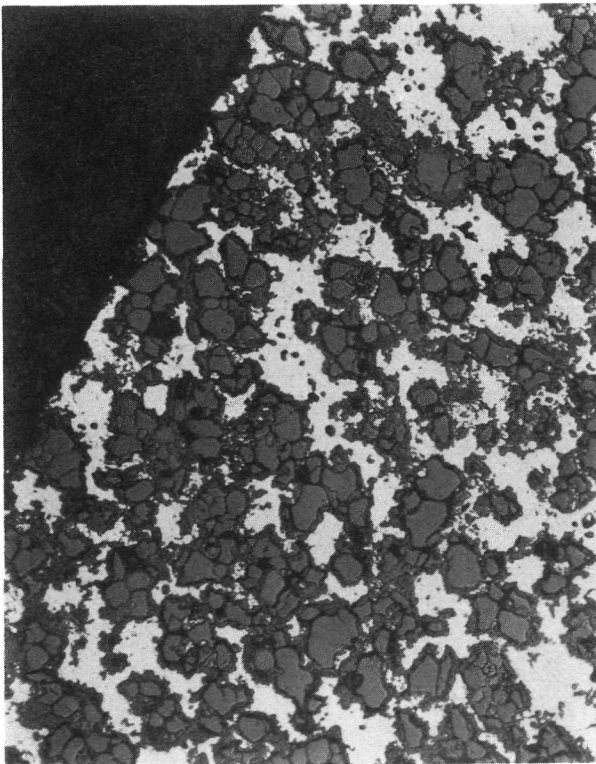


(c) After 72 cycles at 1200° C.

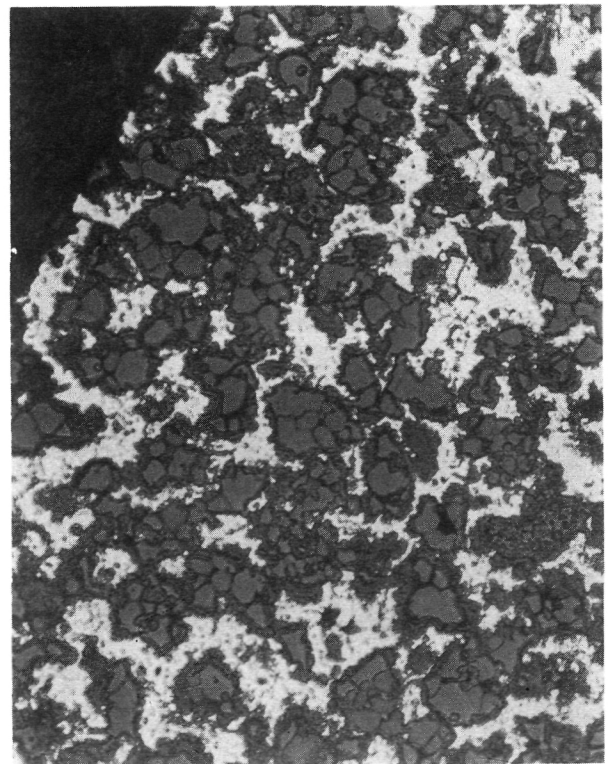
Figure 2. - Microstructure of Ni-Cr-Al-Y – MgO 60-40 cermet as fabricated and after oxidation at 1100° and 1200° C. Etched; X100.



(a) As fabricated.

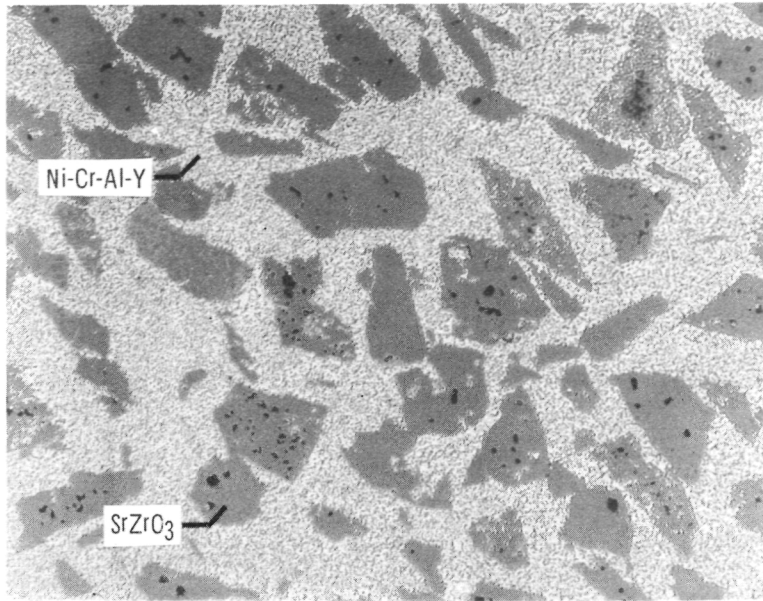


(b) After 90 cycles at 1100° C.

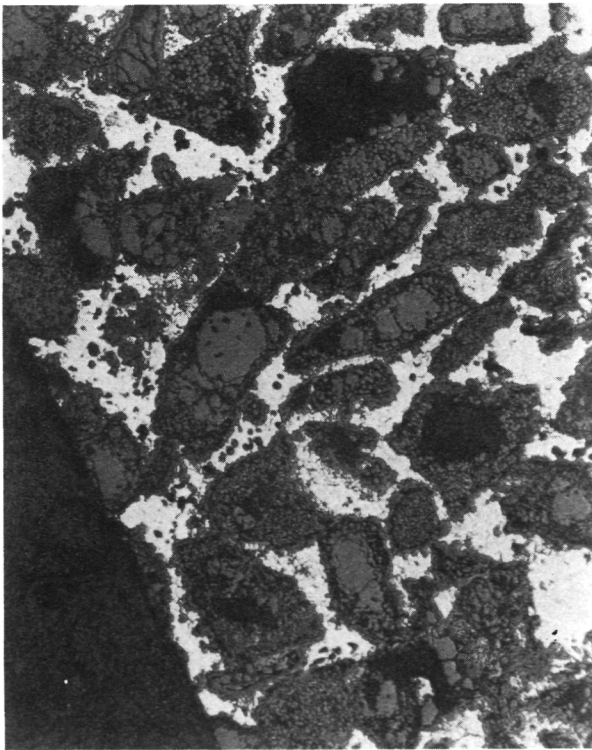


(c) After 31 cycles at 1200° C.

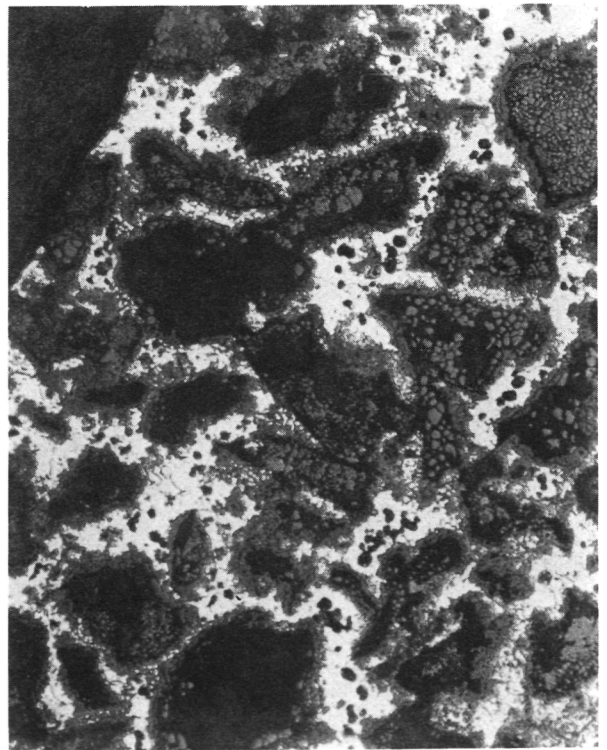
Figure 3. - Microstructure of Ni-Cr-Al-Y - CaZrO₃ 50-50 cermet as fabricated and after oxidation at 1100° and 1200° C. Etched; X100.



(a) As fabricated.

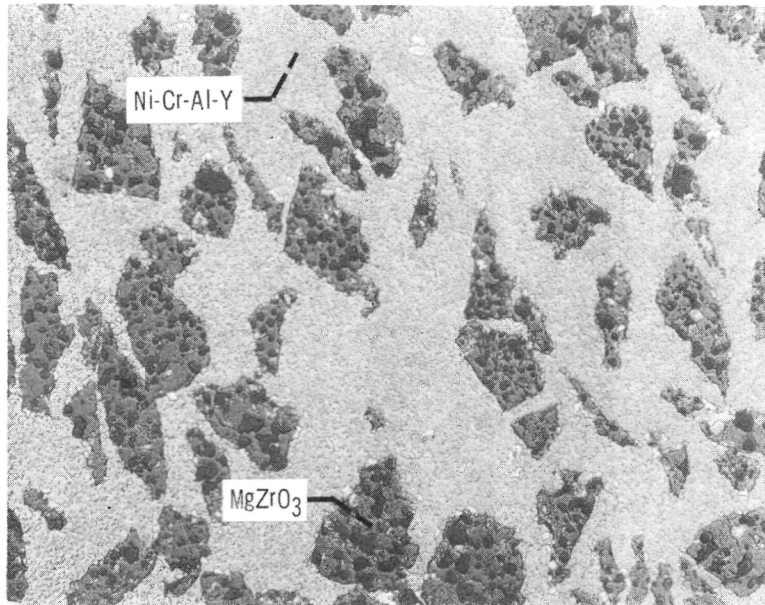


(b) After 90 cycles at 1100° C.



(c) After 72 cycles at 1200° C.

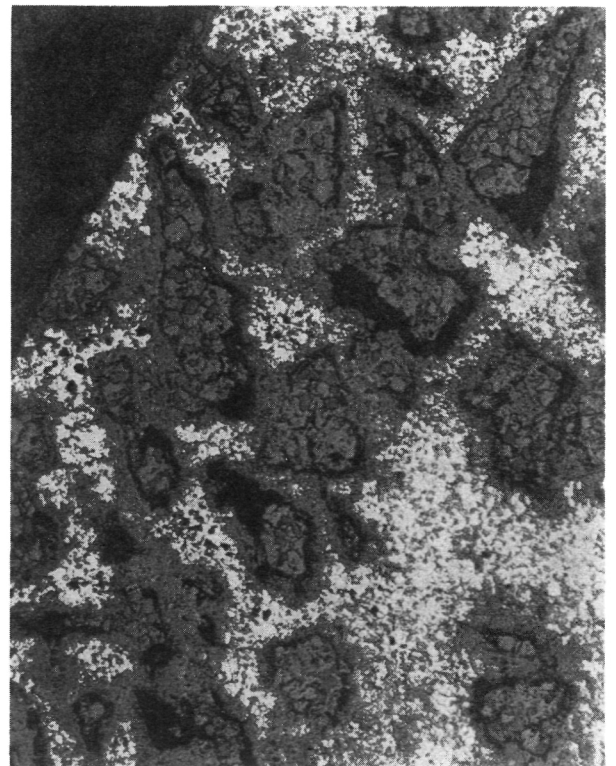
Figure 4. - Microstructure of Ni-Cr-Al-Y - SrZrO₃ 60-40 cermet as fabricated and after oxidation at 1100° and 1200° C. Etched; X100.



(a) As fabricated.



(b) After 90 cycles at 1100°C.



(c) After 72 cycles at 1200°C.

Figure 5. - Microstructure of Ni-Cr-Al-Y - MgZrO₃ 60-40 cermet as fabricated and after oxidation at 1100° and 1200° C.
Etched; X100.

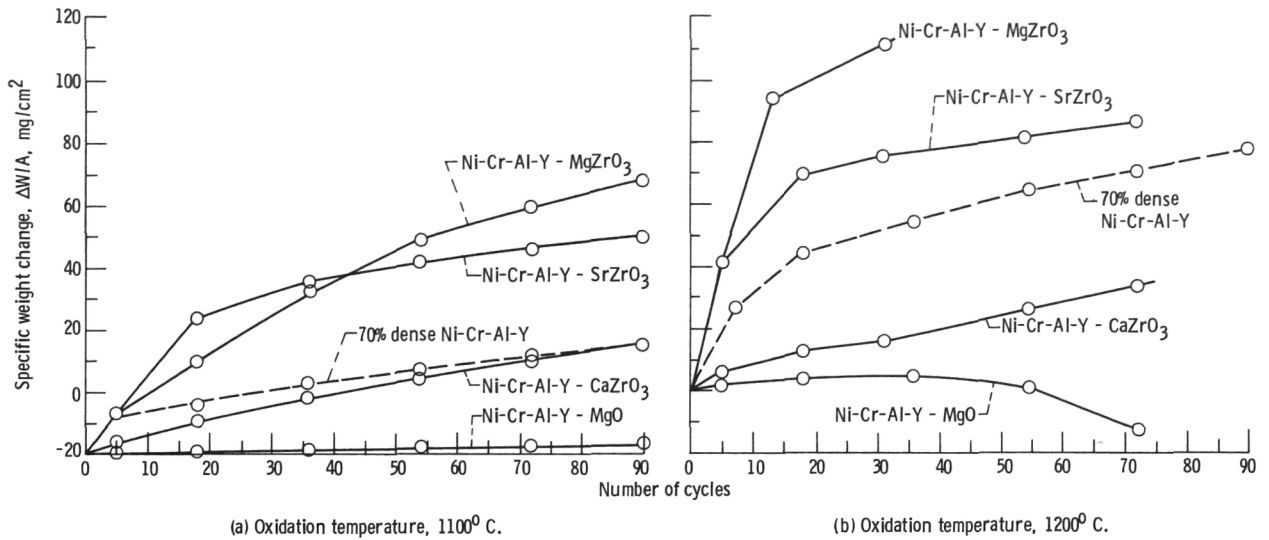


Figure 6. - Sample specific weight change of 60-40 Ni-Cr-Al-Y - MgO , Ni-Cr-Al-Y - CaZrO_3 , Ni-Cr-Al-Y - SrZrO_3 , and Ni-Cr-Al-Y - MgZrO_3 cermets and of 70 percent dense Ni-Cr-Al-Y. 1-Hour cycles in still air.

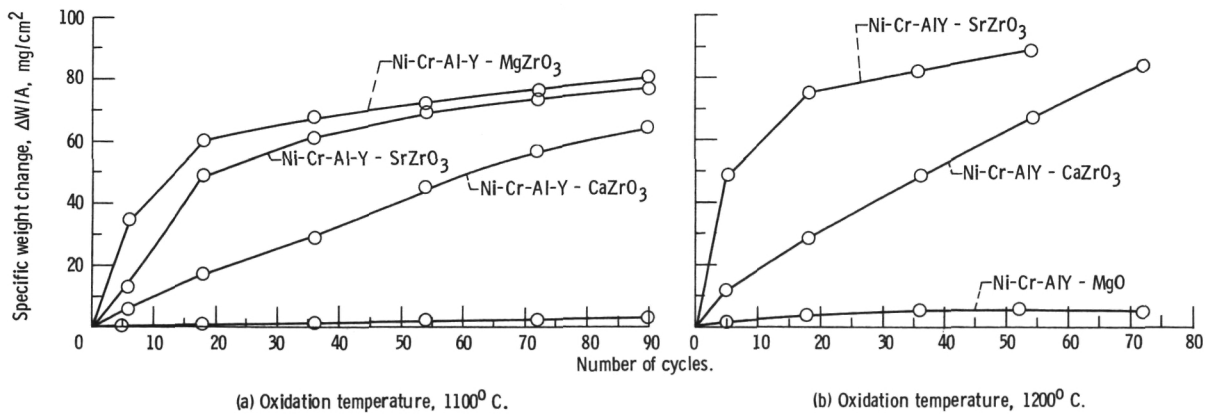
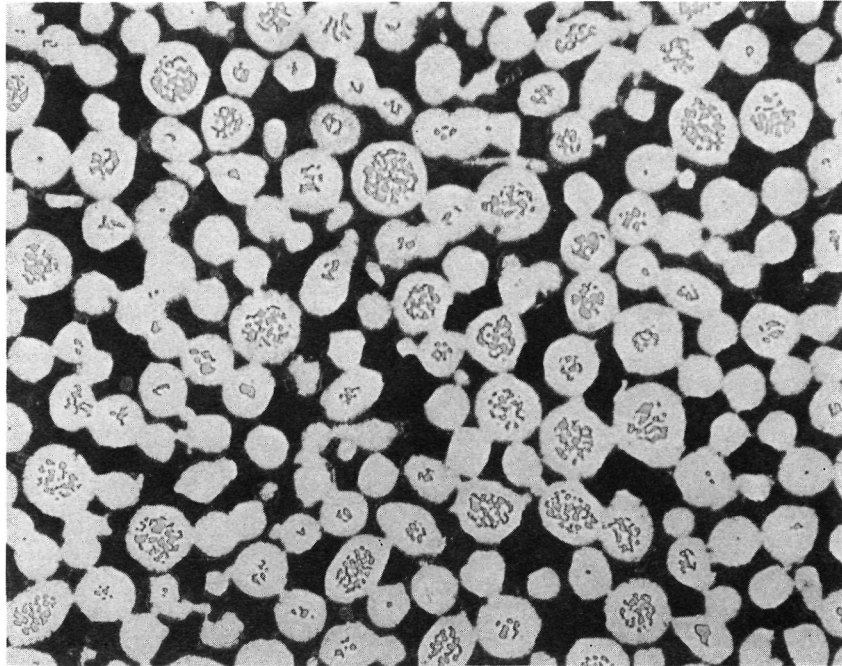
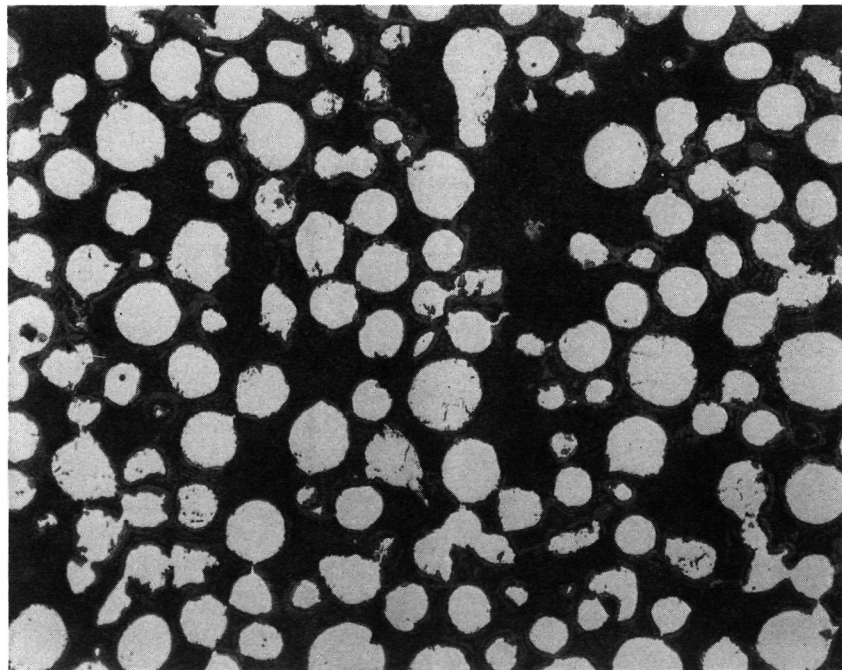


Figure 7. - Sample specific weight change of 50-50 Ni-Cr-Al-Y - MgO , Ni-Cr-Al-Y - CaZrO_3 , Ni-Cr-Al-Y - SrZrO_3 , and Ni-Cr-Al-Y - MgZrO_3 cermets. 1-Hour cycles in still air.



(a) After 90 cycles at 1100°C.



(b) After 90 cycles at 1200°C.

Figure 8. - Microstructure of a developmental 70 percent dense Ni-Cr-Al-Y material after oxidation at 1100° and 1200° C. Etched; X100.



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—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

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