

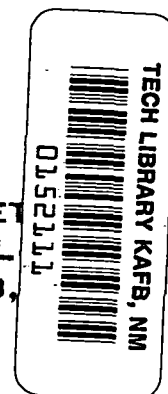
**NASA TECHNICAL  
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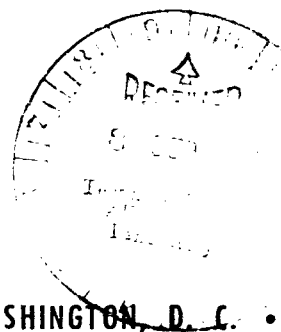


**EFFECT OF MAGNESIUM OXIDE CONTENT  
ON OXIDATION BEHAVIOR OF SOME  
SUPERALLOY-BASE CERMETS**

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## ERRATA

NASA Technical Memorandum X-3265

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August 1975

The composition of the alloy listed as Hastelloy X in table I, page 9, does not fit the specifications for HASTELLOY ® (a registered trademark of the Cabot Corporation) alloy X. Further, an analysis of this alloy indicated deviation from the specifications shown in table I. The following corrected table shows the correct analysis of this alloy, henceforth called Alloy A. Thus, throughout the report, references to Hastelloy X should be read to mean Alloy A. Further, references to Inconel-702 should be read to mean INCONEL ® alloy 702.

N75-29/87

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TABLE I. - CHEMICAL ANALYSIS OF SUPERALLOY POWDERS

Superalloy	Al	C	Cr	Fe	Mo	Mn	Ni	O <sub>2</sub>	Si	S	W	Y
	Content, wt % <sup>a</sup>											
NiCrAlY	5.30	0.012	16.2	96 ppm	----	----	Bal.	75 ppm	60 ppm	60 ppm	----	0.17
Hoskins-875	4.69	-----	21.99	Bal.	----	----	----	160 ppm	0.49	-----	----	----
INCONEL ® <sup>b</sup> alloy 702	3.13	.008	15.95	0.40	----	0.09	Bal.	170 ppm	.12	0.003	----	----
Alloy A	----	.005	20.46	18.90	9.09	<.10	Bal.	300 ppm	-----	-----	0.65	----

<sup>a</sup>Except for those values designated as ppm.

<sup>b</sup>A registered trademark of International Nickel Company.

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16. Abstract <p>The resistance to cyclic oxidation at 1100° and 1200° C of cermets hot pressed from MgO powder (5, 10, 20, and 40 vol %) mixed with NiCrAlY, Hoskins-875, Inconel-702, or Hastelloy-X powders was determined. Cermets of MgO in NiCrAlY and MgO in Hoskins-875 were superior to cermets of MgO in Inconel-702 and MgO in Hastelloy-X in the 5- to 20-vol %-MgO range. Their oxidation resistance was reduced when the MgO content was 40 vol %. The existence of bonding between MgO and the metallic phases was observed.</p>					
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# EFFECT OF MAGNESIUM OXIDE CONTENT ON OXIDATION BEHAVIOR OF SOME SUPERALLOY-BASE CERMETS

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## SUMMARY

The effect of increasing magnesium oxide (MgO) content on the cyclic oxidation resistance of hot-pressed cermets of MgO in NiCrAlY, MgO in Hoskins-875, MgO in Inconel-702, and MgO in Hastelloy-X was investigated. The cermets with magnesium oxide levels of 5, 10, 20, and 40 vol % were examined. The cyclic oxidation behavior of these cermets at 1100° and 1200° C in still air was determined by a thermogravimetric method supplemented by X-ray diffraction analysis and light and electron microscopy. In all instances, MgO prevented grain growth in the metallic phase. No evidence of oxidation along interphase boundaries was detected. Cermets of MgO in NiCrAlY and MgO in Hoskins-875 were superior to cermets of MgO in Inconel-702 and MgO in Hastelloy-X. Their oxidation resistance was degraded only when the MgO content was 40 vol %. The oxidation behavior of MgO-in-Inconel-702 powder cermets containing 5- and 10-vol % MgO was approximately similar to that of pure Inconel-702 compacts. However, 20- and 40-vol % MgO content reduced the oxidation resistance of MgO-in-Inconel-702 powder cermets relative to that of pure Inconel-702.

## INTRODUCTION

Designs for jet engines with higher thrust and lower specific fuel consumption are based on higher operating pressures, temperatures, and rotational speeds. Because engine performance is directly related to minimizing gas path leakage in the turbine, these designs place increasing demands on turbine blade-tip seal technology. One approach to the sealing problem is to replace presently used materials by more oxidation-resistant ones, in such a way that they can be accommodated in the basic shroud design currently in use. It is obvious that superior oxidation resistance should not be the only criterion for defining improved turbine seal materials. They must also resist thermal shock, thermal fatigue, impact, and erosion and yet be abradable without causing the

transfer of blade tip material to the seal face (scabbing).

One approach to producing improved abradable metallic seals is to fabricate metal powder compacts with controlled porosity (20 to 50 percent). In such compacts, the particle-to-particle bonding is sufficiently weak to allow the particle bonds to shear and break away on rubbing by blade tips. An alternate approach is to fabricate 100-percent-dense compacts with a certain amount of inert filler to provide the friability necessary for adequate rubbing characteristics. These rubbing characteristics would be primarily a function of the amount and nature of the filler in the compact. Such an inert filler must not negatively affect the oxidation resistance of the seal material.

The application of cermets to aircraft powerplants is discussed by Meyer and Deutsch in reference 1. Tinklepaugh and Crandall in their book on cermets (ref. 2) present an extensive bibliography on the subject, covering the period 1949-1958. In the last decade, the literature on metal-oxide cermets has been sparse. However, one work (ref. 3) is somewhat related to this subject. It is concerned with the oxidation resistance and mechanical properties of cermet coatings (50 nickel (Ni) - 50 MgO and 50 chromium (Cr) - 50 MgO) on carbon steels. Both types of coatings protected the base metal satisfactorily to 1200<sup>0</sup> C.

This study was conducted to determine the effect of oxide filler content on the oxidation resistance of hot-pressed compacts (cermets) based on Hastelloy-X, Inconel-702, Hoskins-875, and nickel-chromium-aluminum-yttrium (NiCrAlY) powders. Magnesium oxide in amounts of 5, 10, 20, and 40 vol % was used as the inert filler. In this report a powder cermet of MgO in NiCrAlY, for example, is designated as MgO + NiCrAlY. The oxidation behavior of these cermets was studied in still air at 1100<sup>0</sup> and 1200<sup>0</sup> C under cyclic furnace conditions by a thermogravimetric method supplemented by optical and electron microscopy and by X-ray diffraction analysis of the oxide scales.

## EXPERIMENTAL PROCEDURES

### Sample Preparation

The metallic materials Hastelloy-X, Inconel-702, Hoskins-875, and NiCrAlY were procured in powder form. These alloys were selected for their good oxidation resistance. Their chemical analyses are shown in table I. Typical analysis of the MgO used in the preparation of the cermet compacts is as follows:

Compound	Content, wt %
Calcium oxide (CaO)	0.9
Silicon dioxide (SiO <sub>2</sub> )	.45
Iron oxide (Fe <sub>2</sub> O <sub>3</sub> )	.2
Aluminum oxide (Al <sub>2</sub> O <sub>3</sub> )	.25
Magnesium oxide (MgO)	<sup>a</sup> 98.2

<sup>a</sup>By difference.

The metallic powders (-100+200 mesh) were blended with varying amounts of MgO (-270+325 mesh) to obtain mixtures containing 5, 10, 20, and 40 vol % of MgO grain. The mixtures, as well as the pure metallic powders, were packed in stainless-steel cans, which were then sealed in vacuum by electron welding and isostatically compacted at 1290° C under  $10.5 \times 10^7$ -N/m<sup>2</sup> pressure for 1 hour. The heatup time was 2 hours, and the cooldown time was 3 hours. Subsequently, the stainless-steel cans were removed by grinding, and the cermet compacts were cut into specimens approximately 2.0 cm by 1.2 cm by 0.3 cm. Metallographic examination of polished and etched surfaces of the metal and cermet compacts established that isostatic hot pressing produced nearly 100-percent-dense materials. Their densities are listed in table II. The etchant used to reveal the microstructure was a mixture of 33 parts water, 33 parts acetic acid, 33 parts nitric acid, and 1 part hydrofluoric acid. The surface of each specimen was finished with 320-grit emery paper, and all edges were given a small radius by grinding. Prior to testing, the samples were degreased, ultrasonically cleaned, and weighed to the nearest milligram. Their dimensions were then measured with a micrometer to the nearest 0.001 centimeter. Superalloy powders without the addition of MgO were similarly fabricated into specimens and oxidized in order to provide baseline data for comparison.

### Cyclic Oxidation

Cyclic oxidation was carried out at 1100° and 1200° C in a vertical tube furnace for a total of ninety 1-hour cycles. Each exposure cycle was followed by 20 minutes of cooling in ambient air. It took about 1.5 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 furnace consisted of six alumina tubes arranged in a circular fashion in one large heating unit. The bottoms of the tubes were sealed to ensure the condition of still-air oxidation. As shown in figure 1, the specimens were automatically

raised and lowered by pneumatic cylinders that were controlled by reset timers operating solenoid valves. The specimens were weighed after 5, 18, 36, 59, 72, and 90 cycles. When a specimen exhibited rapid oxidation, the experiment was terminated before 90 cycles were reached. After oxidation the specimens were X-rayed on a diffractometer in order to determine the phase composition of the retained oxide scales. The spalled oxide scales were not analyzed. Cross sections of the specimens, representing each cermet composition and each superalloy, were examined metallographically. Electron microscopy was also used in order to examine the boundary between the ceramic and metallic phases.

## RESULTS AND DISCUSSION

The net specific weight changes during cyclic oxidation are shown in figures 2 to 5. These weight changes are the difference between the weight gain by oxidation and the weight loss by spalling. Each point on these plots represents an average of two determinations. The scale of the ordinate axis in those figures illustrating data obtained at 1100° C is 10 times greater than that in figures illustrating 1200° C data. Sample specific weight changes  $\Delta W/A$  after ninety 1-hour cycles at 1100° and 1200° C are summarized in table III. The numbers in the table are the average of the two determinations. The results obtained from X-ray diffraction analysis of retained oxide scale are shown in table IV. The absence of diffraction lines from the metallic phase was used as a qualitative indication of the oxide-scale thickness. For Hastelloy-X, Inconel-702, and NiCrAlY cermets, it was impossible to separate diffraction lines produced by nickel oxide (NiO) and MgO. These two oxides have the same structure and quite similar lattice parameters and are completely soluble in each other. Therefore, both oxides are listed as an MgO-NiO phase. There was an obvious increase in the intensity of this phase's diffraction lines when the content of MgO in a cermet sample was increased.

### MgO + NiCrAlY Cermets

At 1100° C, the NiCrAlY alloy and its MgO cermets showed little weight change during 90 cycles of oxidation, except for the composition which contained 40-vol % MgO. This composition gained weight for 36 cycles but exhibited very rapid weight loss after 72 cycles. Also, at 1200° C, the 40-vol %-MgO composition oxidized rapidly and lost weight at a nearly linear rate (figs. 2(a) and (b)). Metallographic examination of this group of compacts indicated that they were nearly 100 percent dense. Magnesia-free NiCrAlY compacts exhibited extensive grain growth at 1100° and 1200° C. No preferred oxidation along grain boundaries or metal-oxide interfaces was observed (fig. 6). In-



creasing the MgO content in these cermets reduced the intensity of X-ray diffraction lines from the alloy, thus indicating thickening of the retained oxide scale. The component phases in the scale (table IV) were  $\alpha$ -alumina ( $\alpha$ - $\text{Al}_2\text{O}_3$ ), chromia ( $\text{Cr}_2\text{O}_3$ ), spinel, and NiO-MgO solid solution. Their presence depended on the MgO content and the temperature of cyclic oxidation. No  $\text{Al}_2\text{O}_3$  was detected in the 20- and 40-vol %-MgO cermets, and  $\text{Cr}_2\text{O}_3$  was noted only in the latter. As the MgO content in the compacts was increased, the composition of the spinel changed from an aluminate to a more complex spinel with a larger lattice parameter.

#### MgO + Hoskins-875 Cermets

In general, the Hoskins-875 alloy and its MgO cermets displayed oxidation behavior similar to that of the NiCrAlY-type cermets. At  $1100^\circ\text{C}$ , the weight changes were small. The most significant weight change of  $9\text{ mg/cm}^2$  (after 72 cycles) was exhibited by the cermet containing 40-vol % MgO (fig. 3(a)). Again at  $1200^\circ\text{C}$  (fig. 3(b)), the oxidation behavior of the alloy and its 5-, 10-, and 20-vol %-MgO cermets was approximately the same. The cermet with 40-vol % MgO lost weight so rapidly that the oxidation experiment had to be interrupted after 36 cycles.

Metallographic examination (fig. 7) of this group of cermet compacts proved that they were nearly 100 percent dense. A sample of MgO-free Hoskins-875 exhibited an irregular grain growth when oxidized at  $1200^\circ\text{C}$ , but not at  $1100^\circ\text{C}$ . No preferred oxidation along grain boundaries or at metal-oxide interfaces was detected. After oxidation at  $1200^\circ\text{C}$ , the grains of the alloy in the 20- and 40-vol %-MgO cermets had some precipitate in the zone near the surface of specimens. Similar precipitates could be observed after oxidation at  $1100^\circ\text{C}$  but only for the 40-vol %-MgO cermet. This precipitate, which exhibited hexagonal symmetry, is probably  $\text{Al}_2\text{O}_3$  and is due to internal oxidation of the alloy.

Generally, the X-ray diffraction lines from the retained oxide scale and from the alloy were strong. The component oxides in the scale (table IV) were  $\alpha$ - $\text{Al}_2\text{O}_3$ , spinel, and MgO (or MgO-FeO solid solution). However, the scale on Hoskins-875 powder compacts (without MgO) did not contain the spinel phase. Apparently, the presence of MgO in the cermets promoted the formation of a spinel. Furthermore, increasing MgO content was accompanied by an increase in the lattice parameter of the spinel. And in the case of the 40-vol %-MgO cermet,  $\alpha$ - $\text{Al}_2\text{O}_3$  was not detected at all.

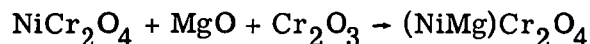
#### MgO + Inconel-702 Cermets

The curves representing the specific weight change against number of oxidation cycles were quite similar for both Inconel-702 and 5- and 10-vol %-MgO + Inconel-702

cermets (figs. 4(a) and (b)). Some weight loss was observed. Cermets with 20- and 40-vol % MgO content behaved differently. At 1100° C, both these cermets showed an initial weight gain followed by a weight loss. At 1200° C, both cermets lost weight nearly from the start of the oxidation experiment, and the rate of weight loss was more rapid, particularly in the case of the 40-vol %-MgO cermet. Metallographic examination of polished and etched surfaces (fig. 8) of selected compositions, before and after oxidation, revealed that the compacts made of Inconel-702 powder with no MgO were not completely dense. During oxidation at 1100° and 1200° C, these compacts underwent oxidation along the grain boundaries and pores (figs. 8(d) and (g)). In the case of the MgO + Inconel-702 cermets, no preferred oxidation was detected either along grain boundaries or along metal-oxide interfaces. The retained oxide scale was thin enough to allow an X-ray diffraction pattern of the metallic phase to be detected. The phases present in the oxide scale (table IV) were spinel, NiO-MgO, and Cr<sub>2</sub>O<sub>3</sub> (at 1100° C) or Al<sub>2</sub>O<sub>3</sub> (at 1200° C).

#### MgO + Hastelloy-X Cermets

At 1100° C, both Hastelloy-X and MgO + Hastelloy-X cermets showed an initial weight increase followed by a weight loss (fig. 5(a)). At 1200° C (fig. 5(b)), the spalling rate was so rapid that the oxidation runs had to be terminated before 90 cycles were completed. At both temperatures the MgO + Hastelloy-X cermets were more oxidation resistant than the superalloy compacts without MgO. Metallographic examination of polished and etched surfaces (figs. 9(a) to (c)) revealed that the compacts were completely dense. It is also evident that MgO inhibited grain growth in the metallic phase (compare figs. 9(g) and (h)). The compacts that were oxidized at 1100° C developed some near-surface porosity or pitting, which apparently had no relation to the presence of MgO. Both Hastelloy-X and MgO + Hastelloy-X cermets exhibited the same type of porosity (figs. 9(d) to (f)). Furthermore, no preferred oxidation was detected either along grain boundaries or along metal-oxide interfaces. The phases present in the retained oxide scale were Cr<sub>2</sub>O<sub>3</sub>, spinel, and MgO-NiO. The lattice parameter of the spinel decreased as the MgO content was increased, presumably because of the following change in composition:



The cermets containing 20- and 40-vol % MgO when oxidized at 1100° C produced a scale with an additional phase, which could not be identified. In general, the retained oxide was thick enough not to allow X-ray diffraction detection of the alloy.

## CONCLUDING REMARKS

The cyclic oxidation data for the four groups of cermets indicated that magnesium oxide (MgO) + Hastelloy-X and MgO + Inconel-702 cermets had poorer oxidation resistance than MgO + NiCrAlY and MgO + Hoskins-875 cermets. Metallographic examination of all the cermets revealed that MgO is indeed a promising ceramic phase in these materials. The "compatibility" between MgO and the superalloys was reflected by the absence of preferred oxidation along metal-oxide interfaces. This compatibility is due to the facts that the thermal expansion coefficient of MgO is rather large ( $13.5 \times 10^{-6}$  cm/cm/ $^{\circ}$ C, ref. 4) and that MgO can form a bond with the oxides that form on alloys. The electron microscope photographs presented in figure 10 are direct evidence for the existence of such a bond in the MgO + NiCrAlY and MgO + Hoskins-875 systems. In general, metal-oxide systems show poor bonding and sintering characteristics. However, in the preceding cases and for alumina-chromium ( $\text{Al}_2\text{O}_3$ -Cr) (ref. 5), there is bond formation. Nickel and cobalt (or their alloys) and Cr, when exposed to oxygen, are known to form tightly adherent oxides which are isomorphous with  $\text{Al}_2\text{O}_3$  or MgO. These oxides (NiO, CoO,  $\text{Cr}_2\text{O}_3$ , etc.) readily go into solid solution with MgO or  $\text{Al}_2\text{O}_3$  and thus form a strong bond.

During the preparation of the cermets, all processing parameters were kept constant; the only variable was MgO content. However, it stands to reason that another variable parameter, namely, the relative powder particle size of the MgO and of the superalloy, offers a further means of modifying the microstructure and properties of such cermets.

## CONCLUSIONS

A study of the cyclic, still-air oxidation at  $1100^{\circ}$  and  $1200^{\circ}$  C of hot-pressed cermets based on nickel-chromium-aluminum-yttrium (NiCrAlY), Hoskins-875, Inconel-702, and Hastelloy-X powders mixed with 5- to 40-vol % magnesium oxide (MgO) revealed the following:

1. Cermets of MgO + NiCrAlY were the most oxidation resistant. They were followed, in order of decreasing resistance, by cermets of MgO + Hoskins-875, MgO + Inconel-702, and MgO + Hastelloy-X.
2. The oxidation resistance of MgO + NiCrAlY and MgO + Hoskins-875 cermets was not negatively affected by MgO content, until the level reached 40 vol %.
3. Increasing the MgO content improved the oxidation resistance of MgO + Hastelloy-X cermets.
4. The presence of MgO in cermets inhibited grain growth in the metallic phase.

5. There is evidence for the existence of good bonding between MgO and the metallic phases.

Lewis Research Center,  
National Aeronautics and Space Administration,  
Cleveland, Ohio, May 20, 1975,  
505-01.

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Hoskins-875	4.69	-----	21.99	Bal.	----	----	----	160 ppm	0.49	-----	----	----
INCONEL® <sup>b</sup> alloy 702	3.13	.008	15.95	0.40	----	0.09	Bal.	170 ppm	.12	0.003	----	----
Alloy A	----	.005	20.46	18.90	9.09	<.10	Bal.	300 ppm	-----	-----	0.65	----

<sup>a</sup>Except for those values designated as ppm.

<sup>b</sup>A registered trademark of International Nickel Company.

TABLE II. - DENSITY OF HOT-PRESSED

CERMETS AS DETERMINED BY

DISPLACEMENT METHOD

Superalloy powder base	MgO content, vol %				
	0	5	10	20	40
	Density, g/cm <sup>3</sup>				
NiCrAlY	7.95	7.87	7.55	7.08	6.27
Hoskins-875	7.22	7.12	7.00	6.52	5.77
Inconel-702	8.10	8.00	7.64	7.26	6.37
Hastelloy-X	8.40	8.20	8.08	7.43	6.44

TABLE III. - SUMMARY OF SAMPLE SPECIFIC WEIGHT CHANGES AFTER NINETY<sup>a</sup>

1-HOUR OXIDATION CYCLES AT 1100° AND 1200° C

[Values for duplicate runs are shown.]

Superalloy powder base	MgO content, vol %									
	0		5		10		20		40	
	Oxidation temperature, °C									
	1100	1200	1100	1200	1100	1200	1100	1200	1100	1200
	Weight change, ΔW/A, mg/cm <sup>2</sup>									
NiCrAlY	-3.3	-7.3	+3.8	-0.18	+0.4	-0.43	+0.74	-24.2	-51.0	<sup>b</sup> -281.0
	-.1	-2.5	+2.9	+.01	+.5	-1.04	+.6	-12.5	-49.1	<sup>b</sup> -292.0
Hoskins-875	-0.6	-4.4	+0.8	+0.93	+1.4	+1.36	+2.6	+5.38	+6.7	<sup>c</sup> -403.8
	-.2	-4.3	-3.2	+1.53	+1.4	+2.61	+2.5	-10.9	-2.6	<sup>c</sup> -443.8
Inconel-702	+5.1	-1.24	-3.8	-32.2	-3.5	-18.2	+1.7	-251.0	-38.7	<sup>d</sup> -277.2
	-3.9	-1.32	-2.8	-17.5	+1.4	-8.45		-253.0	-16.3	<sup>d</sup> -273.2
Hastelloy-X	<sup>e</sup> -69.6	<sup>b</sup> -432.6	<sup>e</sup> +0.2	<sup>b</sup> -410.1	<sup>e</sup> -7.5	-597.3	-1.2	-306.0	-5.5	<sup>d</sup> -128.0
	<sup>e</sup> -3.2		<sup>e</sup> +.4		<sup>e</sup> -6.2	-715.8		-393.9	-8.9	<sup>d</sup> -336.3

<sup>a</sup>Except as indicated.<sup>b</sup>65 Cycles.<sup>c</sup>36 Cycles.<sup>d</sup>72 Cycles.<sup>e</sup>87 Cycles.

TABLE IV. - X-RAY DIFFRACTION ANALYSIS OF RETAINED OXIDE SCALE OF ALLOYS AND MgO

## CERMETS, WITH RELATIVE PATTERN INTENSITIES

Superalloy powder base	MgO content, vol %									
	0		5		10		20		40	
	Oxidation temperature, °C									
	1100	1200	1100	1200	1100	1200	1100	1200	1100	1200
	Alloy and scale content (relative pattern intensity) <sup>a</sup>									
NiCrAlY	Alloy (vs) $\alpha$ -Al <sub>2</sub> O <sub>3</sub> (m) Spinel <sup>b</sup> (m) 8.05 -----	Alloy (vs) $\alpha$ -Al <sub>2</sub> O <sub>3</sub> (s) Spinel (m) 8.05 NiO (w)	Alloy (vs) $\alpha$ -Al <sub>2</sub> O <sub>3</sub> (m) Spinel (m) -----	Alloy (vs) $\alpha$ -Al <sub>2</sub> O <sub>3</sub> (w) Spinel (m) 8.05 MgO + NiO (w)	Alloy (s) $\alpha$ -Al <sub>2</sub> O <sub>3</sub> (w) Spinel (m) MgO + NiO (m)	Alloy (s) $\alpha$ -Al <sub>2</sub> O <sub>3</sub> (w) Spinel (s) -----	Alloy (m) Spinel (m) -----	Alloy (w) Spinel (m) 8.20 MgO + NiO (vs)	Alloy (w) Spinel (m) -----	Alloy (w) Spinel (m) 8.25 MgO (vs) Cr <sub>2</sub> O <sub>3</sub> (w)
Hoskins-875	Alloy (vs) $\alpha$ -Al <sub>2</sub> O <sub>3</sub> (m)	Alloy (m) $\alpha$ -Al <sub>2</sub> O <sub>3</sub> (vs)	Alloy (vs) $\alpha$ -Al <sub>2</sub> O <sub>3</sub> (w) Spinel (w) 8.10	Alloy (s) $\alpha$ -Al <sub>2</sub> O <sub>3</sub> (m) Spinel (m) 8.05	Alloy (vs) $\alpha$ -Al <sub>2</sub> O <sub>3</sub> (w) Spinel (w) 8.10 MgO-FeO (w)	Alloy (vs) $\alpha$ -Al <sub>2</sub> O <sub>3</sub> (w) Spinel (m) 8.15 MgO-FeO (m)	Alloy (s) $\alpha$ -Al <sub>2</sub> O <sub>3</sub> (vw) Spinel (w) 8.10 MgO-FeO (s)	Alloy (s) $\alpha$ -Al <sub>2</sub> O <sub>3</sub> (t) Spinel (s) 8.10 MgO-FeO (vs)	Alloy (m) Spinel (w) 8.35 MgO-FeO (vs)	Alloy (s) Spinel (s) 8.35 -----
Inconel-702	Alloy (s) Cr <sub>2</sub> O <sub>3</sub> (m) Spinel (m) 8.35 NiO (vw)	Alloy (vs) Cr <sub>2</sub> O <sub>3</sub> (m) Spinel (w) 8.30 NiO (vw)	Alloy (vs) Cr <sub>2</sub> O <sub>3</sub> (vw) Spinel (vw) 8.35 MgO-NiO (s)	Alloy (vs) $\alpha$ -Al <sub>2</sub> O <sub>3</sub> (m) Spinel (m) 8.10 MgO-NiO (vs)	Alloy (s) Cr <sub>2</sub> O <sub>3</sub> (vw) Spinel (m) 8.30 MgO-NiO (vs)	Alloy (vs) $\alpha$ -Al <sub>2</sub> O <sub>3</sub> (w) Spinel (v) 8.10 MgO-NiO (m) Cr <sub>2</sub> O <sub>3</sub> (t)	Alloy (m) Cr <sub>2</sub> O <sub>3</sub> (vw) Spinel (m) 8.30 MgO-NiO (vs)	Alloy (w) Spinel (t) ----- MgO-NiO (s)	Alloy (m) Cr <sub>2</sub> O <sub>3</sub> (vw) Spinel (vw) 8.30 MgO-NiO (vs)	Alloy (s) Spinel (m) 8.35 MgO-NiO (vs)
Hastelloy-X	Cr <sub>2</sub> O <sub>3</sub> (s) Spinel (m) 8.35 NiO (w)	Cr <sub>2</sub> O <sub>3</sub> (vw) Spinel (s) 8.35 NiO (w)	Cr <sub>2</sub> O <sub>3</sub> (m) Spinel (s) 8.35 MgO-NiO (w)	Cr <sub>2</sub> O <sub>3</sub> (vw) Spinel (s) 8.35 MgO-NiO (m)	Cr <sub>2</sub> O <sub>3</sub> (s) Spinel (m) MgO-NiO (w)	Cr <sub>2</sub> O <sub>3</sub> (vw) Spinel (s) 8.35 MgO-NiO (m) NiMoO <sub>4</sub> <sup>c</sup>	Cr <sub>2</sub> O <sub>3</sub> (vw) Spinel (s) 8.30 MgO-NiO (s) NiMoO <sub>4</sub> <sup>c</sup>	Cr <sub>2</sub> O <sub>3</sub> (vw) Spinel (m) 8.30 MgO-NiO (s)	Cr <sub>2</sub> O <sub>3</sub> (vw) Spinel (w) 8.30 MgO-NiO (vs)	Cr <sub>2</sub> O <sub>3</sub> (vw) Spinel (w) 8.30 MgO-NiO (s)

<sup>a</sup>Relative pattern intensities: vs, very strong; s, strong; m, medium; w, weak; vw, very weak; t, trace.<sup>b</sup>Number below denotes estimated lattice parameter of spinel.<sup>c</sup>Unidentified phase.

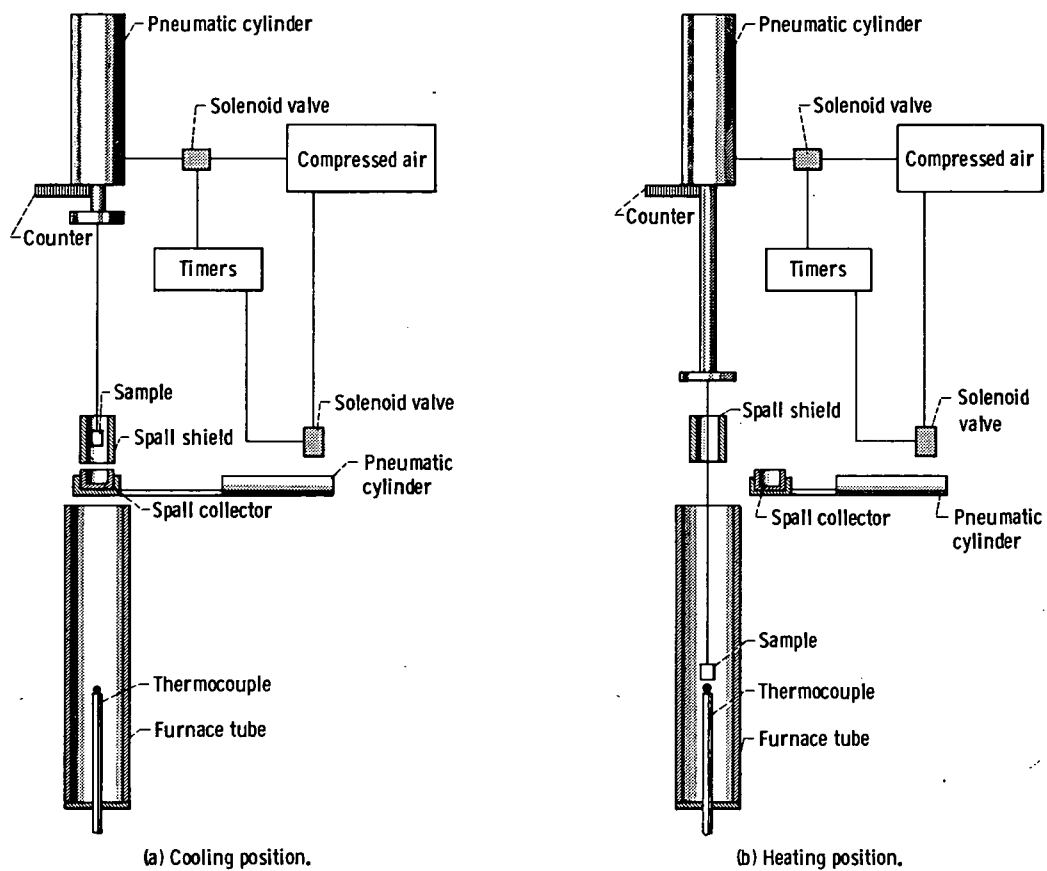


Figure 1. - Schematic drawing of automatic high-temperature cyclic oxidation test apparatus.



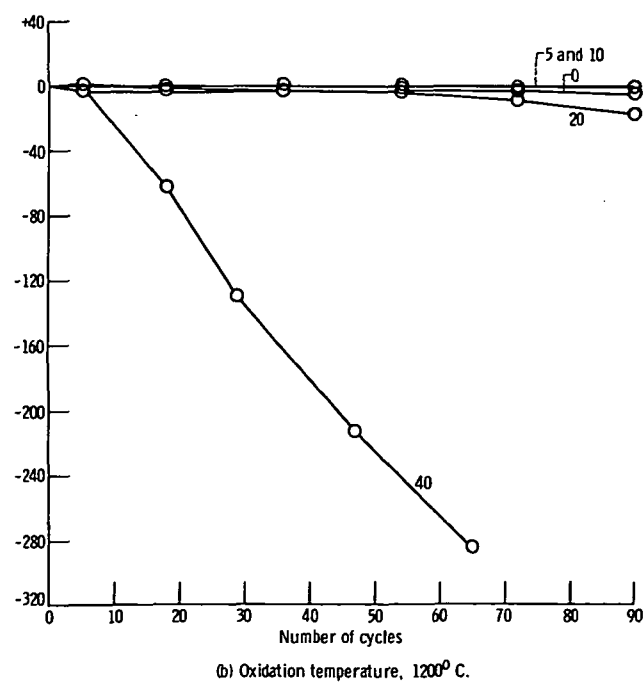
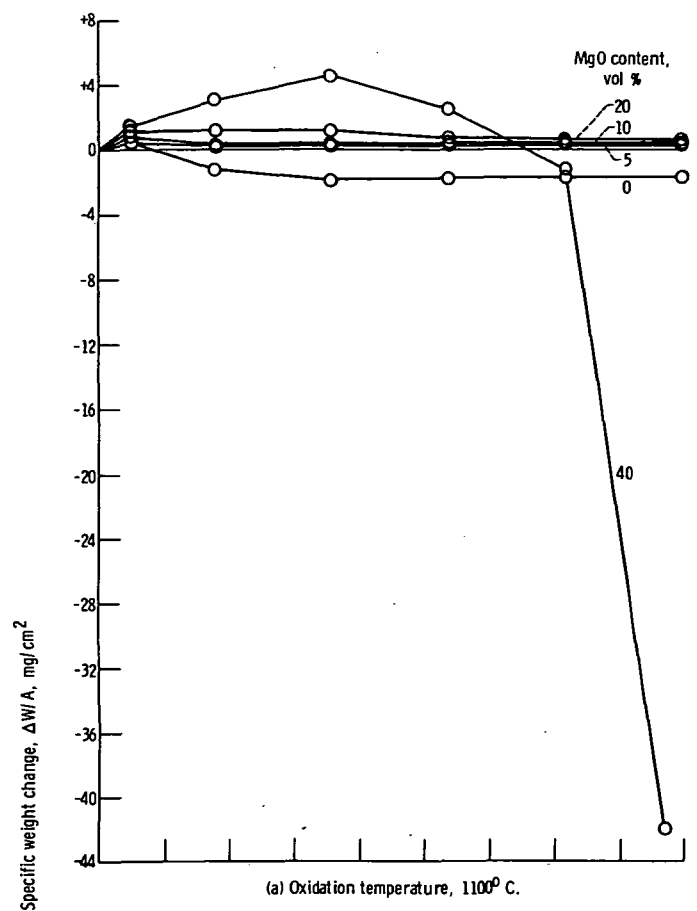


Figure 2 - Sample specific weight change of NiCrAlY alloy and MgO + NiCrAlY cermet as function of number of 1-hour cycles in still air.

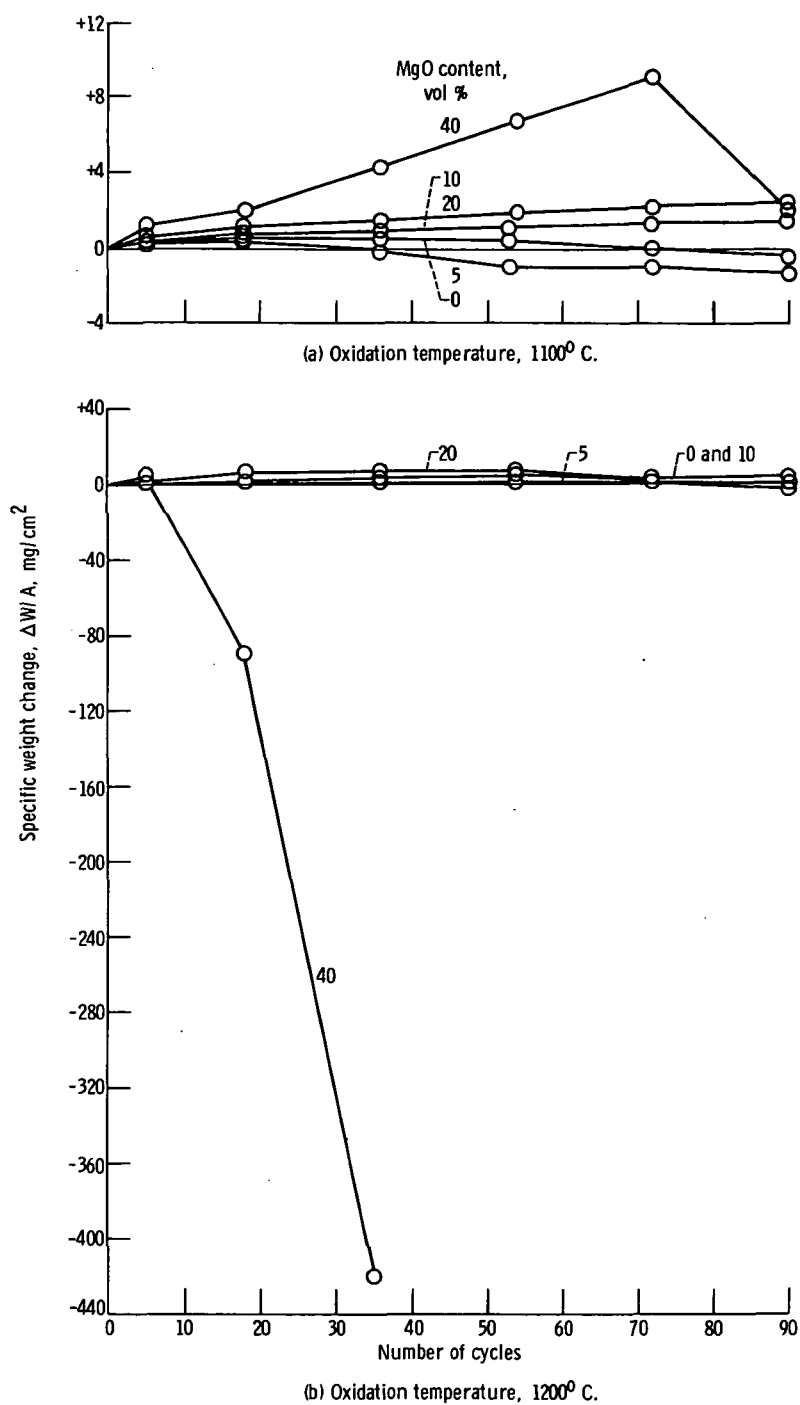
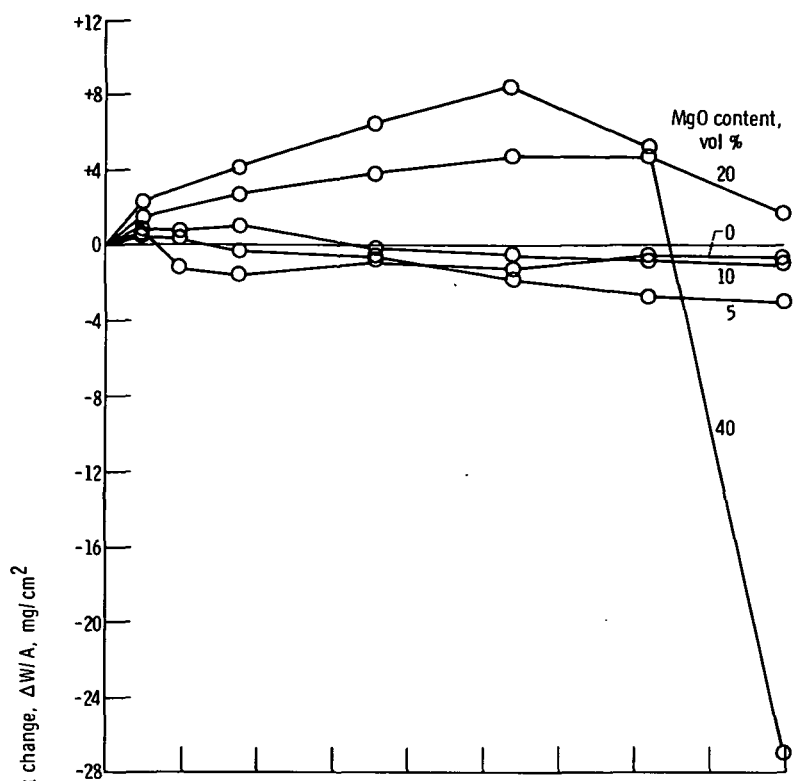
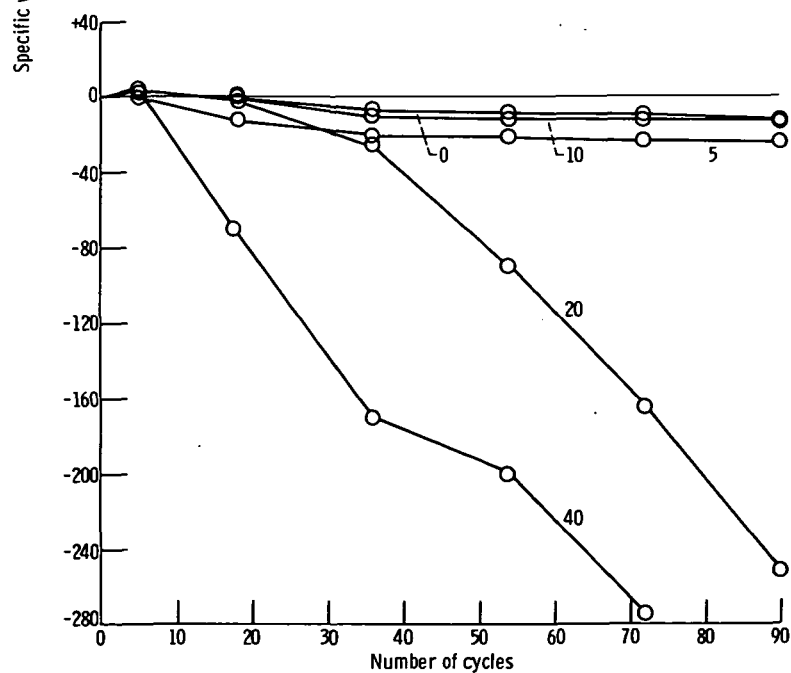


Figure 3. - Sample specific weight change of Hoskins-875 alloy and MgO + Hoskins-875 cermets as function of number of 1-hour cycles in still air.



(a) Oxidation temperature,  $1100^\circ\text{C}$ .



(b) Oxidation temperature,  $1200^\circ\text{C}$ .

Figure 4. - Sample specific weight change of Inconel-702 alloy and MgO + Inconel-702 cermet as function of number of 1-hour cycles in still air.

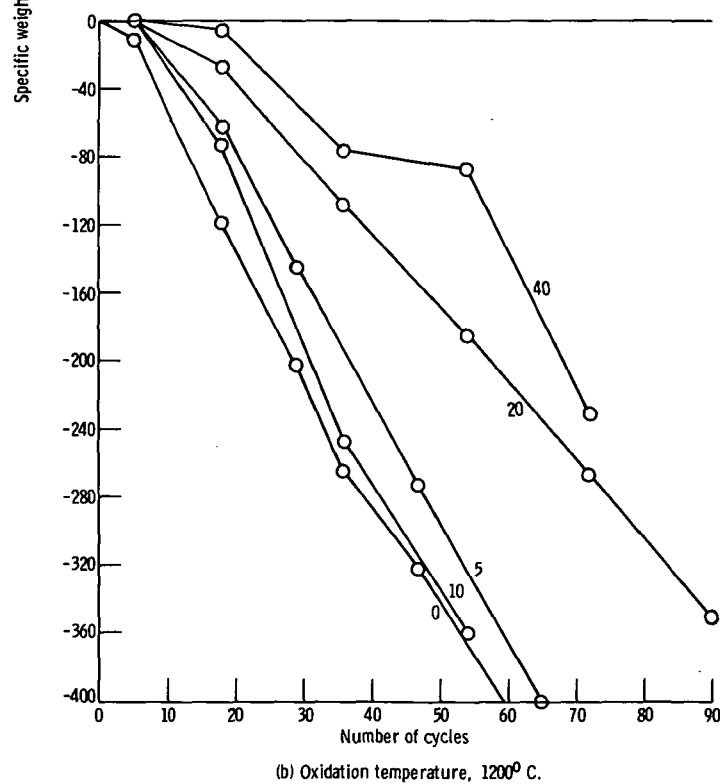
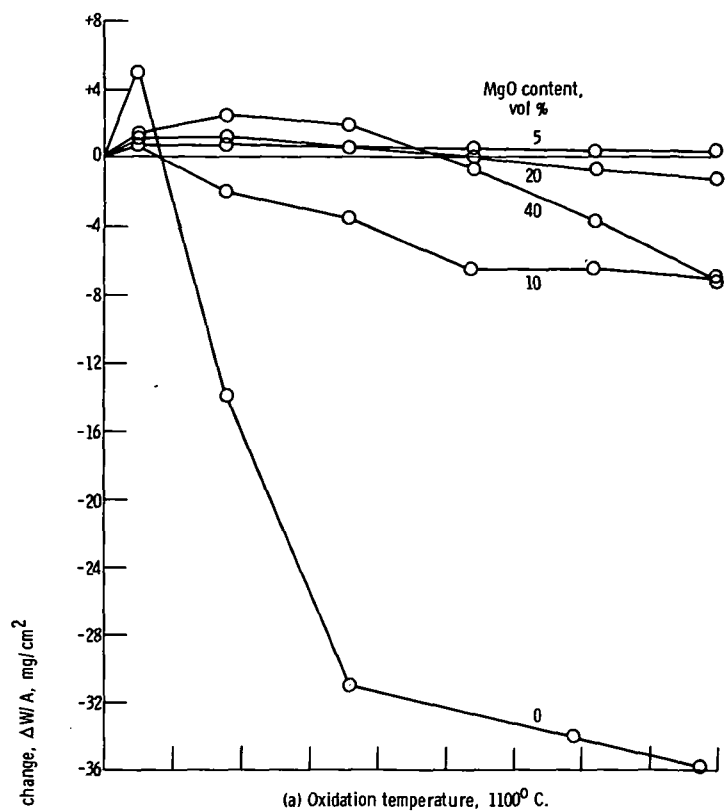
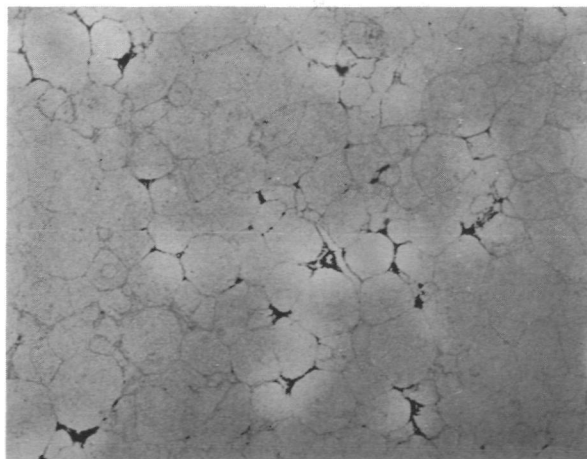
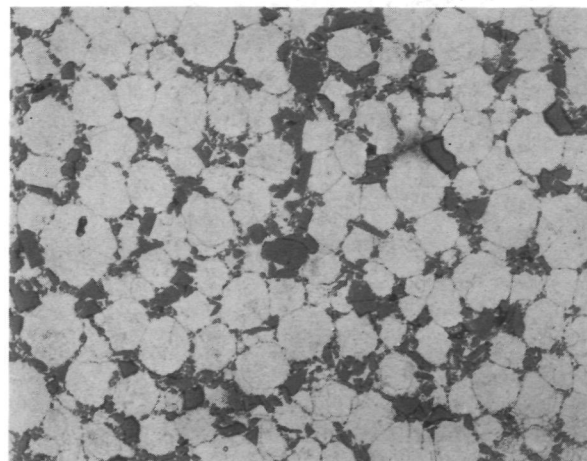


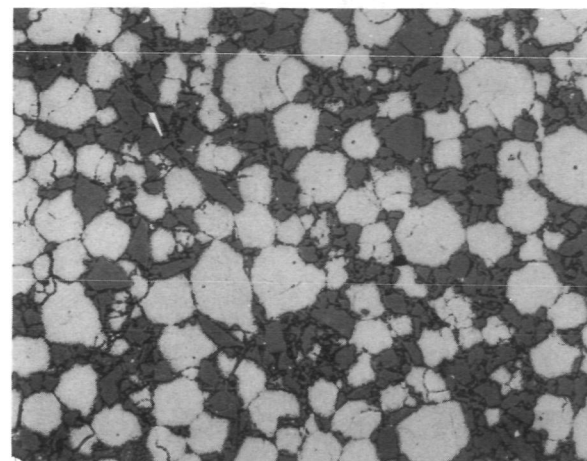
Figure 5. - Sample specific weight change of Hastelloy-X alloy and MgO + Hastelloy-X cermets as function of number of 1-hour cycles in still air.



(a) NiCrAlY as fabricated.

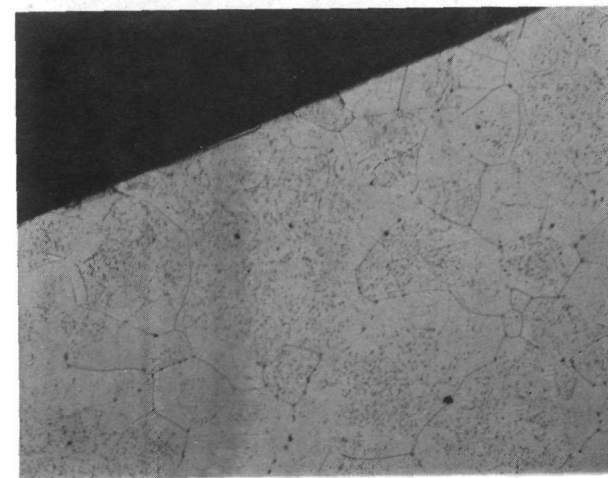


(b) 20-Vol%-MgO + NiCrAlY cermet as fabricated.

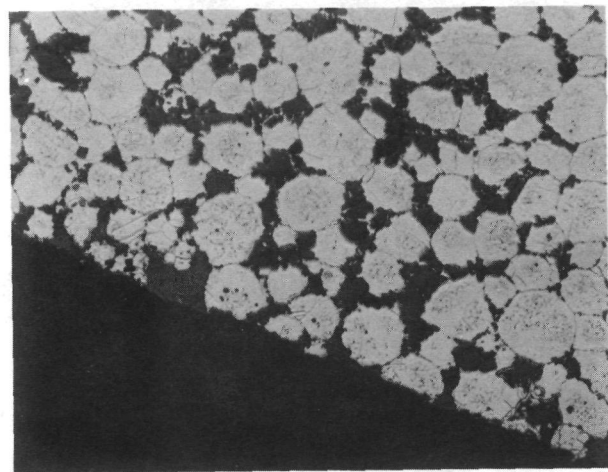


(c) 40-Vol%-MgO + NiCrAlY cermet as fabricated.

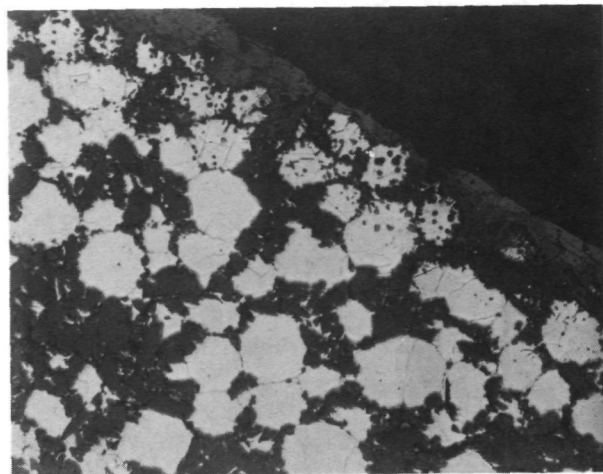
Figure 6. - Microstructure of NiCrAlY and MgO + NiCrAlY cermets as fabricated (isostatically compacted at  $1290^{\circ}\text{C}$  under  $10.5 \times 10^7\text{-N/m}^2$  pressure) and after oxidation at  $1100^{\circ}\text{C}$  and  $1200^{\circ}\text{C}$ . Etched:  $\times 100$ . (Reduced 30 percent in reproducing.)



(d) NiCrAlY oxidized for ninety 1-hour cycles at 1100°C.

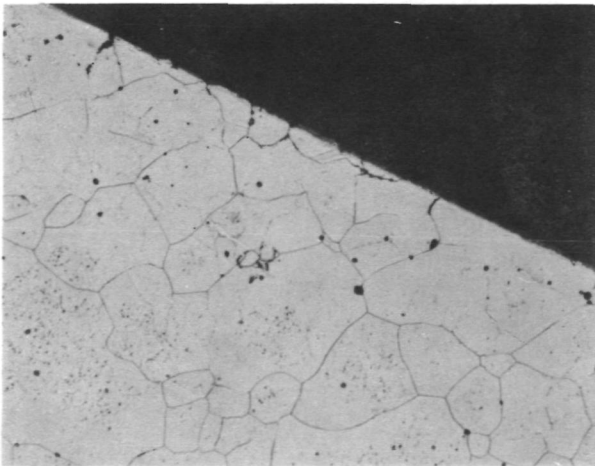


(e) 20-Vol%-MgO + NiCrAlY cermet oxidized for ninety 1-hour cycles at 1100°C.

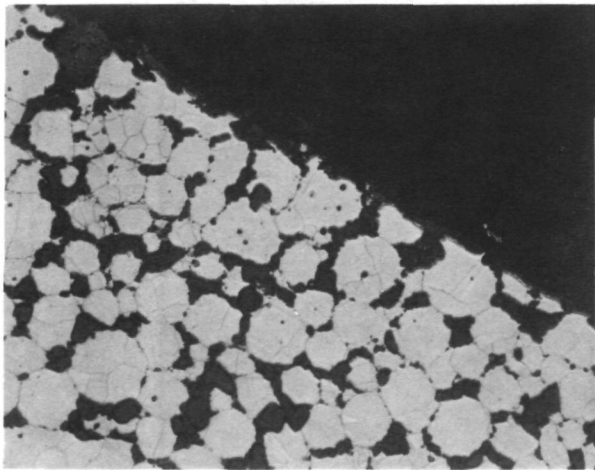


(f) 40-Vol%-MgO + NiCrAlY cermet oxidized for ninety 1-hour cycles at 1100°C.

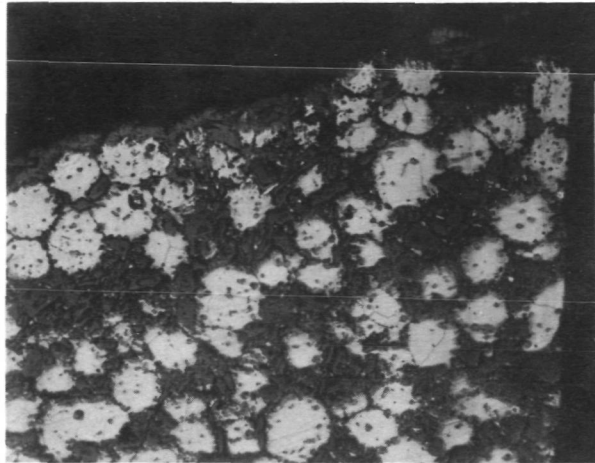
Figure 6. - Continued.



(g) NiCrAlY oxidized for ninety 1-hour cycles at 1200° C.

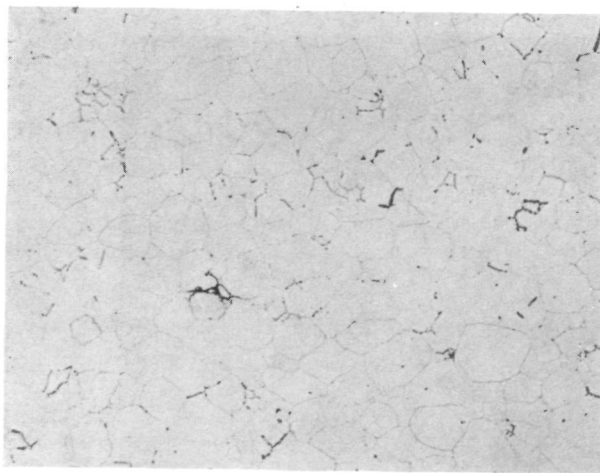


(h) 20-Vol%-MgO + NiCrAlY cermet oxidized for ninety 1-hour cycles at 1200° C.

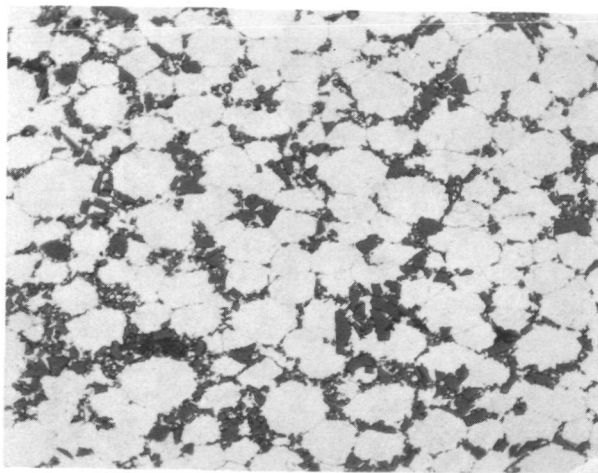


(i) 40-Vol%-MgO + NiCrAlY cermet oxidized for sixty-five 1-hour cycles at 1200° C.

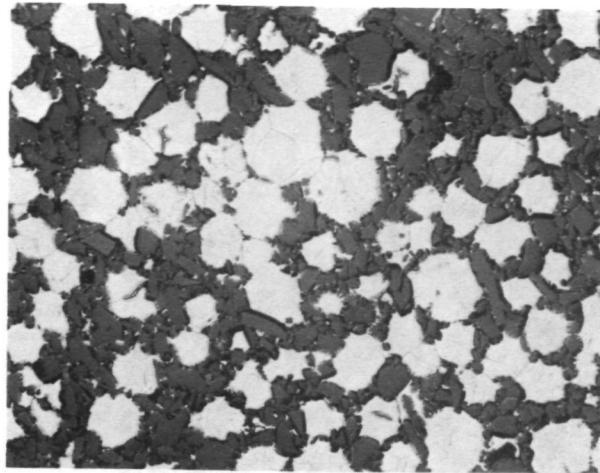
Figure 6. - Concluded.



(a) Hoskins-875 as fabricated.



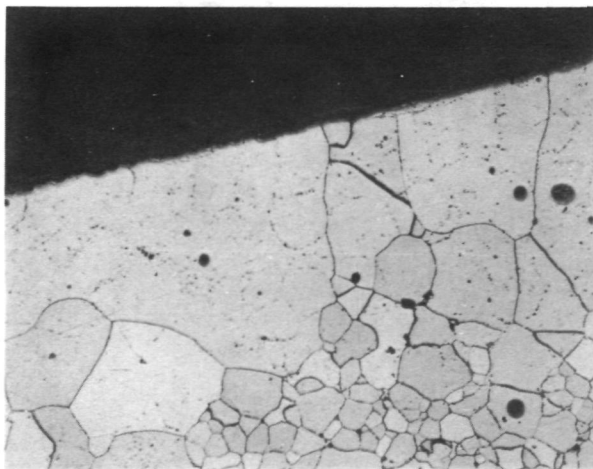
(b) 20-Vol%-MgO + Hoskins-875 cermet as fabricated.



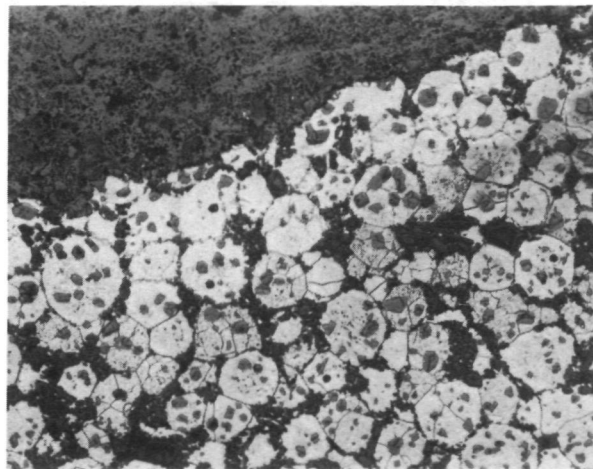
(c) 40-Vol%-MgO + Hoskins-875 cermet as fabricated.

Figure 7. - Microstructure of Hoskins-875 and MgO + Hoskins-875 cermets as fabricated (isostatically compacted at 1290° C under 10.5x10<sup>7</sup>-N/m<sup>2</sup> pressure) and after oxidation at 1100° and 1200° C. Etched; x100. (Reduced 30 percent in reproducing.)

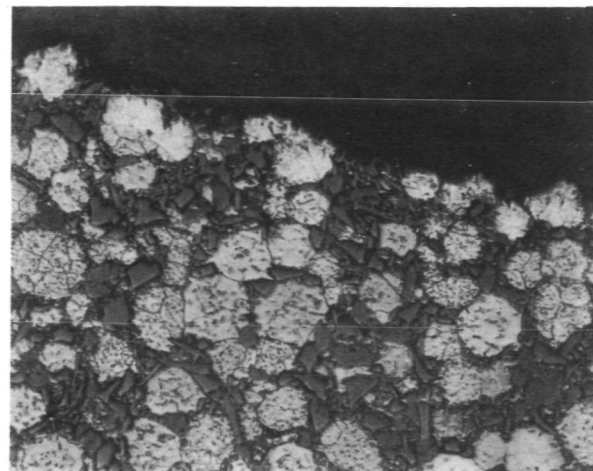




(d) Hoskins-875 oxidized for ninety 1-hour cycles at 1100° C.

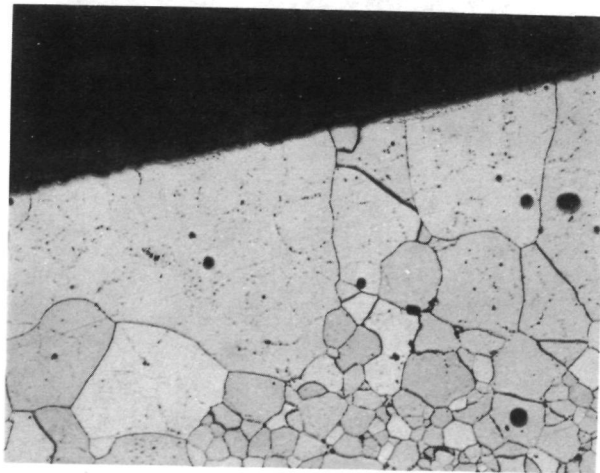


(e) 20-Vol%-MgO + Hoskins-875 cermet oxidized for ninety 1-hour cycles at 1100° C.

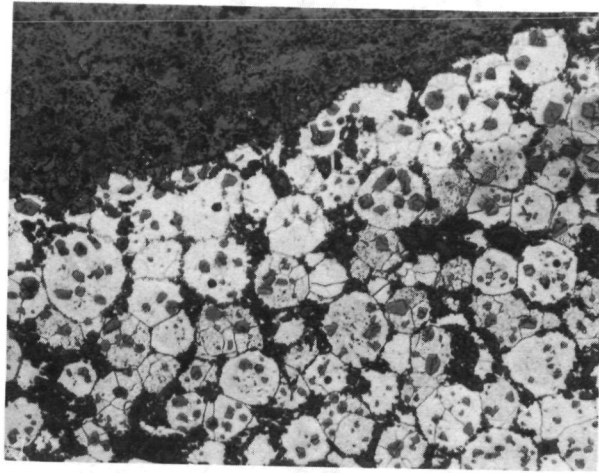


(f) 40-Vol%-MgO + Hoskins-875 cermet oxidized for ninety 1-hour cycles at 1100° C.

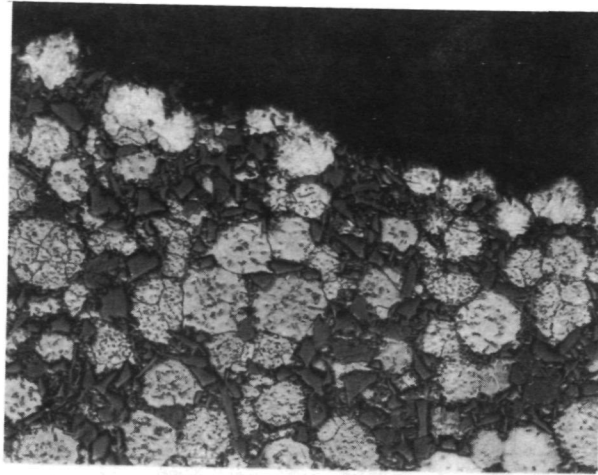
Figure 7. - Continued.



(g) Hoskins-875 oxidized for ninety 1-hour cycles at 1200° C.

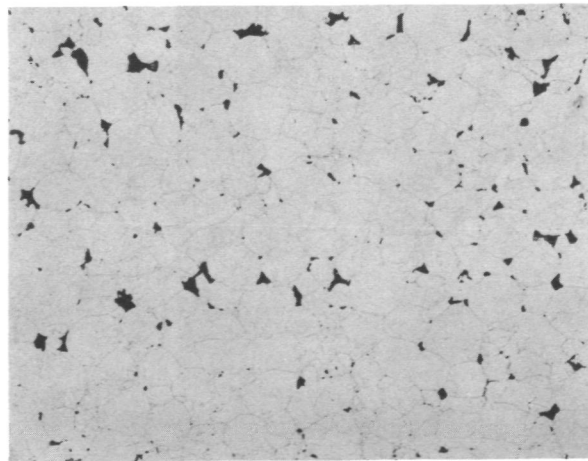


(h) 20-Vol%-MgO + Hoskins-875 cermet oxidized for ninety 1-hour cycles at 1200° C.

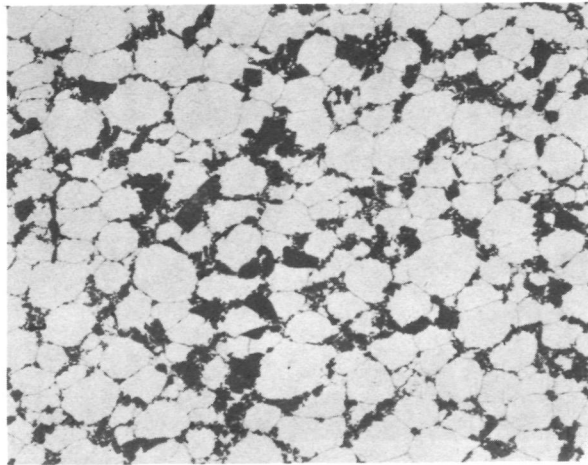


(i) 40-Vol%-MgO + Hoskins-875 cermet oxidized for thirty-six 1-hour cycles at 1200° C.

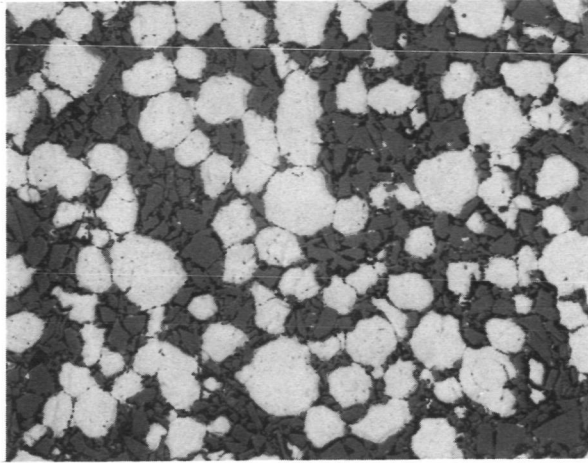
Figure 7. - Concluded.



(a) Inconel-702 as fabricated.

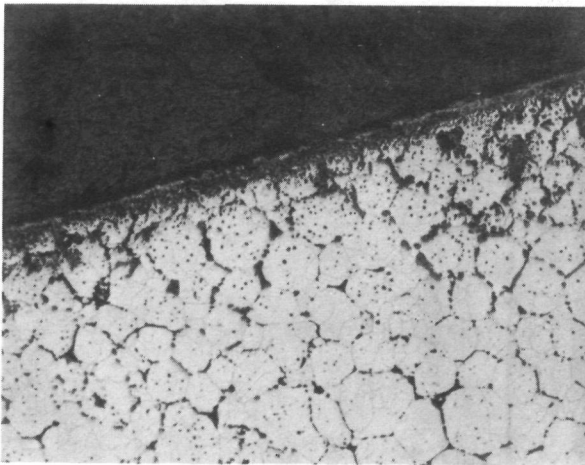


(b) 20-Vol%-MgO + Inconel-702 cermet as fabricated.

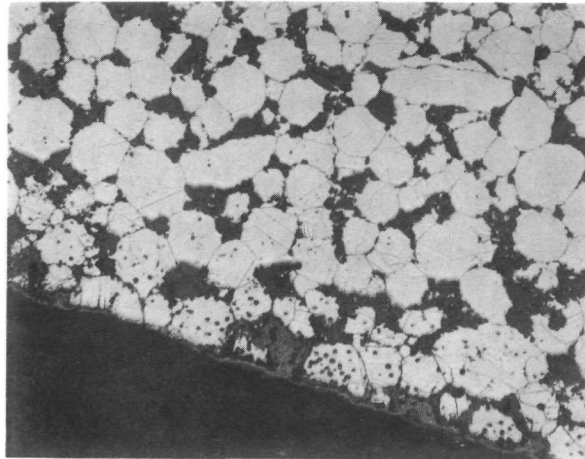


(c) 40-Vol%-MgO + Inconel-702 cermet as fabricated.

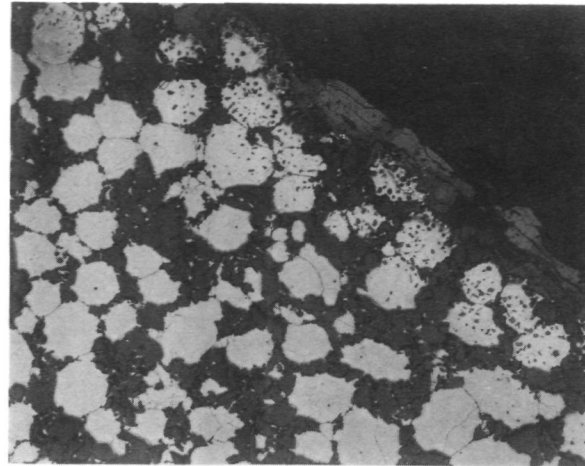
Figure 8. - Microstructure of Inconel-702 and MgO + Inconel-702 cermets as fabricated (isostatically compacted at 1290° C under  $10.5 \times 10^7$  N/m<sup>2</sup> pressure) and after oxidation at 1100° and 1200° C. Etched;  $\times 100$ .  
(Reduced 30 percent in reproducing.)



(d) Inconel-702 oxidized for ninety 1-hour cycles at 1100°C.

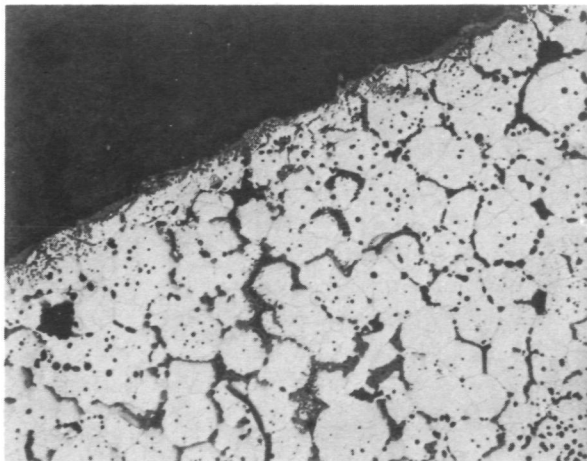


(e) 20-Vol%-MgO + Inconel-702 cermet oxidized for ninety 1-hour cycles at 1100°C.

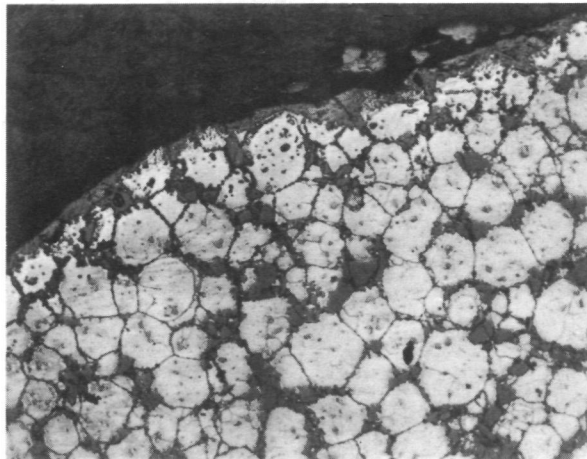


(f) 40-Vol%-MgO + Inconel-702 cermet oxidized for ninety 1-hour cycles at 1100°C.

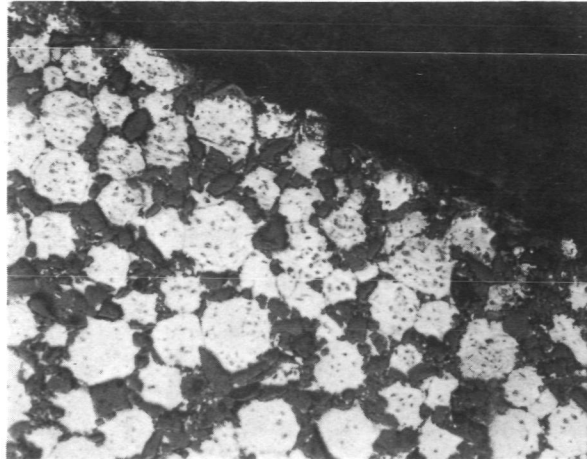
Figure 8. - Continued.



(g) Inconel-702 oxidized for ninety 1-hour cycles at 1200°C.

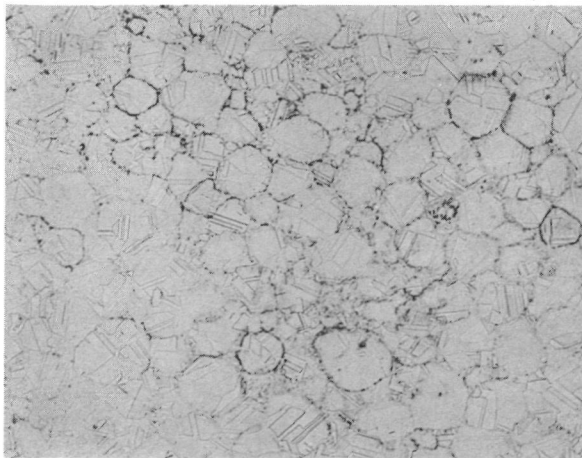


(h) 20-Vol%-MgO + Inconel-702 cermet oxidized for ninety 1-hour cycles at 1200°C.

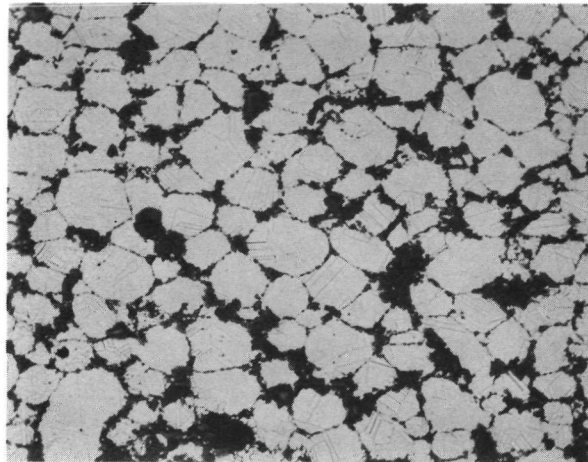


(i) 40-Vol%-MgO + Inconel-702 cermet oxidized for seventy-two 1-hour cycles at 1200°C.

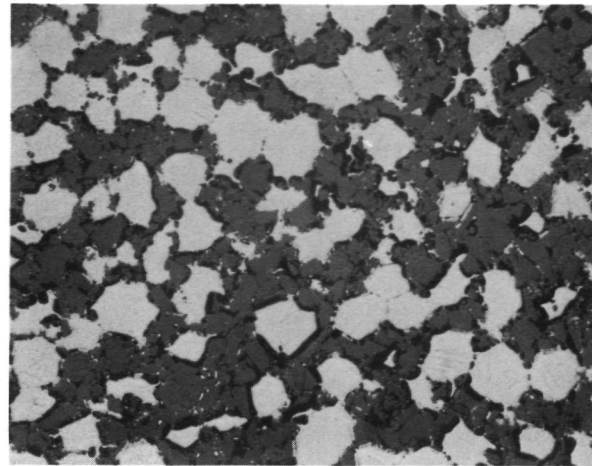
Figure 8. - Concluded.



(a) Hastelloy-X as fabricated.

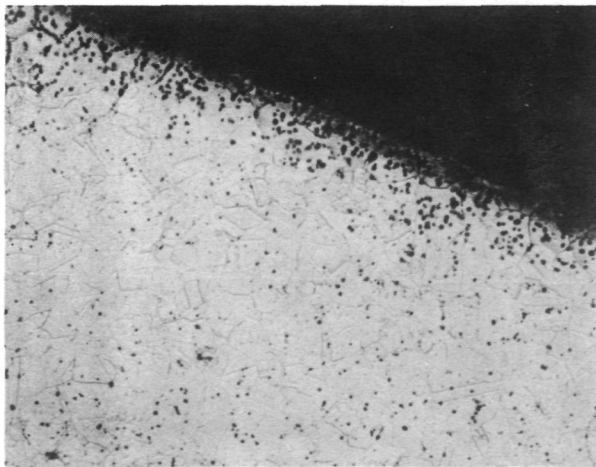


(b) 20-Vol%-MgO + Hastelloy-X cermet as fabricated.

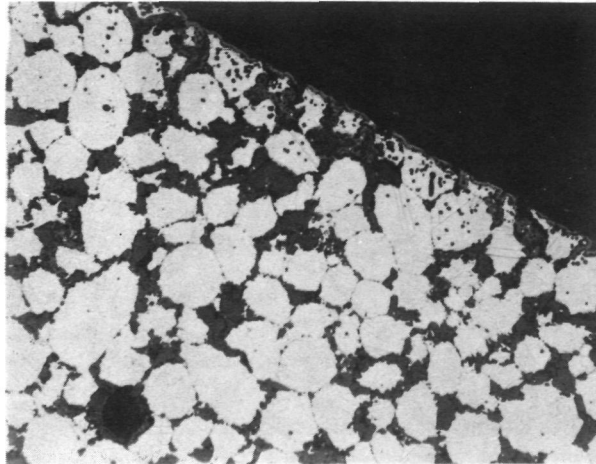


(c) 40-Vol%-MgO + Hastelloy-X cermet as fabricated.

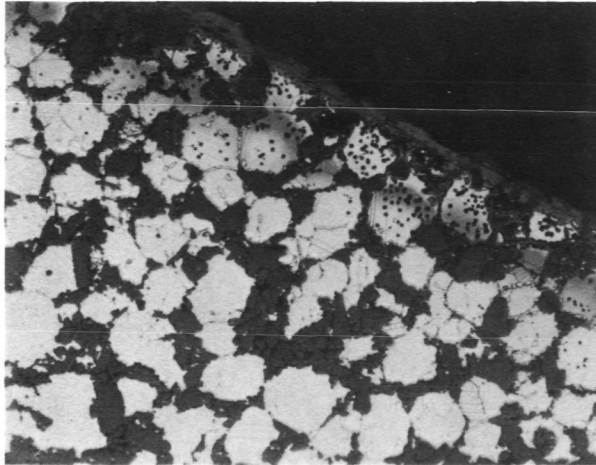
Figure 9. - Microstructure of Hastelloy-X and MgO + Hastelloy-X cermets as fabricated (isostatically compacted at 1290° C under 10.5x10<sup>7</sup>-N/m<sup>2</sup> pressure) and after oxidation at 1100° and 1200° C. Etched; ×100.  
(Reduced 30 percent in reproducing.)



(d) Hastelloy-X oxidized for ninety 1-hour cycles at 1100°C.



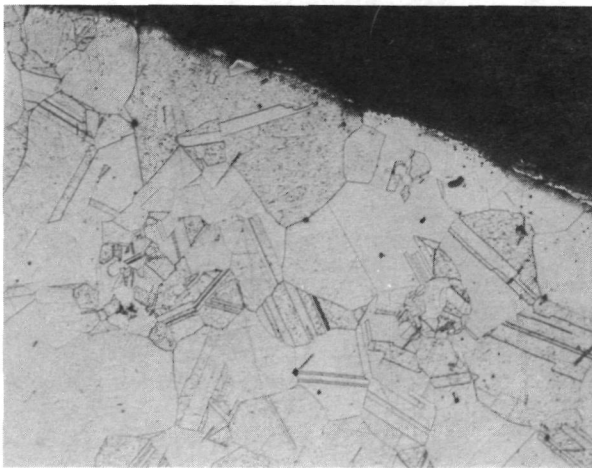
(e) 20-Vol%-MgO + Hastelloy-X cermet oxidized for ninety 1-hour cycles at 1100°C.



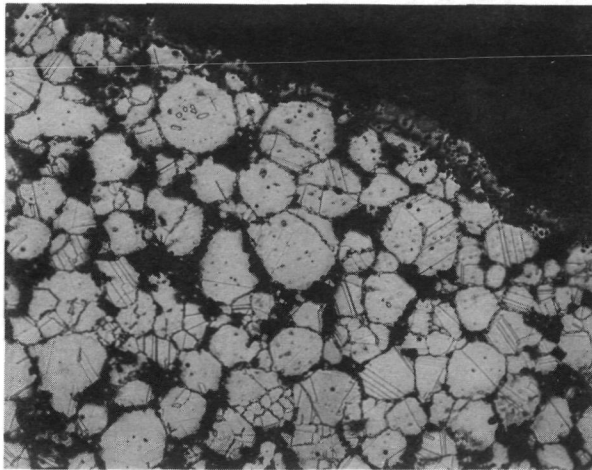
(f) 40-Vol%-MgO + Hastelloy-X cermet oxidized for ninety 1-hour cycles at 1100°C.

Figure 9. - Continued.

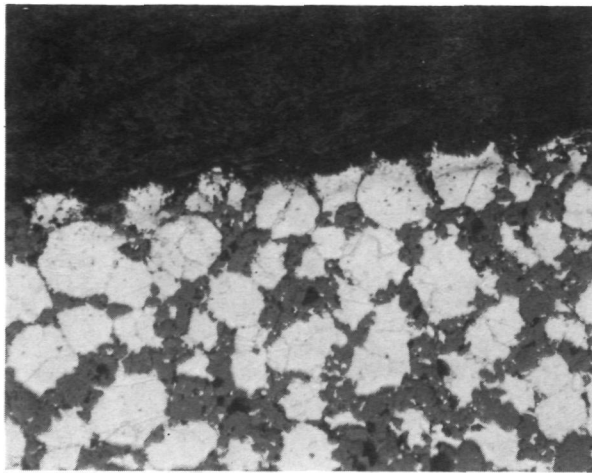




(g) Hastelloy-X oxidized for sixty-five 1-hour cycles at 1200° C.



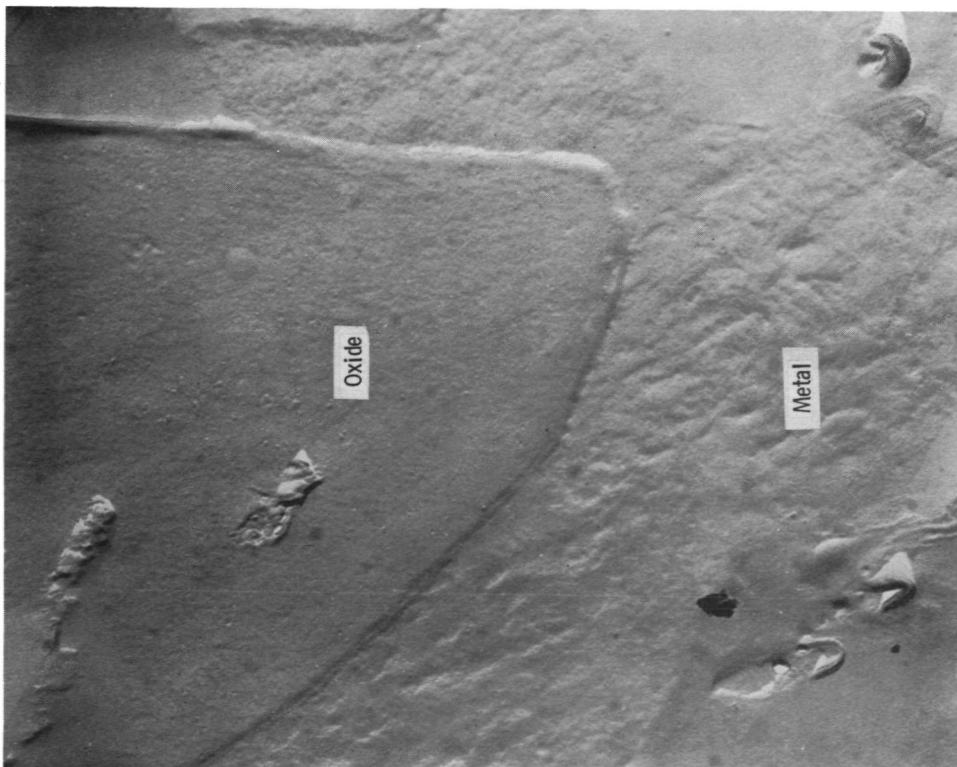
(h) 20-Vol%-MgO + Hastelloy-X cermet oxidized for ninety 1-hour cycles at 1200° C.



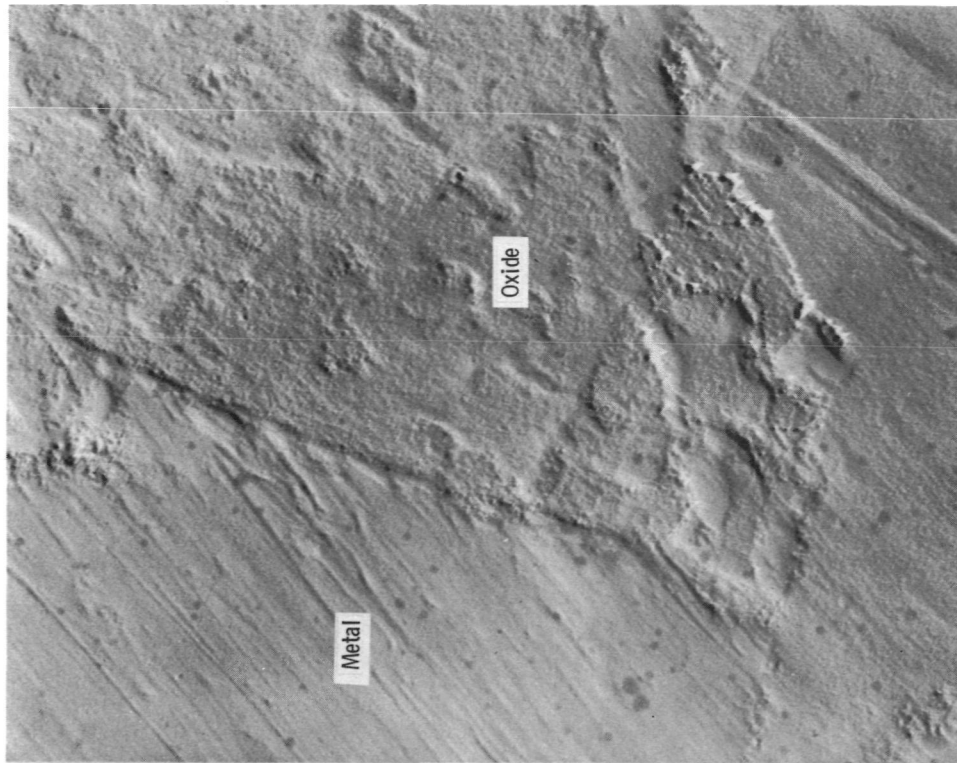
(i) 40-Vol%-MgO + Hastelloy-X cermet oxidized for seventy-two 1-hour cycles at 1200° C.

Figure 9. - Concluded.





(a) 20-Vol % MgO + NiCrAlY.



(b) 20-Vol % MgO + Hoskins-875.

Figure 10. - Electron micrographs of MgO + alloy cermet's after oxidation for ninety 1-hour cycles at 1200° C. Etched; X25 000. (Reduced 45 percent in reproducing.)



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