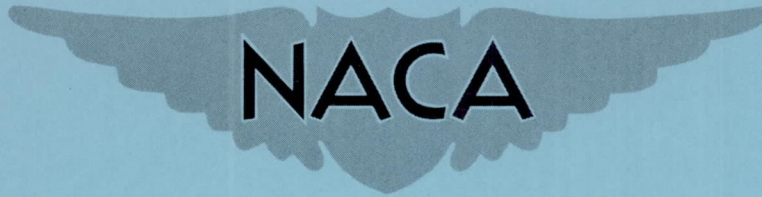


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RESEARCH MEMORANDUM

SOME PROPERTIES OF BERYLLIUM OXIDE AND BERYLLIUM

OXIDE - COLUMBIUM CERAMALS

By C. F. Robards and J. J. Gangler

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RESEARCH MEMORANDUM

SOME PROPERTIES OF BERYLLIUM OXIDE AND BERYLLIUM
OXIDE - COLUMBIUM CERAMALS

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SUMMARY

Because of the potentially excellent refractory properties of beryllium oxide, a brief investigation was made of its short-time tensile strength at a temperature of 1800° F and relative thermal-shock resistance from temperatures of 1800° and 2000° F to room temperature. The effect of additions of 2, 5, 8, 10, 12, and 15 percent by weight of columbium metal on the thermal-shock resistance of beryllium oxide was studied.

Metallographic examination indicated that the metallic phase coalesced into pockets. Beryllium oxide had a tensile strength as high as 6160 pounds per square inch at 1800° F. The original columbium underwent a phase change, as indicated by X-ray analysis of the ceramals. The addition of columbium up to 15 percent by weight failed to improve the resistance to thermal shock. The phase change and the failure of the columbium to wet the beryllium oxide may explain the poor thermal-shock resistance of these ceramals.

INTRODUCTION

Beryllium oxide BeO has the highest thermal-shock resistance of the better-known oxide bodies. In an effort to extend the life of this material in thermal shock, an attempt was made at the NACA Lewis laboratory to use a metallic binder in the manner that has been successful in the carbide-tool industry. The carbide-tool materials generally use a metallic matrix to hold the carbide grains, thus giving the body the mechanical-shock resistance needed without materially sacrificing the hardness or cutting quality of the tool. In a search for a metal to use as a binder element, columbium Cb was selected because of its high melting point.

MATERIALS AND FABRICATION

The fabrication parameters and other fabrication information used in this investigation are listed in the following table.

Ceramic powder.	High-purity BeO
Mesh size	-300
Metal powder, percent Cb.	98.5
Mesh size	-325
Mixing method	Ball mill
Fabrication method.	Hot press
Fabricator.	Commercial
Composition of bodies tested:	
Tensile test specimens.	100 percent BeO
Thermal-shock test specimens.	100 percent BeO and BeO with 2, 5, 8, 10, 12, and 15 percent by weight of Cb

APPARATUS AND PROCEDURE

Tensile experiments. - The tensile experiments were conducted as generally described in reference 1 using the following equipment and procedure:

Equipment used.	Commercial universal tester
Procedure	Soak at $1900^{\circ} \pm 20^{\circ}$ F 4 hr Pull at $1800^{\circ} \pm 10^{\circ}$ F at 400 lb/min to fracture
Specimen alinement.	Bend stress did not exceed 20-percent alinement stress at room temperature

Thermal-shock experiments. - Thermal-shock experiments were made in the NACA thermal-shock unit, in which each specimen was alternately heated and cooled (reference 1) under the following conditions:

Furnace temperature, °F.	^a 1800±10
	2000±20
Time in furnace, min10
Quenching-air temperature, °F.85
Quenching-air velocity, ft/sec	250
Quenching-air mass flow, lb/min.50
Time in air stream, min.	5
Specimen disk dimensions, in.:	
Diameter	2
Thickness	1/4

^aBodies that withstood 25 cycles at 1800° F were then thermal-shock tested from 2000° F until failure.

RESULTS

Tensile investigation. - Two specimens of the 100 percent BeO composition were pulled in tension at 1800° F. The tensile values of 5900 pounds per square inch and 6160 pounds per square inch obtained for the two specimens at this temperature are considered in good agreement. The fractures may be described as rough, which is characteristic of brittle materials when they fracture by pure tensile stresses. Further descriptions of the types of fracture occurring in oxide bodies are presented in reference 2. A typical microstructure of these bodies is shown in figure 1. This photomicrograph shows large equiaxed grains of pure beryllium oxide with the pores that remained after fabrication. It can be noted that the pores are spheroidized and appear at grain boundaries as well as within the grains.

Thermal-shock investigation. - The results of the thermal-shock investigation conducted on the BeO and BeO plus Cb bodies are presented in table I. Included for comparison are some data on other oxide bodies, which have previously been reported in reference 2. The 100 percent BeO bodies showed a decided superiority over the zircon, magnesium oxide, and stabilized zirconia bodies investigated under the same conditions. Addition of Cb to the BeO did not improve the shock resistance, instead it seemed to lower the shock resistance in three of the six compositions subjected to these shock conditions.

1380

Metallurgical and X-ray examination. - A metallurgical check was conducted to determine the reason for failure of the Cb to improve the shock resistance of the BeO. A study of the microstructures showed that the metallic phase failed to coat the BeO grains and thus to form a more thermal-shock resistant body. The photomicrograph (fig. 2) shows how the metal coalesced into large pockets. This result is not unusual because BeO has long been recognized as one of the more stable refractories for crucibles for liquid metals. Calculations of the internal stresses present when these coalesced pockets exist in nonductile bodies revealed that stresses exceeding the strength of the material are possible.

The metallic phase was no longer pure, body-centered, cubic Cb, as shown by X-ray diffraction patterns of several of the bodies; a new phase had developed during either the fabrication or testing of the materials. The diffraction patterns for the new phase were checked against the known diffraction patterns for columbium pentoxide Cb_2O_5 and columbium monoxide CbO as well as the original Cb. (See fig. 3.) No agreement was obtained. The original lines of BeO remained undisturbed.

Study of the polished and etched specimens of several BeO plus Cb bodies revealed a very small grain size and areas in which no recrystallization of the BeO had occurred. A typical area of the long needle-like grains that are mentioned in reference 3 as being characteristic of the BeO powder as manufactured are shown in figure 4. Recrystallization should eliminate such grains as these and leave only equiaxed grains similar to those shown in figure 1. This lack of recrystallization is indicative of insufficient sintering and may help to explain the inconsistent results in the thermal-shock study.

Density and thermal-expansion measurements. - Density measurements were made on all compositions. These experimental values are plotted in figure 5 in comparison with the calculated values obtained when pure mixtures of the two materials are assumed. The experimental values were somewhat erratic and considerably lower than the calculated values. These deviations, however, may be due to the phase change detected by X-ray diffraction and the lack of optimum fabrication conditions.

Thermal-expansion measurements were made on the 15 percent Cb body with a dilatation interferometer. The expansion coefficients are plotted in figure 6 for comparison with expansion of pure BeO from reference 3. No significant differences are noted in the thermal expansions of the 100 percent BeO and 85 percent BeO plus the

15 percent Cb bodies. Expansion measurements of the 15 percent Cb body were stopped at 500° C (932° F), where a reaction of the body with the interferometer plates occurred.

SUMMARY OF RESULTS

The following results were obtained from the investigation of the short-time tensile strength and thermal-shock resistance of beryllium oxide and beryllium oxide plus columbium ceramals:

1. The metallic phase in the beryllium oxide plus columbium bodies tended to coalesce into pockets rather than to surround the individual grains of beryllium oxide.
2. Beryllium oxide showed superior shock resistance to all other oxide bodies previously tested.
3. Beryllium oxide, which has a tensile strength of at least 6160 pounds per square inch at 1800° F, compared favorably with zirconia, the best oxide body (6750 lb/sq in.) previously studied.
4. The original columbium had undergone a phase change as indicated by X-ray analysis.

CONCLUDING REMARKS

From the investigation of beryllium oxide and beryllium oxide plus columbium ceramals, the following observations were made:

1. The generally accepted belief that the addition of a metal to a ceramic will improve the thermal-shock resistance is not supported by the data on beryllium oxide plus columbium bodies because metal contents up to 15-percent columbium by weight failed to improve the shock resistance of beryllium oxide.
2. The phase change and the failure of the columbium to wet the beryllium oxide may explain the poor thermal-shock resistance of these ceramals.

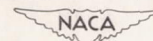
Lewis Flight Propulsion Laboratory,
National Advisory Committee for Aeronautics,
Cleveland.

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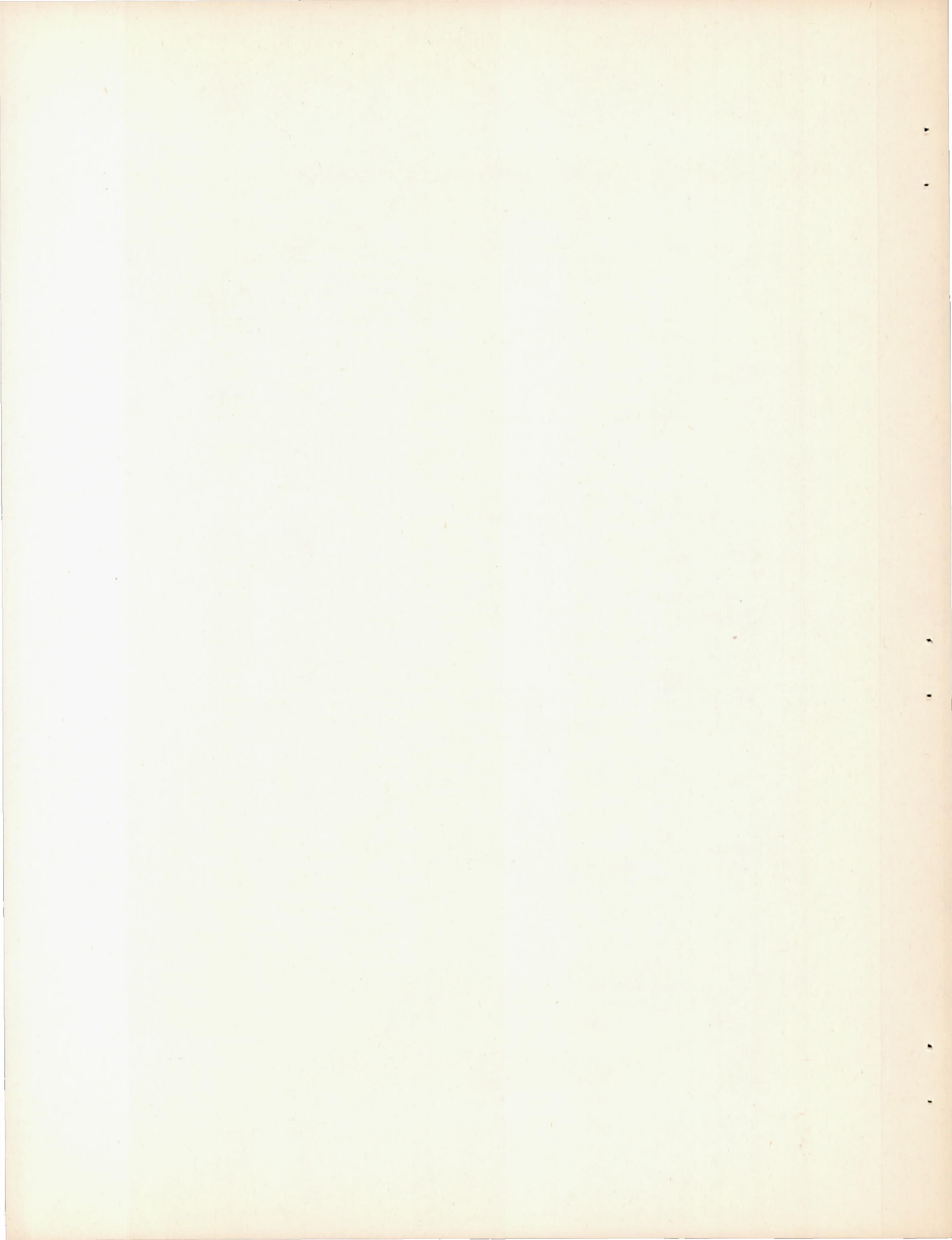
1. Hoffman, Charles A., Ault, G. Mervin, and Gangler, James J.:
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Turbine-Blade Material. NACA TN 1836, 1949.
2. Gangler, James J., Robards, Chester F., and McNutt, James E.:
Physical Properties of Seven Hot-Pressed Ceramics.
NACA TN 1911, 1949.
3. White, H. E., and Shremp, R. M.: Beryllium Oxide: I. Jour.
Am. Ceramic Soc., vol. 22, no. 6, June 1939, pp. 185-189.

TABLE I - RESULTS OF THERMAL-SHOCK EVALUATION

Specimen	Nominal composition (percent)	Number of cycles before failure	
		1800° F	2000° F
1A1	Beryllium oxide, 100	25	3
1B1	Beryllium oxide, 98; columbium, 2	8	
1C1	Beryllium oxide, 95; columbium, 5	16	
1C4	Beryllium oxide, 95; columbium, 5	25	1
1D1	Beryllium oxide, 92; columbium, 8	18	
1D4	Beryllium oxide, 92; columbium, 8	16	
1E1	Beryllium oxide, 90; columbium, 10	20	
1E4	Beryllium oxide, 90; columbium, 10	25	1
1F1	Beryllium oxide, 88; columbium, 12	25	1
1F4	Beryllium oxide, 88; columbium, 12	25	2
1G1	Beryllium oxide, 85; columbium, 15	1	
7A10	Zircon, 100	a ₁	
7A11	Zircon, 100	a _{1/2}	
2A7	Magnesium oxide, 100	a _{1/2}	
8A11	Zirconium oxide, 94; calcium oxide, 6	a ₀	
8A12	Zirconium oxide, 94; calcium oxide, 6	a ₀	



^aData from reference 2.



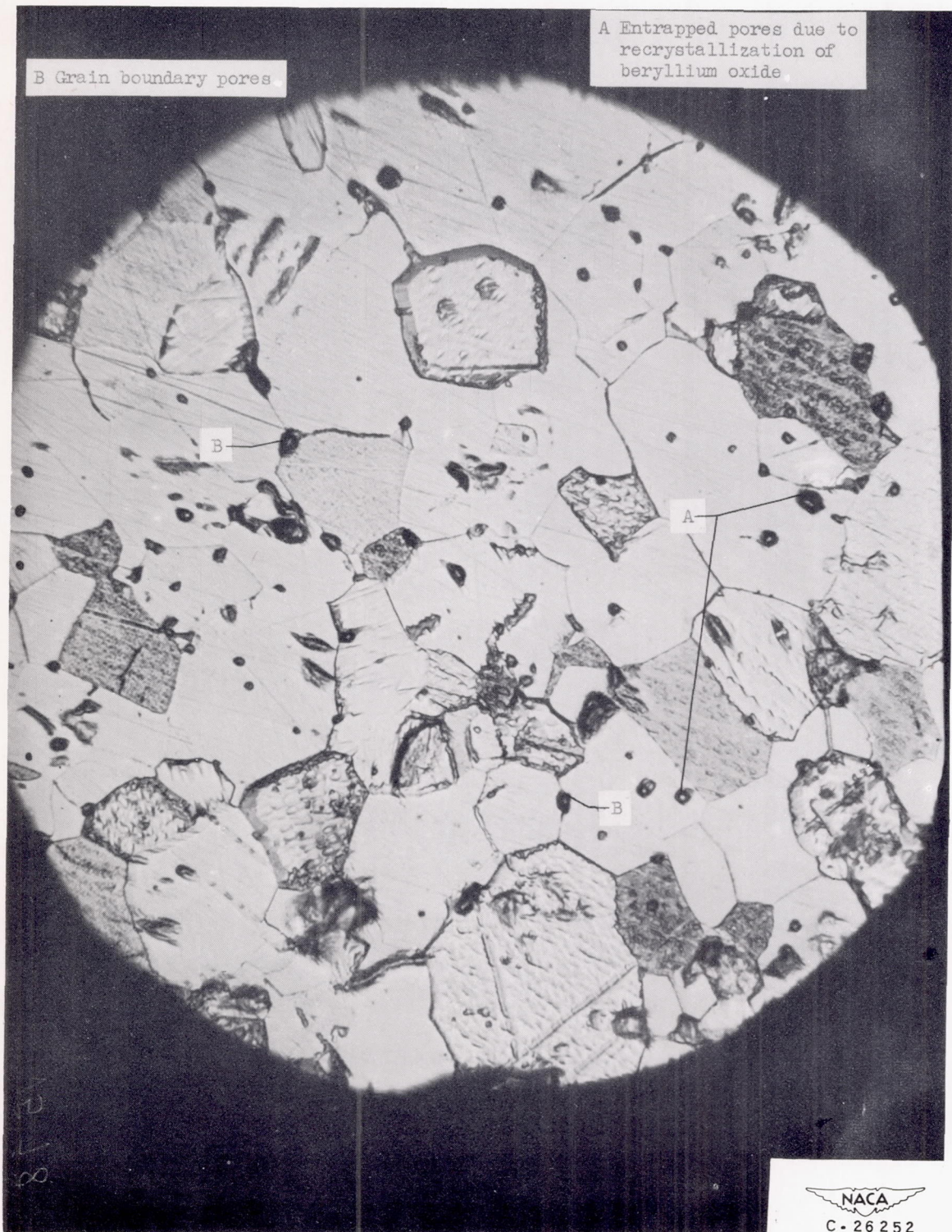


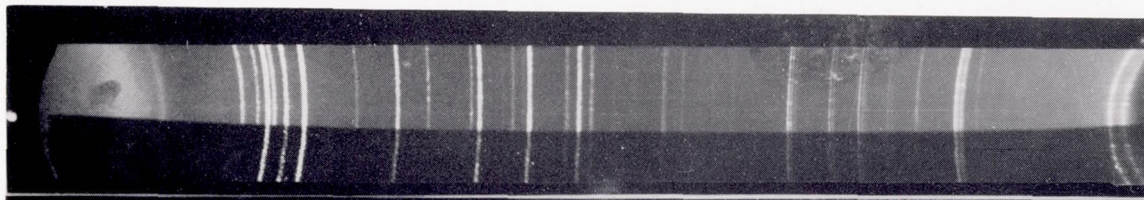
Figure 1. - Photomicrograph of 100-percent beryllium oxide showing preferential etching of grains. X1000.



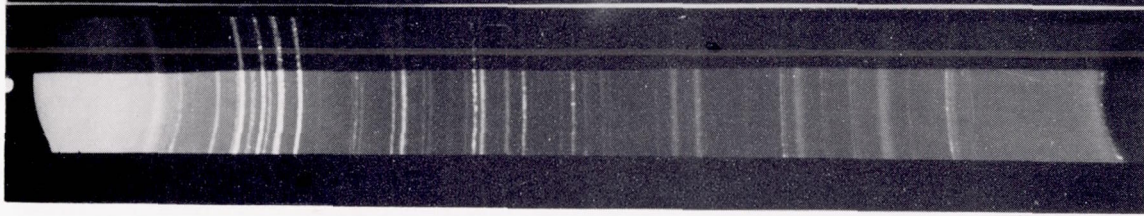


Figure 2. - Photomicrograph of 85-percent beryllium oxide plus 15-percent columbium showing coalesced metallic phase. X250.

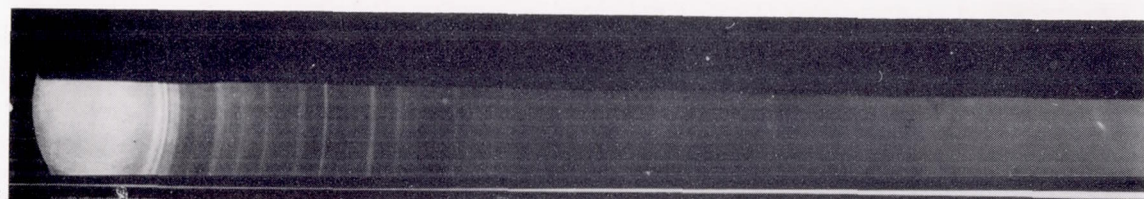




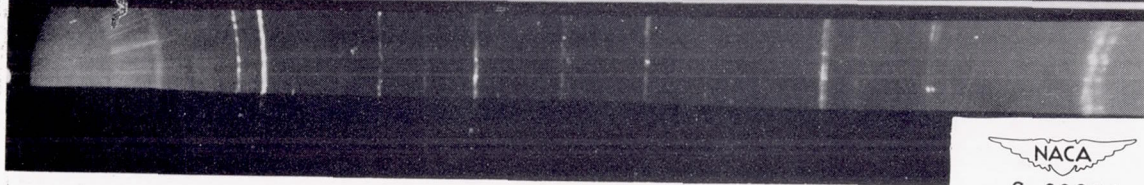
100-percent
beryllium oxide



85-percent
beryllium oxide plus
15-percent columbium



100-percent
columbium pentoxide



100-percent
columbium

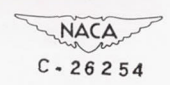
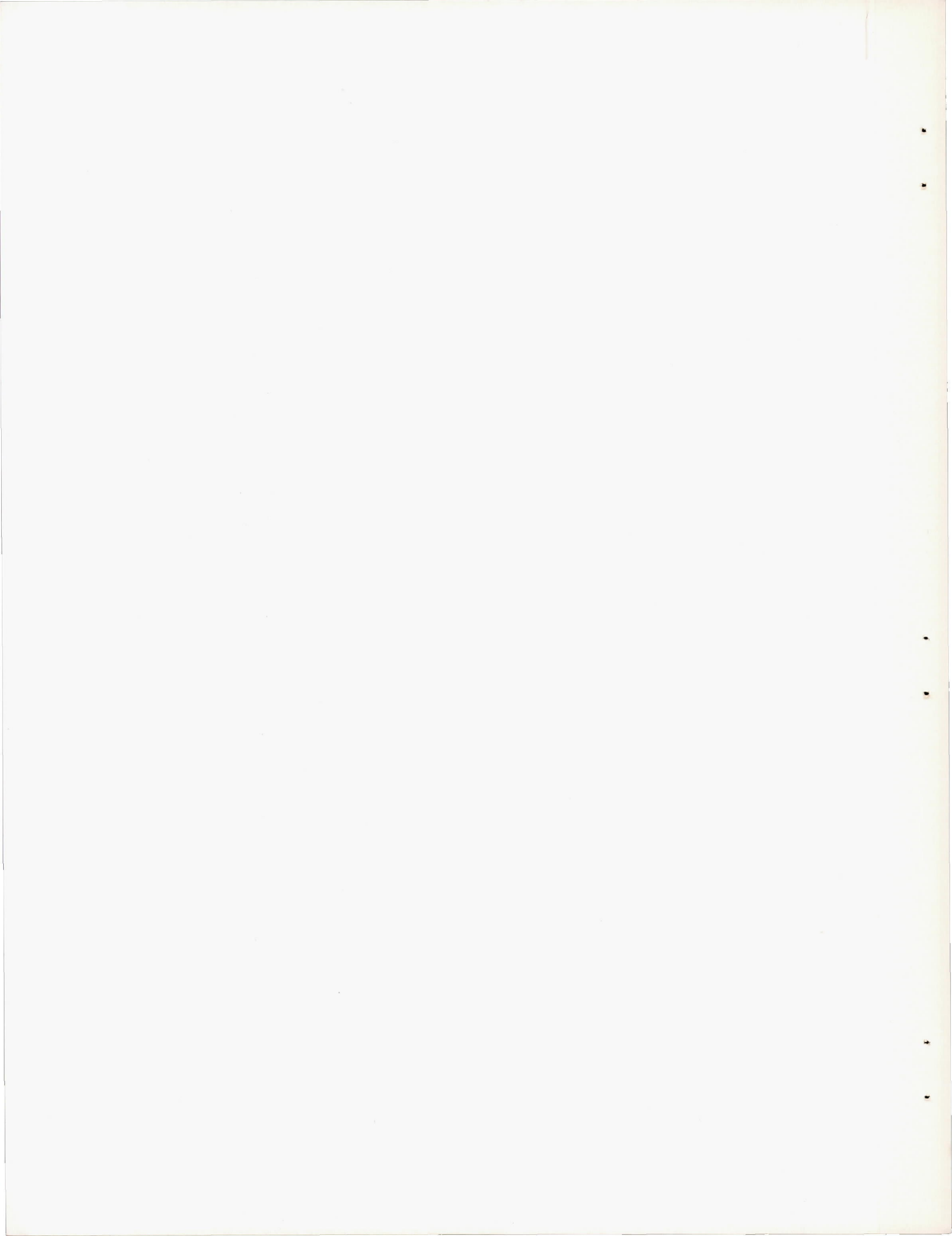


Figure 3. - X-ray diffraction powder patterns; Cu K α radiation.



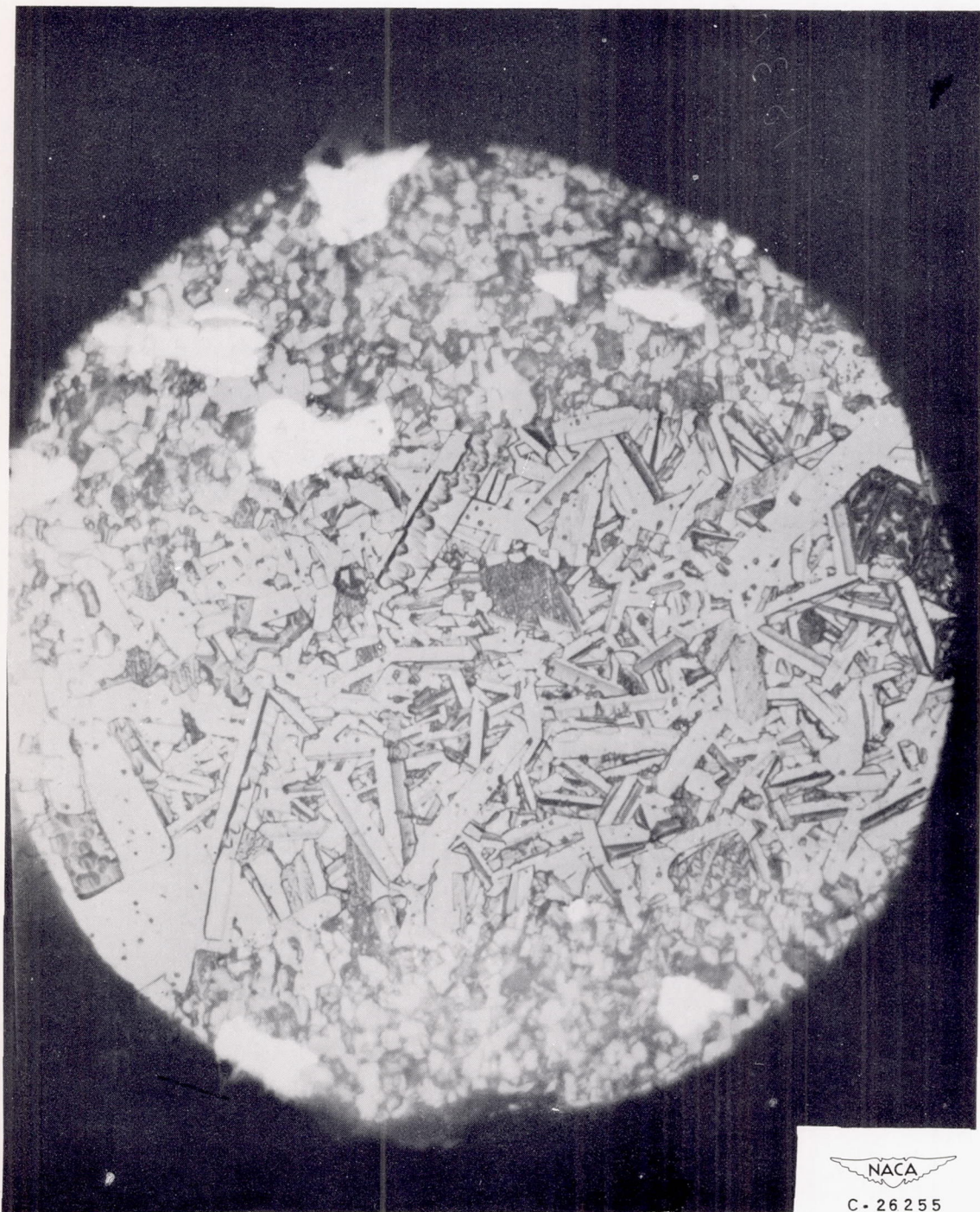
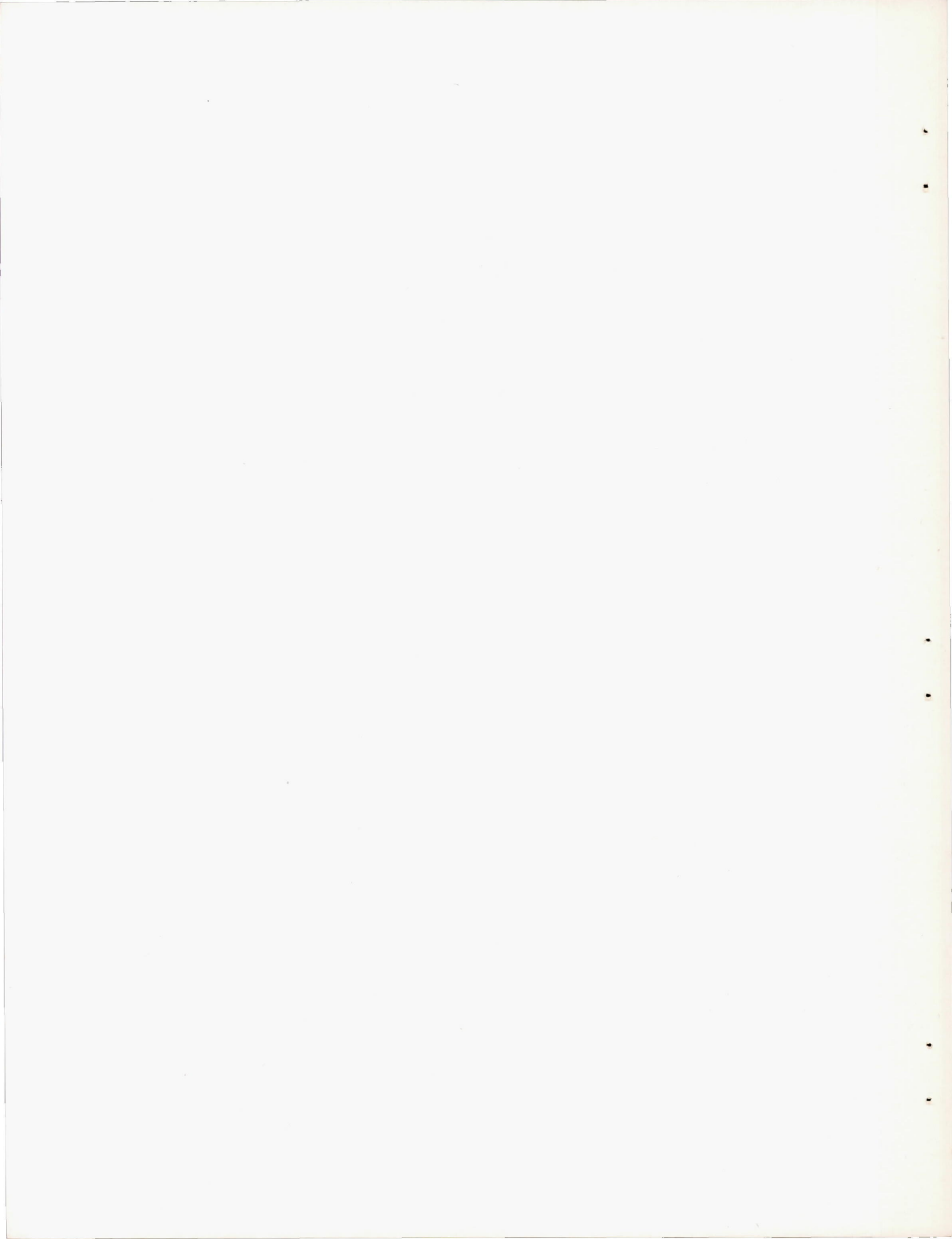


Figure 4. - Photomicrograph of 85-percent beryllium oxide plus 15-percent columbium showing small equiaxed grains and region of unrecrystallized columnar grain. X1000.



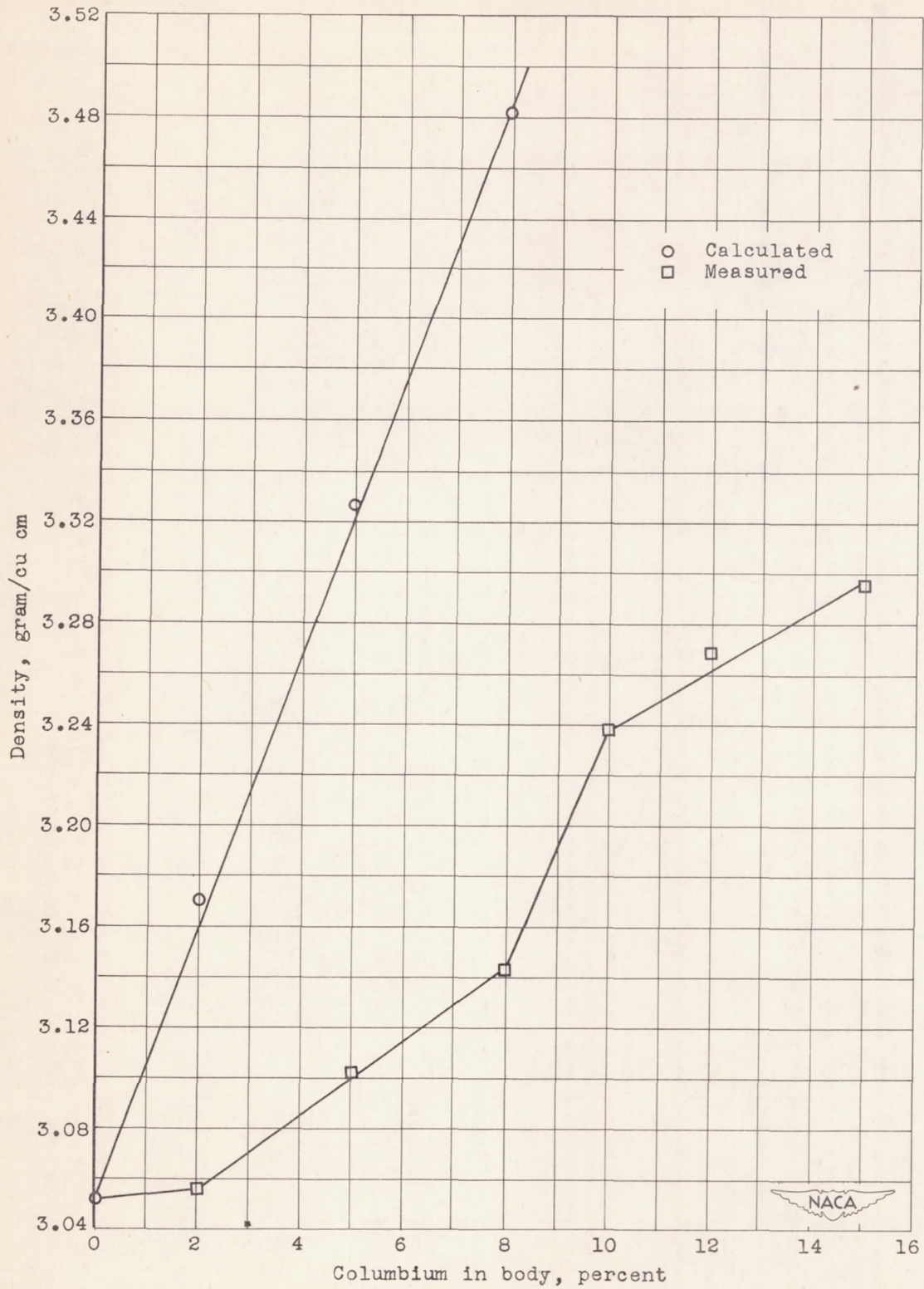


Figure 5. - Variation in density of beryllium oxide with columbium additions.

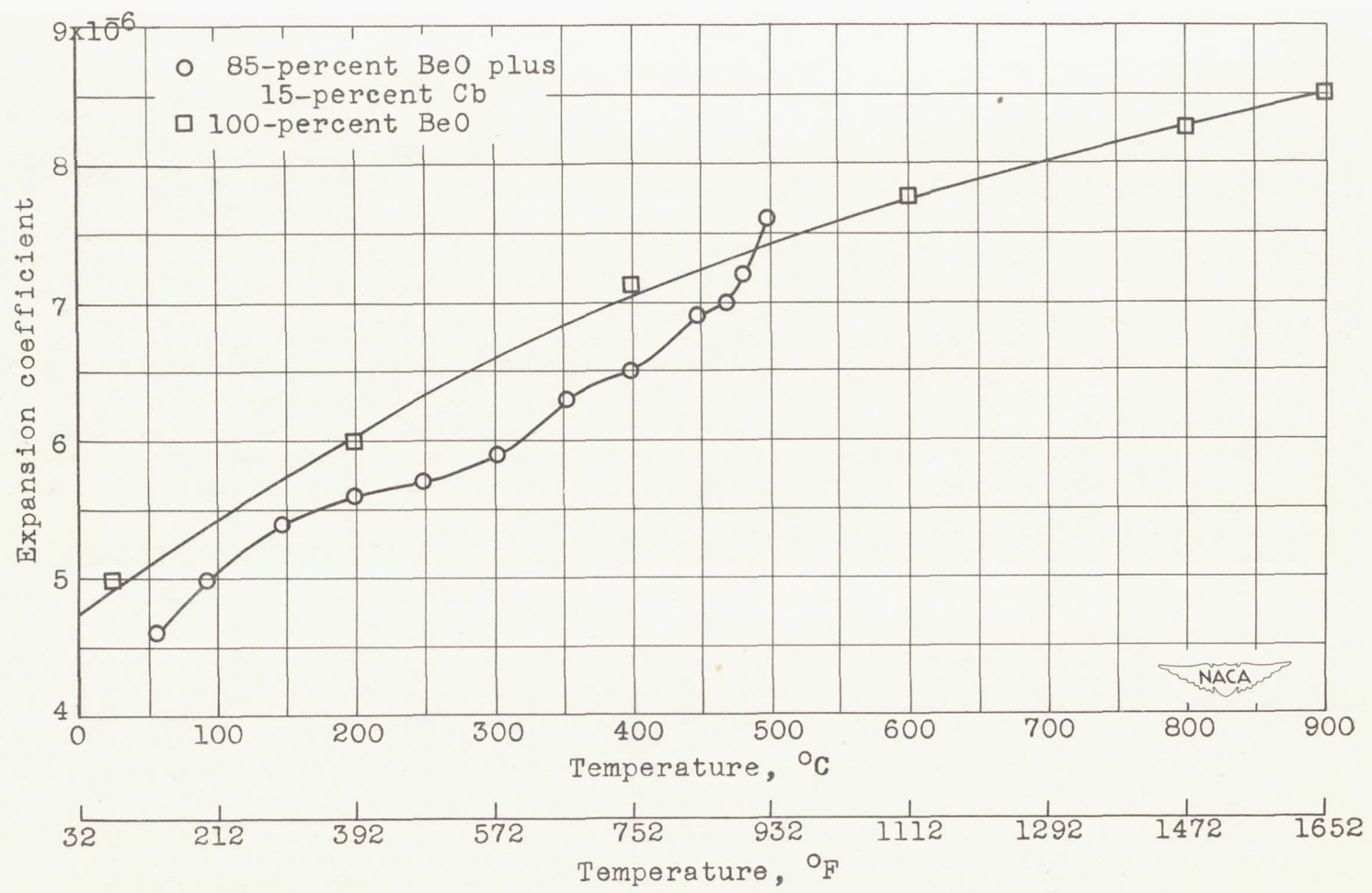


Figure 6. - Thermal-expansion characteristics of beryllium oxide compared with beryllium oxide plus columbium. Data for 100-percent beryllium oxide obtained from reference 3.