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BONDING INVESTIGATION OF TITANIUM CARBIDE WITH VARIOUS ELEMENTS

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SUMMARY

Of 15 elements individually fused on solid high-density titanium carbide at minimum fusion temperatures in a helium atmosphere and at atmospheric pressure, only nickel, cobalt, chromium, and silicon produced bonds with titanium carbide. Metallographic studies of the interface between the element and the titanium carbide in the four bonds indicated that nickel and cobalt appeared to produce extensive interlocking bonds, with nickel showing the greater penetration. The observed interfaces provided information concerning some of the reactions involved in bonding of elements to solid titanium carbide. The 11 elements that did not bond with titanium carbide under the conditions in this experiment were: aluminum, beryllium, columbium, gold, iron, lead, magnesium, manganese, platinum, titanium, and vanadium.

INTRODUCTION

Efforts to increase the power of aircraft turbines have raised a number of power-plant problems. one of which is the need for materials suitable for use at temperatures higher than those currently prevalent. Some materials that show considerable promise for high-temperature application in jet engines have been fabricated from combinations of ceramics and metals (ceramals) by the sintering process. This process affords a means of producing compositions at temperatures below the melting point of the ceramic constituent. Controlled sintering of a powdered ceramal compact is generally recognized as a method capable of producing satisfactory homogeneity and grain size. Investigation of high-temperature properties and of mechanisms for alloying and for bonding of metallic and ceramic materials is necessary for the maximum ulitization of ceramals in aircraft power plants.

An investigation was made at the NACA Lewis laboratory to study the bonding of titanium carbide with various elements. A secondary purpose was to accumulate information regarding the mechanism of bonding. Although the conditions of this investigation were not directly analogous to those prevalent in the sintering of a body of

intimately mixed powdered constituents, the reactions between the powdered elements and the solid titanium carbide in this bonding investigation appeared indicative of those between intimately mixed, discrete particles as in a sintered powder body.

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Titanium carbide was selected as the base material for bonding studies and theoretical evaluations because of its relatively high thermal-shock resistance (reference 1). Cupped, hot-pressed bodies of titanium carbide were obtained from the Norton Company, Chippawa Falls, Canada. Metallographic examinations of sectioned specimens were made in order to determine the type of bond between the titanium carbide and the elements. In those cases where the metal pellet separated from the titanium carbide cupped surface during a careful microspecimen preparation, evaluation of the bonding characteristics was impossible. In each instance that the interface failed in the preparation of the microspecimen, additional specimens were made and observed for confirmation.

MATERIALS

Cupped titanium carbide bodies were fabricated by the Norton Company from material having a particle size of approximately 5 microns. A composite spectrographic chemical analysis of the bodies (based upon separate determinations by two laboratories) is given in table I. The excess carbon, according to the manufacturer, appeared to facilitate compression of the titanium carbide powder. Apparent density measurements of the bodies averaged 4.78 grams per cubic centimeter. The calculated theoretical density of titanium carbide is 4.91 grams per cubic centimeter (reference 1).

Aluminum, beryllium, chromium, cobalt, columbium, gold, iron, lead, magnesium, manganese, nickel, platinum, silicon, titanium, and vanadium were selected for investigation. Elimination of other metallic elements was done on the basis of availability and temperature limitations of equipment. The elements are described in table II.

APPARATUS AND PROCEDURE

A graphite-electric-resistance, muffle-type furnace employing a helium atmosphere was used in fusing the powdered elements in the carbide cups. A schematic diagram of the furnace is shown in figure 1. The 15 elements of the particle sizes shown in table II were fused on cupped, hot-pressed titanium carbide bodies. Impurities introduced in the helium atmosphere were of the type common to controlled atmospheres at atmospheric pressure for sintering furnaces.

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The powdered element was placed in the titanium carbide cup (fig. 2(a)) and was mounded approximately 1/4 inch above the top horizontal surface of the carbide body (fig. 2(b)). A graphite boat, shaped to the contour of the furnace muffle tube, was used to hold the loaded refractory cup. The loaded boat was placed in the first warm (approximately 500° F) zone of the furnace. After 5 minutes in this preheat zone, the boat was pushed into the hot zone. The temperature of the hot zone during each test is given in table II and was generally about 100° F above the melting points given in the International Critical Tables (reference 2). While in the hot zone, the powder mound was observed through a glass-covered observation window. When the powder mound slumped, the loaded boat was immediately placed in the second warm zone for 5 minutes; then the boat was placed in the furnace cold zone for 5 minutes and finally removed from the furnace. The flow rate of helium gas through the furnace was increased from 50 to 250 cubic feet per hour preceding and during any operation involving opening the furnace door while a specimen was in the warm or hot zone.

A diamond circular saw used to section the prepared titanium carbide specimens (fig. 2(c)) left a good surface at the cut, facilitating polishing operations. The specimen was mounted on bakelite and then polished on silk laps with diamond abrasives. A hot aqueous solution of potassium hydroxide - potassium ferricyanide was used as etchant. Scratch-hardness impressions were made with a Bierbaum microcharacter employing a load of 9 grams because a load of 3 grams would not visibly scratch many of the surfaces investigated. Widths of the scratch impressions were optically measured with a calibrated eyepiece and a microscope, resolving at a magnification of 2000. The microhardness number was calculated using the formula

$$k = \frac{10^4}{\left(\frac{\lambda}{2}\right)^2}$$

where

k microhardness number

λ width of scratch, microns

The X-ray diffraction patterns of the residue material at the original titanium carbide - metal interface in the nonadhering specimens were photographed by the Debye-Scherrer-Hull powder method,

using different size cameras, that is, diameters of 57.3 and 143.2 millimeters.

The sole known pattern observed was that of titanium carbide; however, some patterns contained several unidentified lines. The titanium carbide was acquired as a contaminant from the process of accumulating the residue in preparation for the X-ray diffraction photography.

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RESULTS

Preliminary Examination

Nickel, cobalt, chromium, and silicon individually bonded with titanium carbide sufficiently well to permit metallurgical examination. The silicon - titanium carbide specimens were fragile, that is, they were more easily broken in the sectioning operation than those of nickel, cobalt, or chromium with titanium carbide.

Metallographic Examination

The metallographic investigations were based on numerous microscopic observations as well as the reported photomicrographs. A typical example of the internal structure of the original titanium carbide is shown in figure 3. The photomicrographs of titanium carbide matrix with nickel, cobalt, chromium, or silicon are shown in figures 4, 5, 6, and 7, respectively. Approximate location of the original titanium carbide surface is indicated by a horizontal line in each case and is hereinafter termed the "original interface."

Nickel (fig. 4) and cobalt (fig. 5) formed distinct, interconnecting networks around the original titanium carbide grains. Nickel penetrated farther below the original interface than cobalt, as determined by numerous microscopic observations. Although chromium (fig. 6) clearly penetrated the carbide, it did not form a well-defined, interlocking microstructure. Silicon (fig. 7) did not appear to penetrate between the carbide grains. The nickel and cobalt remained as continuous phases down to the titanium carbide particles in the photomicrographs. Solution of titanium carbide was apparently followed by its precipitation as separate particles upon cooling in the nickel-rich as well as in the cobalt-rich zones, as shown by the angular, relatively small particles. With the chromium and the silicon, new phases appeared to have formed adjacent to the titanium carbide phase.

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Hardness Measurements

The average Bierbaum microhardness values of the specimens are presented in table III. In all cases, the titanium carbide-rich zone was harder than the element-rich zone. At the original interface of nickel, cobalt, or chromium with titanium carbide, the hardness values were between those of the element-rich and the titanium carbide-rich zones. Average hardness at the original interface was highest for the cobalt - titanium carbide specimen. Surveys of the actual element hardnesses approaching the interface of the bond indicated that these hardnesses were relatively constant.

DISCUSSION OF RESULTS

Metallographic Examination

The criterion of metallographic evaluation of the element - titanium carbide bond in this investigation was taken as the presence of a mechanical interlocking structure or the formation of a new phase, with no consideration given to the strength of the element. The value of an element as a binder was assumed proportional to the extent of penetration of the matrix by an interconnected phase.

Microstructures of the nickel - and cobalt - titanium carbide specimens indicate that these metals penetrated the titanium carbide along many of the grain boundaries. Silicon, however, did not penetrate between the grains to any appreciable extent. The penetration of chromium appeared to be intermediate. The type of penetration exhibited by nickel and cobalt is similar to one of the types described in reference 3, which states that "if the interphase tension energy is less than one half that of the grain boundary, ... the second phase will penetrate along the boundary indefinitely." The temperatures employed were sufficiently high for equilibrium angles between phases to form near the original interface.

The titanium carbide is soluble (perhaps to a limited extent) in the molten metal. This solubility is further evidenced by the presence of precipitates in the metal-rich zones in the microstructures. The fact that these precipitates are noncontinuous would probably endow a ceramal body of these materials with relatively good thermal-shock resistance.

Both chromium and silicon adhered to titanium carbide after cooling below the melting point. The amount of penetration, however, suggests that neither element surrounds the titanium carbide

particles so thoroughly as nickel and cobalt. Also, the absorption of either carbon or titanium, or both, may have formed continuous insoluble phases in the element-rich zone, which, upon cooling, probably are not so ductile and thus not so resistant to thermal shock as a solid solution containing noncontinuous phases. This effect may have been responsible for the fragility of the bond between silicon and titanium carbide. 1343

Hardness Measurements

The Bierbaum microhardness readings (table III) indicate that the hardness of each element-rich zone was fairly uniform. Larger quantities of titanium carbide were present in the nickel and the cobalt interfaces (compared with those of chromium and silicon), which may account for the higher hardness of these interfaces.

The increase in hardness between the element-rich zone and the original interface indicates a marked difference in either material composition or condition, or both, for specimens of titanium carbide with nickel, cobalt, or chromium. This increase is not true in the case of the silicon specimen. Also, the increment in hardness between the original interface and the titanium carbide zone for all the described bonded specimens shows a change in material composition or condition, or both.

SUMMARY OF RESULTS

Solid titanium carbide was used as the base material in bonding investigations with 15 elements. Of these elements, only nickel, cobalt, chromium, and silicon bonded with titanium carbide; nickel and cobalt produced the most extensive bonds, and chromium and silicon the least extensive bonds. Aluminum, beryllium, columbium, gold, iron, lead, magnesium, manganese, platinum, titanium, and vanadium did not bond with titanium carbide. The appearance of small, angular-shaped precipitates in the nickel-rich and cobalt-rich zones somewhat substantiated the solubility (limited perhaps) of titanium carbide in nickel and cobalt. Employment of nickel, cobalt, and possibly chromium as binder materials for sintered titanium carbide bodies appeared promising.

Lewis Flight Propulsion Laboratory,
National Advisory Committee for Aeronautics,
Cleveland, Ohio, July 20, 1950.

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REFERENCES

1. Gangler, James J., Robards, Chester F., and McNutt, James E.: Physical Properties at Elevated Temperature of Seven Hot-Pressed Ceramics. NACA TN 1911, 1949.

2. Anon.: International Critical Tables. Vol. I. McGraw-Hill Book Co., Inc., 1926, pp. 103-105.

3. Smith, Cyril Stanley: Grains, Phases, and Interfaces: an Interpretation of Microstructure. Metals Tech., vol. 15, no. 4, June 1948, pp. 1-37.

TABLE I - COMPOSITION[a] OF CUPPED

TITANIUM CARBIDE BODIES

Constituent	Weight (percent)
Titanium carbide	[b]96.50 ±0.86
Boron	Trace
Carbon(excess)	1.90 ±0.60
Columbium	.10
Cobalt	.05
Chromium	Trace
Iron	.01
Nickel	.08
Silicon	.30 ±0.20
Titania and/or titanium nitride	[b]1.00
Tungsten	.06 ±0.06

[a]Composite composition based upon reports from two laboratories.
[b]Calculated value.

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TABLE II - POWDERED ELEMENTS

Element	Furnace temperature (hot zone) (°F)	Purity (percent) (a,b)	Particle size (Tyler mesh)	Supplier
Aluminum	1300	99	-100	Charles Hardy, Inc.
Beryllium	2550	Premium grade	-325	The Brush Beryllium Co.
Chromium	3000	99.5	300	Charles Hardy, Inc.
Cobalt	2800	97.5	300	Charles Hardy, Inc.
Columbium	3650	99.8	-400	Charles Hardy, Inc.
Gold	2050	99.9	-100	Charles Hardy, Inc.
Iron	2900	98.5	300	Charles Hardy, Inc.
Lead	700	----------	10	----------
Magnesium	1300	----------	Ribbon	----------
Manganese	2400	99	99.9 percent -325	Metals Disintegrating Co., Inc.
Nickel	2750	99.9	300	Charles Hardy, Inc.
Platinum	3300	99.9	-100	Charles Hardy, Inc.
Silicon	2680	97	-325	Charles Hardy, Inc.
Titanium	3350	98.5	95 percent -325	Metal Hydrides, Inc.
Vanadium	3200	92	20	A. D. Mackay

[a]As reported by suppliers.
[b]Excluding occluded gases.

TABLE III - BIERBAUM MICROHARDNESS VALUES[a]

Specimen	Average hardness numbers		
	Element-rich zone	Original interface	Titanium carbide-rich zone
Nickel - titanium carbide	620	10,900	13,800
Cobalt - titanium carbide	1200	16,400	18,300
Chromium - titanium carbide	760	2,800	16,400
Silicon - titanium carbide	5400	5,800	17,800

[a]Bierbaum microcharacter load of 9 grams.

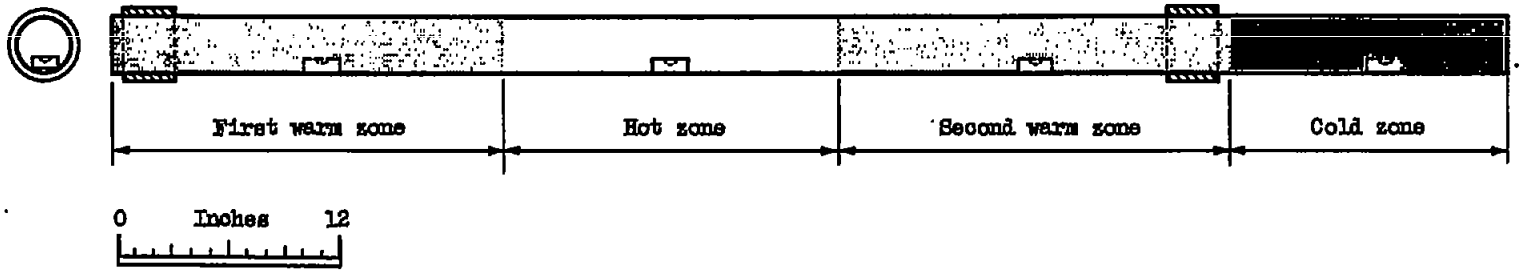
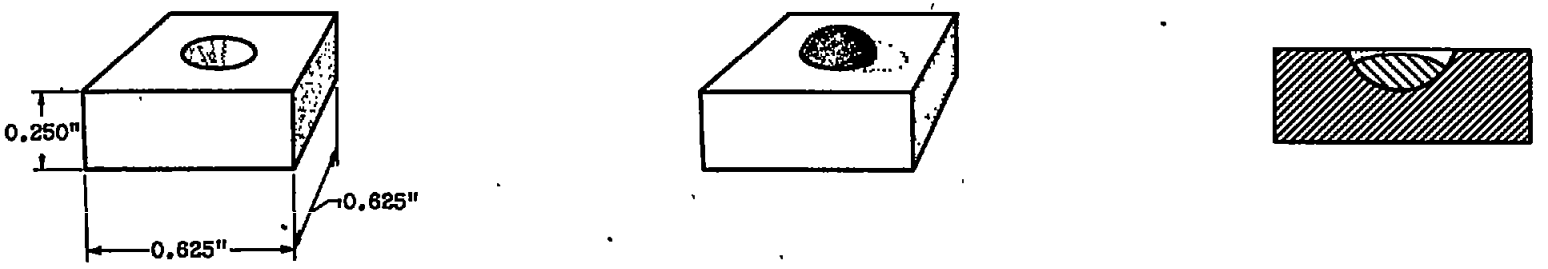


Figure 1. - Schematic diagram of muffle-type furnace used for bonding.



(a) Unused.

(b) Powder in cup prior to fusing.

(c) Sectioned specimen.

Figure 2. - Titanium carbide body.

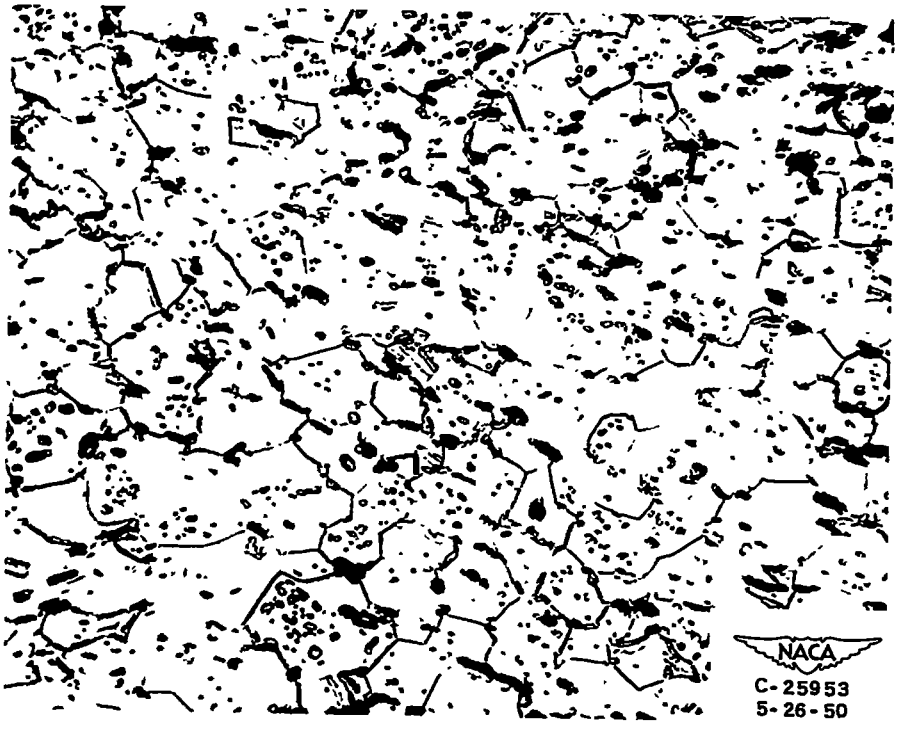


Figure 3. - Titanium carbide. Etchant, potassium hydroxide - potassium ferricyanide. X1000.

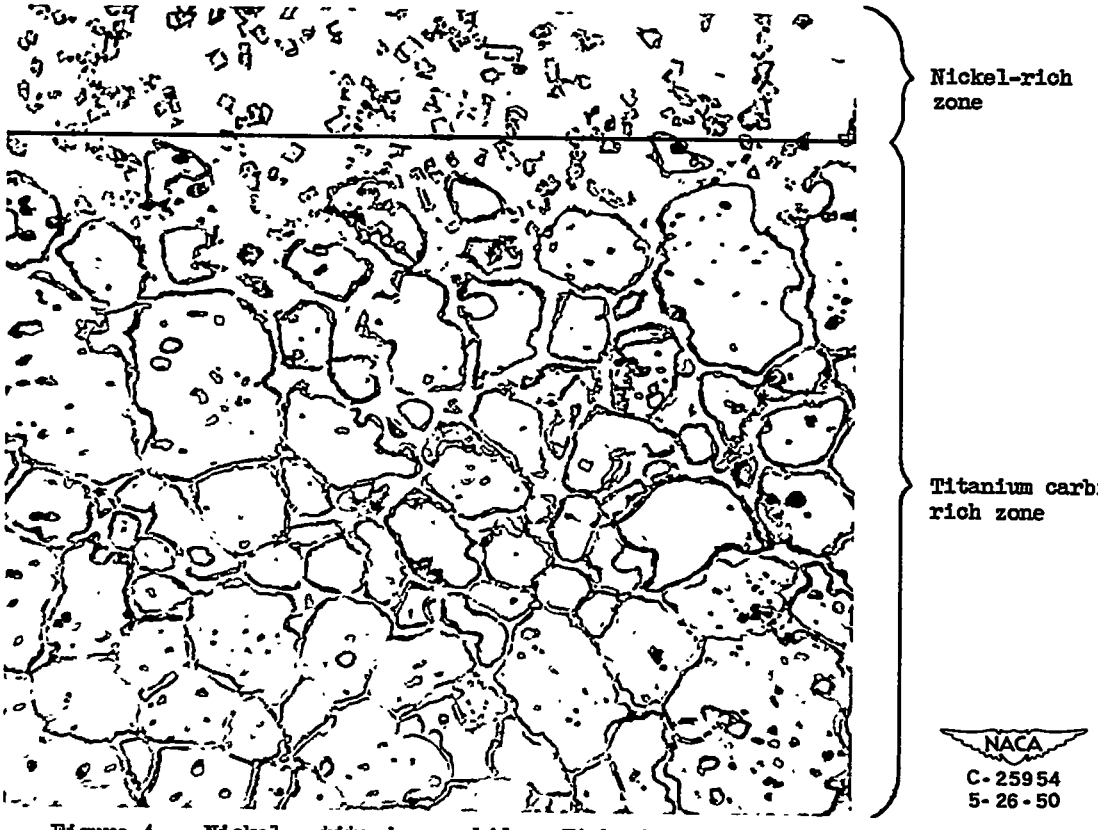


Figure 4. - Nickel - titanium carbide. Etchant, potassium hydroxide - potassium ferricyanide. X1000.

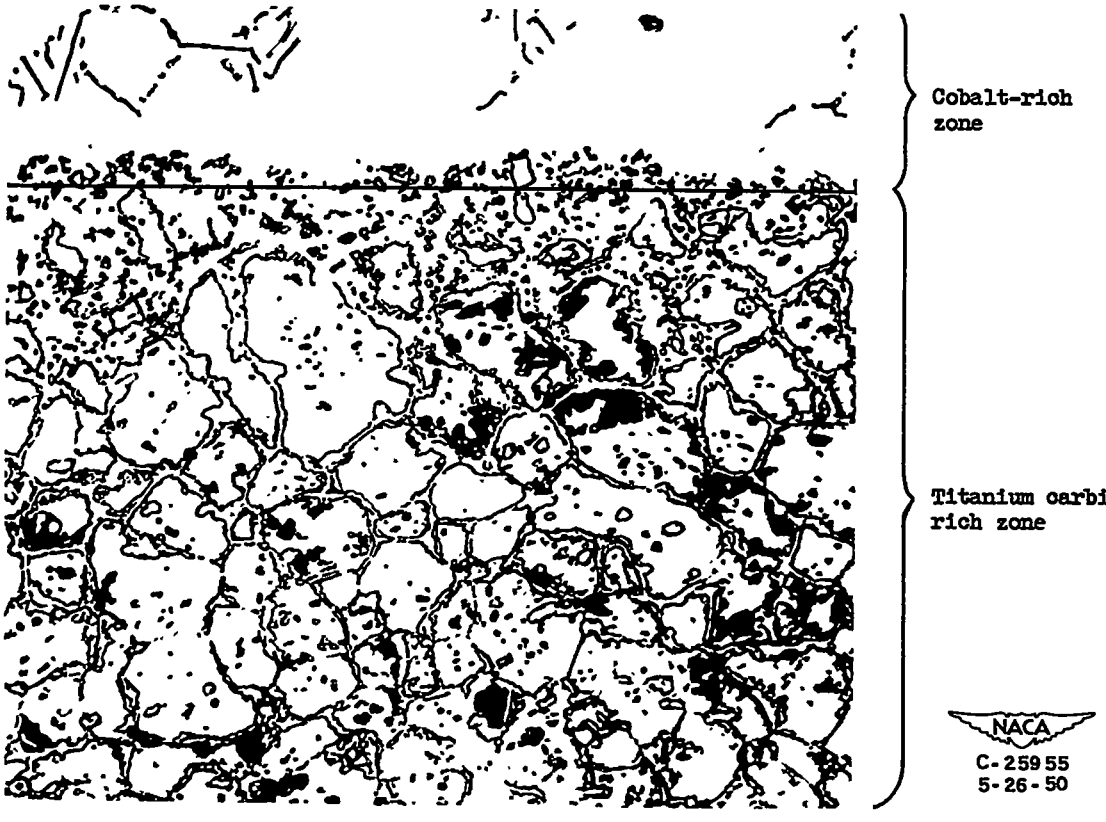


Figure 5. - Cobalt - titanium carbide. Etchant, potassium hydroxide - potassium ferricyanide. X1000.

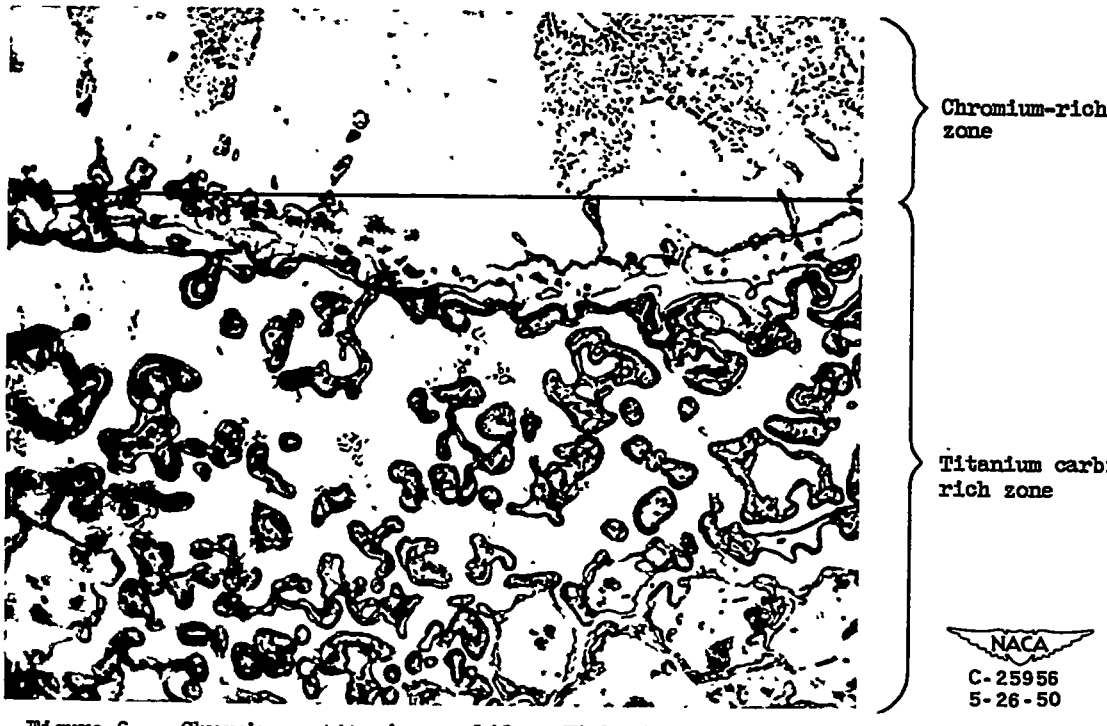


Figure 6. - Chromium - titanium carbide. Etchant, potassium hydroxide - potassium ferricyanide. X1000.

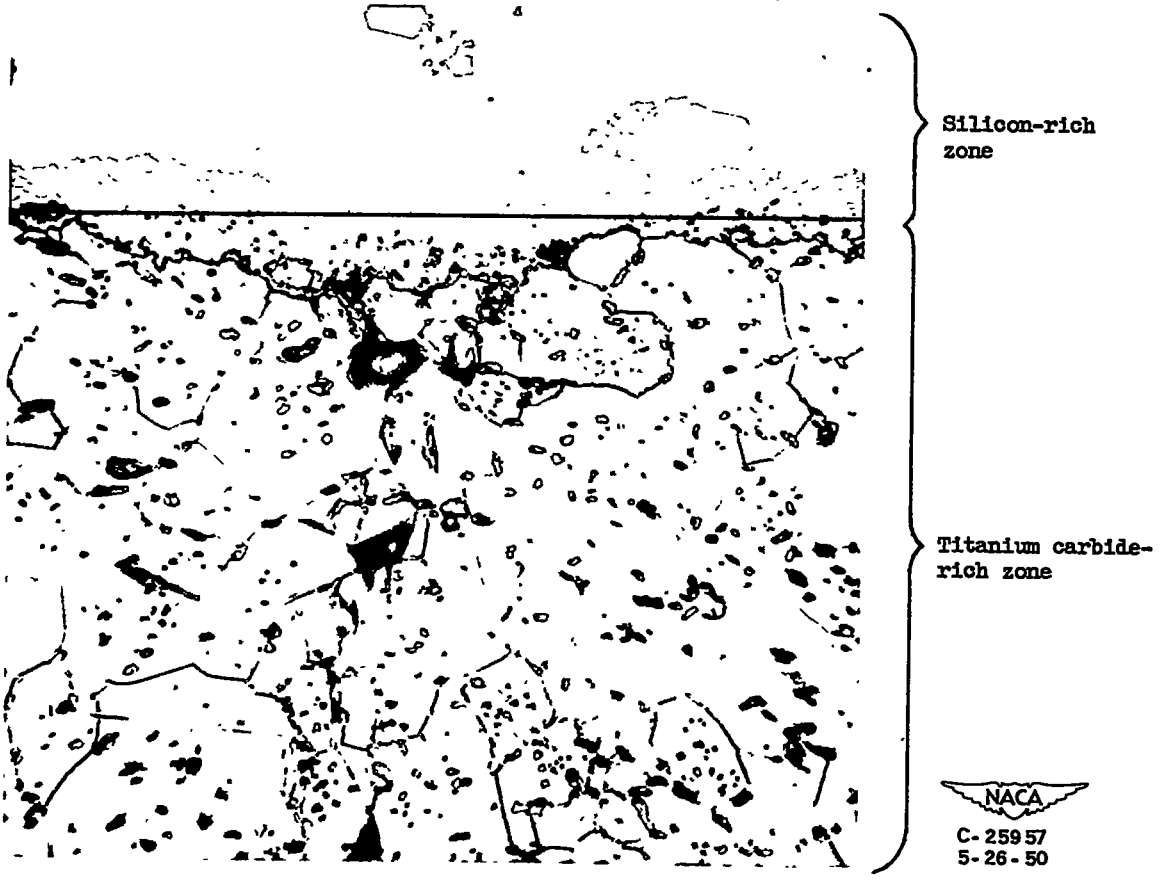


Figure 7. - Silicon - titanium carbide. Etchant, potassium hydroxide - potassium ferricyanide. X1000.

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