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Investigation of Infiltrated and Sintered Titanium Carbide

Goetzel, Claus G.; Adamec, John B.; Ellis, John L. and Others (Submitters) April 52 58pp. photos, tables, graphs

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INVESTIGATION OF INFILTRATED AND SINTERED TITANIUM CARBIDE

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FOREWORD

This report serves as a final and summary report of the research and development investigation carried on under U. S. Air Force Contract No. AF 33(038)-18103, Expenditure Order No. 605-253-SR-3A, said contract having been entered into by the Materials Laboratory, Research Division, Wright Air Development Center, Wright-Patterson Air Force Base, Dayton, Ohio, and the Sintercast Corporation of America, on October 2, 1950.

All work on this contract was performed under the general supervision of Dr. Claus G. Goetzel, Director of Research of the Sintercast Corporation of America. Project Engineer was Mr. John B. Adamec, with Mr. John L. Ellis, Chief Engineer, assisting in the fabrication of the stress rupture test bars, and Mrs. Dorothy Trauberman, Research Associate, assisting in the metallographic and thermal shock testing, and in the preparation of this report.

We are indebted to Dr. John P. Nielsen, Professor of Metal Science, New York University, for his direction of the X-ray diffraction analysis work of the oxide products, and to Mr. Elmars Ense, Research Associate, Metallurgy Laboratory, College of Engineering, New York University, who conducted the detailed investigation.

We are also obliged to Messrs. Eugene P. Poluschkin, Richard B. Wagner, and John Derbyshire for their collaboration on some of the technical details of this report, including the preparation of photographs, photomicrographs, and drawings.

ABSTRACT

An investigation was carried out for the purpose of evaluating composite bodies consisting of titanium carbide and nickel-base alloys and intermetallic compounds, produced by the infiltration and conventional powder metallurgical processes for use as structural materials in high-temperature components of aircraft engines.

The nickel alloys and the one compound tested were Nichrome V, Hastelloy "C", Inconel, And NiAl, respectively. Production techniques were developed for nine in. long stress rupture test bars, containing Inconel as infiltrant. These were produced by the infiltration process and submitted to the Materials Laboratory, Research Division, Wright Air Development Center for stress rupture tests together with specimens of similar composition made by sintering.

Tests carried out on the material were:

1. oxidation testing in air at 1600, 1800 and 2000°F;
2. X-ray diffraction analysis of the oxide products;
3. thermal shock testing at 2500 and 2300°F;
4. modulus of transverse rupture at room temperature;
5. modulus of transverse rupture at 1800°F;
6. ductility at 1800°F.

It was found that, among the materials tested, titanium carbide-Inconel, infiltrated by the capillary infiltration method, had the most favorable combination of high-temperature strength, ductility, and oxidation resistance at elevated temperatures. Its facility of production, however, was inferior to that of titanium carbide infiltrated with other nickel alloys. While it was possible to produce from Inconel-infiltrated titanium carbide 3 x 1/2 x 1/2 in. specimens of great physical and structural uniformity, nine in. long bars required for stress rupture specimens still possessed some regions of less homogeneity as evidenced by microporosity.

In the modulus of transverse rupture tests, the Inconel-infiltrated type of material proved to be far superior in strength at room temperature, strength at 1800°F, and bending capacity at 1800°F, over a cemented titanium carbide of similar composition made by cold-pressing and sintering.

PUBLICATION REVIEW

Manuscript Copy of this report has been reviewed and found satisfactory for publication.

FOR THE COMMANDING GENERAL:

M. E. SORTE
Lt. Col., USAF
Chief, Materials Laboratory
Research Division

INTRODUCTION

The general scope of the investigation, constituting the subject matter of this report, was to conduct research and development on materials having the physical and chemical properties suitable for use as structural components, at high temperatures, in aircraft power plants. More specifically, the investigation concerned itself with the development of composite powder metallurgical bodies consisting of two distinct, interspersed phases, a refractory metal carbide and a high temperature oxidation resistant metallic alloy.

The trend towards design for higher operating temperatures in modern jet engines has produced requirements of high temperature oxidation resistance, strength properties, thermal shock resistance, etc., which seem to point favorably towards the utilization of "cermets" of the type described above. Such duplex materials can be designed to exhibit a range of physical and chemical properties throughout the range of usable temperatures, i.e., 1600 to 2000°F, far superior to those of either of the material components alone.

A considerable number of previous investigations have been carried on in the development, by the methods and processes of powder metallurgy, of the aforementioned type of duplex materials which would exhibit good oxidation and creep resistance as well as show satisfactory wear resistance, ductility, and strength under the stringent conditions of operation in a jet engine at temperatures of approximately 1800°F.

Aside from the conventional powder metallurgical processes of hot-pressing and cold-pressing and sintering, the process of impregnation was found to be particularly promising as a method for producing the type of material under consideration. The theory of this process postulates that when a skeleton of a refractory metal carbide is impregnated with a metallic phase, two intertwined and individually coherent networks, the metallic and the refractory metal carbide phases, coexist in the final composite structure. The properties of the final body, therefore, are a combination of the two phases present, with the accent of the particular set of properties depending on whether the carbide or the metallic phase is the predominant

one. The high creep resistance, hardness, and strength at elevated temperatures of a refractory metal carbide, such as titanium carbide, could thus be paired with the high temperature oxidation resistance and ductility characteristics of such alloys as Hastelloy "C", Nichrome V, Inconel, or others.

Early investigations showed that the impregnation technique was applicable to the production of composite bodies of refractory metals, as well as their carbides and borides, and the comparatively low melting metallic phases of nickel-, cobalt-, and iron-base alloys. The melting and solubility characteristics of such systems, with particular reference to titanium carbide, were investigated by the Ohio State University [1]. It had been noted, however, that such composite bodies, especially when based on tungsten, molybdenum, or tungsten carbide, displayed poor oxidation resistance at elevated temperatures. Attempts were made to solve this problem by surrounding the skeleton grains with an oxidation resistant alloy [2], encasing the entire composite body with a stable, oxide-forming case, and alloying the refractory skeleton material to form a multi-carbide composite [2),3)]. Those investigations met only with partial success. They did, however, show that nickel- and cobalt-base alloy infiltrants resulted in more oxidation resistant composites than did those of iron-base alloys. The former alloys also showed superior strength and ductility characteristics at 1800°F.

The Sintercast Corporation of America conducted extensive investigations in this field during the time preceding this contract, and concentrated their efforts on titanium carbide as the refractory component. The basic work of this investigation consisted of:

1. Modification and refinement of commercial grades of titanium carbide by carefully controlled heat treatment under special atmospheres with or without the addition of other metallic constituents in order to form multi-carbides.

2. Determination of the effect of process variables such as particle size, time and temperature of hot-pressing and sintering, on the structure and density of refractory metal carbide skeletons.

3. Determination of the effect of the refractory metal carbide skeleton density on subsequent impregnation with lower melting metallic phases.

4. Development of methods for testing strength and ductility characteristics at room and elevated temperatures for skeleton and composite bodies, as well as high temperature oxidation resistance of the latter. Analytical chemistry, microscopy, and radiography methods were used for the investigation of internal structure, composition, soundness, and homogeneity of the bodies.

5. A correlation of properties determined by these methods as related to process variables, and the composition of the skeleton material, infiltrant type, and the final composite body.

SECTION I

DEVELOPMENT OF PRODUCTION TECHNIQUES FOR MAKING TEST BARS

The test bars produced ranged from 3 x 5/8 x 1/4 in. to 9 x 1/2 x 1/2 in. in accordance with the requirements of production and testing equipment as well as contract specifications. The two primary methods of production used during this investigation were the impregnation method and the conventional powder metallurgical methods of hot-pressing and cold-pressing and sintering.

The nature of the contract and type of work necessitated limiting the investigation to titanium carbide as most promising for the refractory metal carbide phase, and to select as the metallic infiltrant or binder phase the nickel-base alloys Inconel, Nichrome-V, Hastelloy "C" and the intermetallic compound NiAl.

A. Impregnation Method

This method consisted essentially of producing a porous titanium carbide skeleton body by hot-pressing titanium carbide powder to which approximately 10% of metallic binder powder was admixed, to a density of 60 to 70%, and, during a subsequent, separate operation, introducing the molten metallic infiltrant.

1. Skeleton Production

a. Powder Preparation

The titanium carbide powder, as standardized for the production of test bar skeletons, consisted of commercial grade titanium carbide powder, heat-treated under a purified hydrogen atmosphere at a temperature in excess of 3000°F. The resultant cake was then comminuted to 140-mesh powder by crushing. Heat-treating of the commercial powder under a nitrogen atmosphere at temperatures up to 4200°F did not result in an improvement over the hydrogen-heat-treated type of powder. The addition of 2% of aluminum powder to titanium

carbide and a subsequent heat-treatment under desiccated hydrogen at 4000°F did likewise not result in a noticeable improvement of the properties of the test bars or facilitate their production.

b. Standard Method of Test Bar Skeleton Production

The test bar skeletons, 3 x 3/8 x 1/4 in., 3 x 1/2 x 1/2 in. and 9 x 1/2 x 1/2 in., were produced by conventional hot-pressing techniques in graphite molds from 140-mesh heat-treated titanium carbide powder mixed with 10% nickel binder. Figs. 1 and 2 show the graphite mold assembly and the finished skeleton for the 9 in. type bar, respectively. The operation consisted of forcing the two punches (A and B) into the mold case (D), at increasing pressures up to 1500 psi when the assembly had been heated to 2900-3000°F by a surrounding high frequency coil. The punches, traveling along a tapered keyway in the mold case, translated the lateral pressure exerted on them by the press into a transverse pressure on the powder contained in the mold (C) between the punches. It was found that best results of infiltration ease and structural uniformity were obtained by holding the pressure at the peak temperature for a certain period of time in strict relation to the specimen and mold size. The skeleton bars were pressed to a density of 60 to 70%. The 9 in. long bars were ground to octagonal cross section before impregnation. Hot-pressing of the skeletons under nitrogen or a subsequent heat treatment under nitrogen did not result in any significant improvement.

c. Skeleton Strength

A series of tests to determine the modulus of transverse rupture of skeletons composed of titanium carbide with and without binder, both hot-pressed, cold-pressed and sintered, and hot-pressed and sintered in Norblack under desiccated hydrogen, were carried out. The reason for the above tests lay in the belief that the skeleton strength has a significant effect upon the strength of the final composite body. It was found that the bars made from binder-free titanium carbide were too fragile to be tested. Otherwise, the method of manufacture seemed

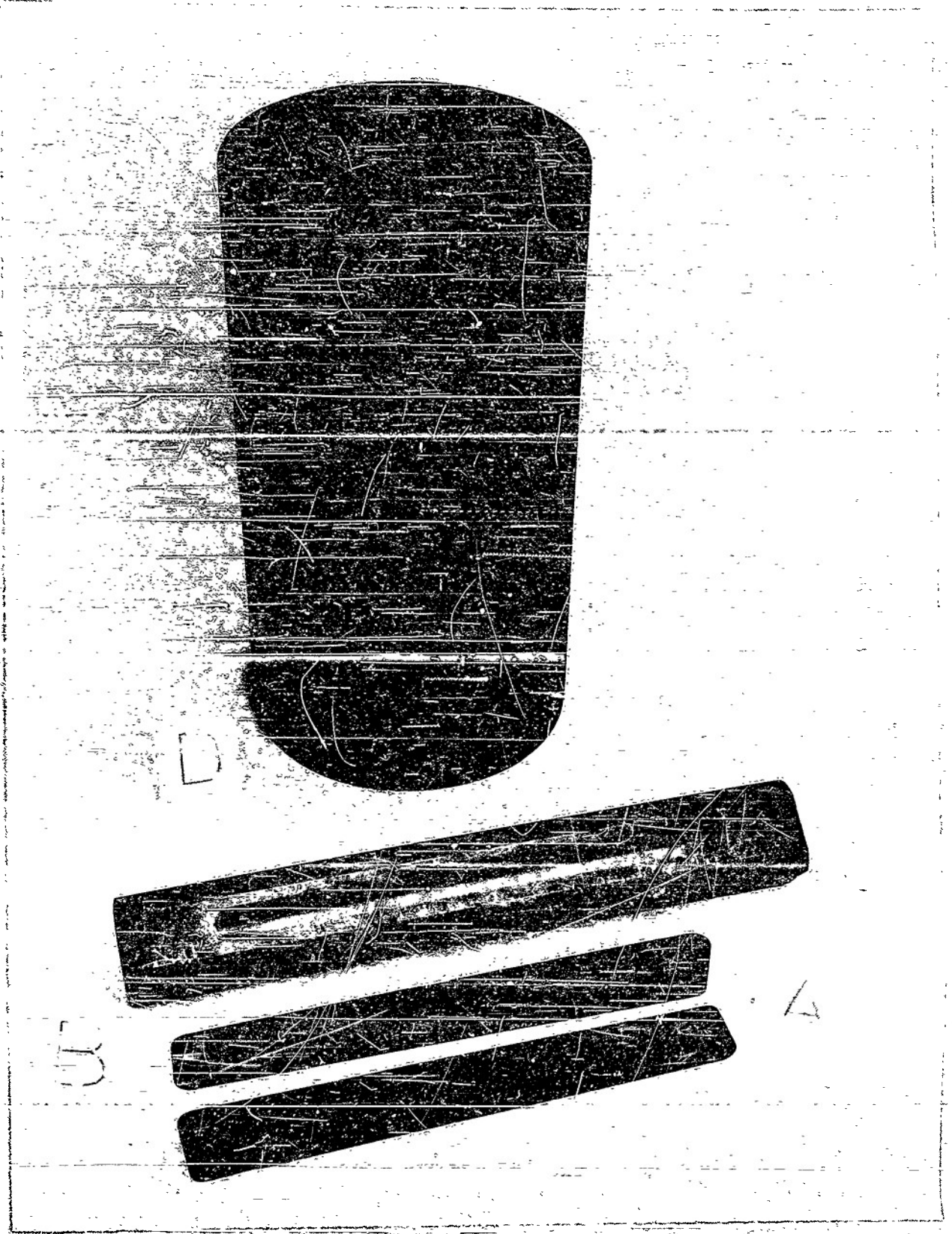

FIGURE 1

View of Disassembled Graphite Mold Used for Hot-Pressing Nine Inch Long Titanium Carbide Skeleton Bars, Consisting of (A) and (B) Tapered Punches, (C) Mold, and (D) Mold Case.

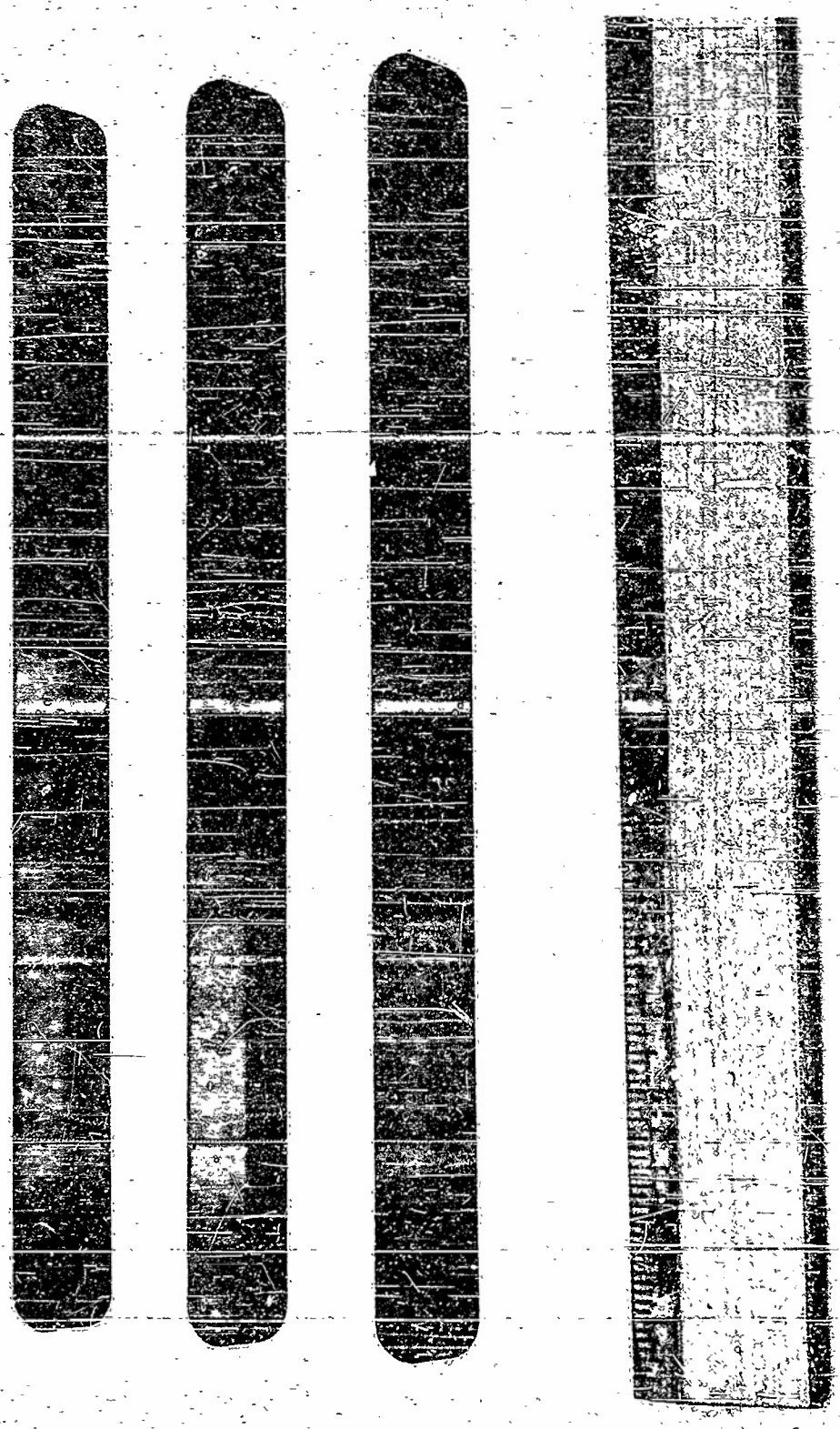

FIGURE 2

View of a Series of Nine Inch Long Hot-Pressed Titanium Carbide Skeleton Bars.

to have no appreciable effect on the strength with the exception of an improvement by sintering in Norblack under a hydrogen atmosphere above the melting point of the binder subsequent to hot-pressing. Table I shows a typical set of values for skeletons with 10% metallic binder as tested at room temperature and at 1800°F. The increased strength of the sintered skeletons is believed to be due to an increase in the effectiveness of bonding of the titanium carbide particles by the nickel as accomplished by diffusion processes allowed to progress further by prolonged sintering at a temperature above binder liquefaction, as compared to the short-time exposure of the body to the same temperature during hot-pressing.

2. Impregnation of Refractory Metal Carbide Skeletons with a Metallic Phase

a. Impregnation Methods

A number of methods of impregnating the refractory metal carbide skeleton with a molten metallic phase were investigated for the purpose of finding the method which would result in the most homogeneously penetrated pore-free skeleton of optimum properties. All impregnation operations were carried out in horizontal, graphite-tube, high frequency heated furnaces under a desiccated hydrogen atmosphere. All molds and mold investments were made of refractory or refractory cements. Aside from the different methods of infiltration, the effect of penetration into and dissolution of the skeleton by the molten infiltrant was investigated relative to the temperature and length of time of infiltration. The following are the primary methods of infiltration investigated.

(1) Transverse Capillary Infiltration Method

This method proved to be the most satisfactory and was consequently adapted as the standard method for production of the 9 in. type of test bars to be delivered as specified by the contract. In this method, the skeleton bar was placed in a ceramic crucible or vessel

TABLE I

Comparison of Modulus of Rupture Strength at Room Temperature and 1800°F of Titanium Carbide Base Skeletons Produced by Hot-Pressing 1/

Hot-Pressing Temp., °F	Sintering		Density %	Modulus of Transverse Rupture, psi	
	Time hrs	Temp. °F		Room Temp.	1800°F
2900	1	2550	61.2	-	8040
2900	-	-	60.4	-	5490
2900	1	2550	58.4	9000	-
2900	-	-	59.0	5950	-

1/ All compositions contained 10% nickel by weight

and the proper infiltrant allotment, in the form of strip or shot, placed on top or bottom, or both, of the bar. Capillary force-activated infiltration of the infiltrant into the skeleton then occurred during heating of the assembly for times of one hour or less at temperatures above the melting point of the infiltrant. It was found that test bars of a 1/2 x 1/2 in. cross section exhibiting no macroporosity, little or no microporosity, and a generally satisfactory uniformity of properties in the longitudinal direction, could be produced by this method with most infiltrants, provided the skeletons had previously been properly prepared as described above. Fig. 3 shows a 9 in. bar inside a ceramic vessel after infiltration. It was found essential in this method that excessive dissolution of the carbide skeleton should not be allowed to occur due to prolonged time at infiltration temperature and greatly excessive amounts of infiltrant. Figs. 4 and 5 show, at 200 and 1200 times magnification, respectively, the core-free, virtually microporosity-free structure of a 60-65% dense titanium carbide skeleton infiltrated with Hastelloy "C". The macrophotograph, Fig. 6, of the same specimen shows the relatively uniform structure of the specimen throughout. Figs. 7, 8, and 9 show the micro- and macro-structures of a comparable test specimen infiltrated with Inconel. In this case, although there is no evidence of coring or other macroscopic non-uniformity, microporosity could not entirely be eliminated. Table II shows the effect of the direction of infiltration on the modulus of rupture values, and the effective elimination of this phenomenon by infiltration from two opposite directions. Fig. 10 shows the variation in modulus of transverse rupture over the length of a ground 9 in. representative stress rupture bar. The variation in properties can be presumed to be caused by local microporosity areas.

(2) Longitudinal Capillary Infiltration Method

The procedure in this method varied from that in (1) by the fact that the molten infiltrant, driven by capillary force, was allowed to penetrate from one or both ends of the test bar skeleton only, the sides of the skeleton being blocked from contact with the molten in-

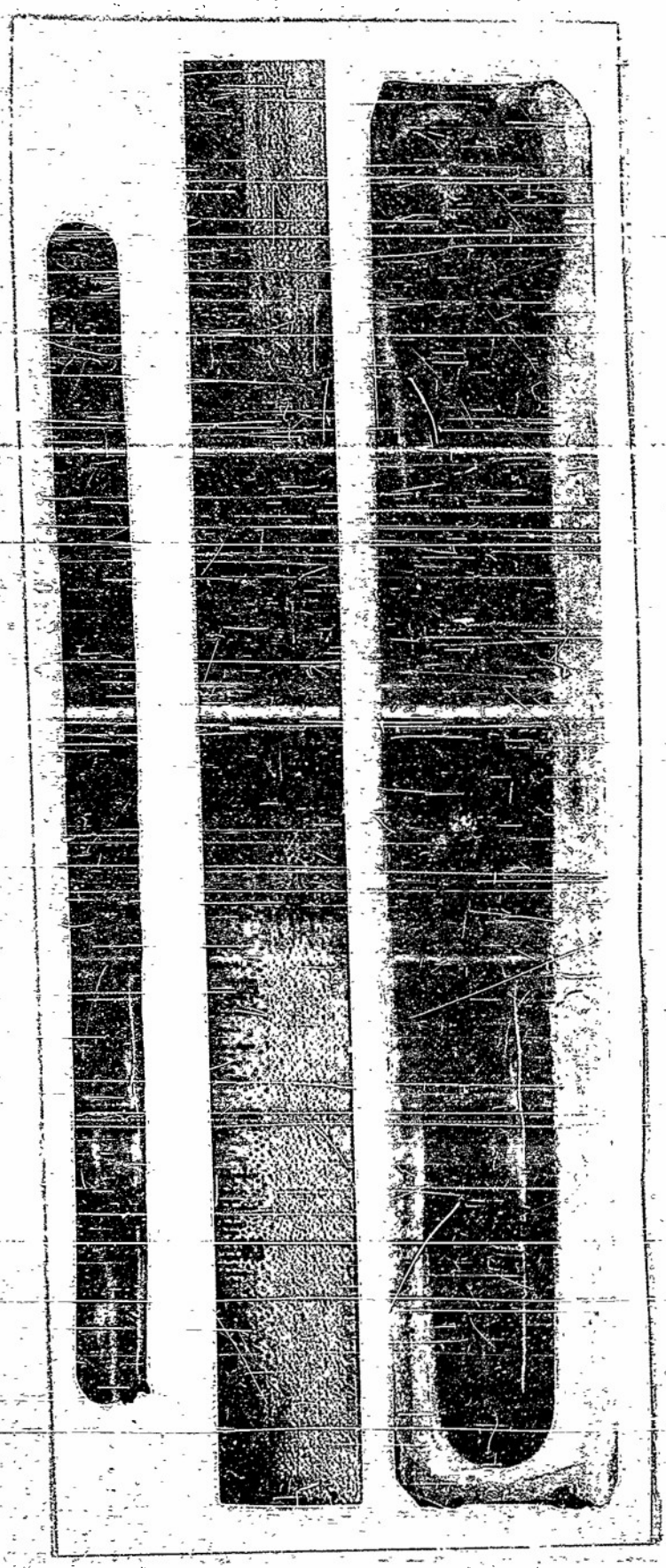

FIGURE 5

Composite View of Nine-Inch Long Titanium Carbide Bar After Transverse Impregnation With Nickel-Chromium Alloy. Bottom, Bar in Open Infiltration Vessel; Top, Appearance of Bar.

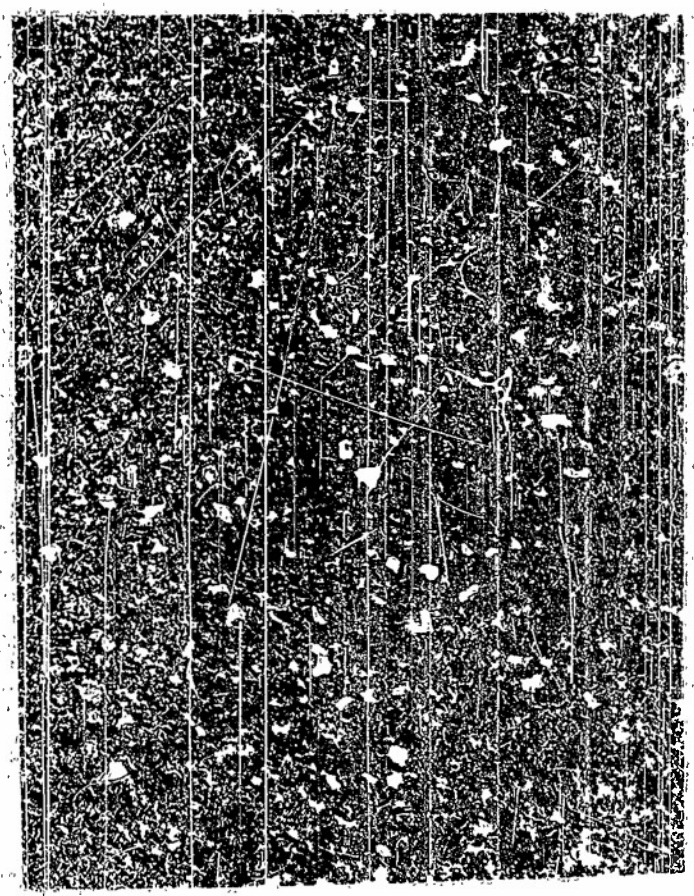

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FIGURE 4

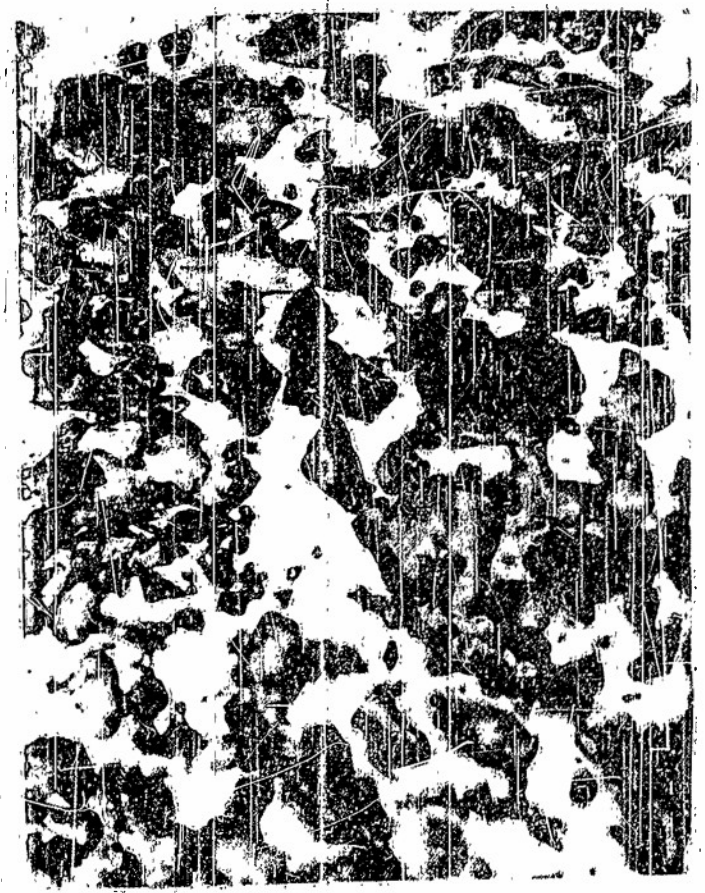

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FIGURE 5

Photomicrographs of Hastelloy "C" Impregnated Titanium Carbide Taken at Center of Specimen. Etched with Murakami Reagent.

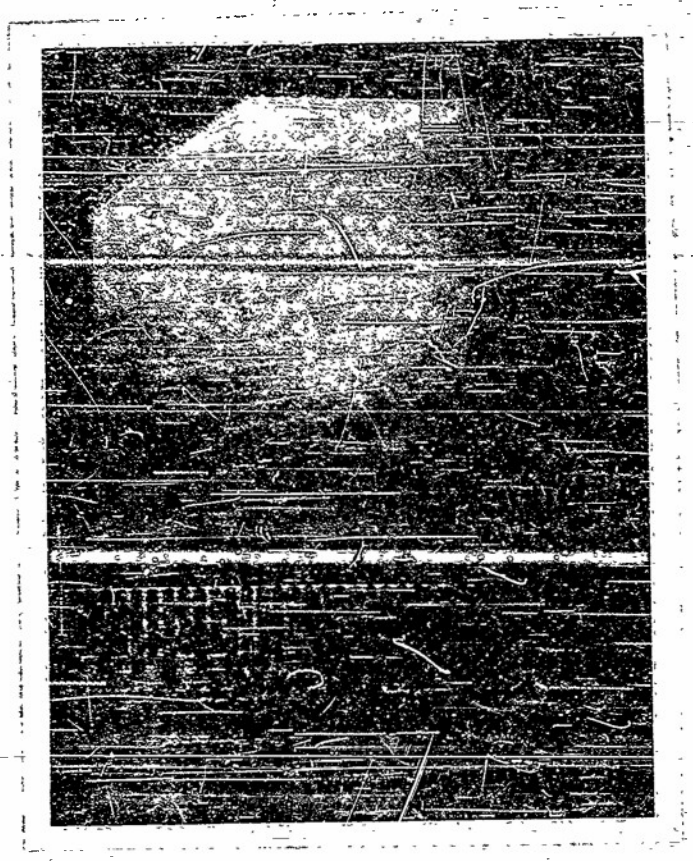

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FIGURE 6

Macrosection of Hastelloy "C" Impregnated Titanium Carbide. As polished.

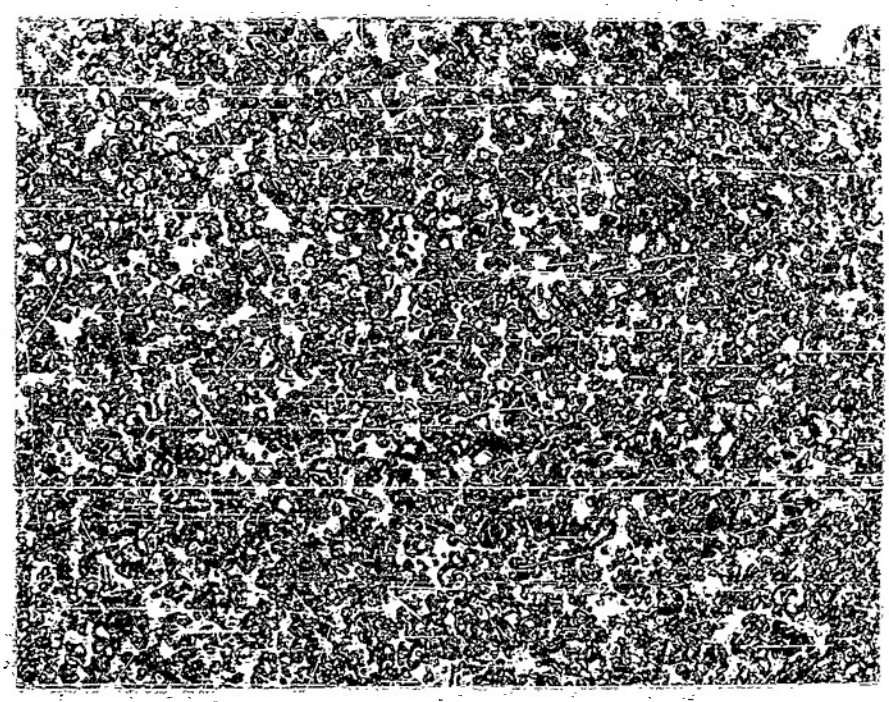

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FIGURE 7

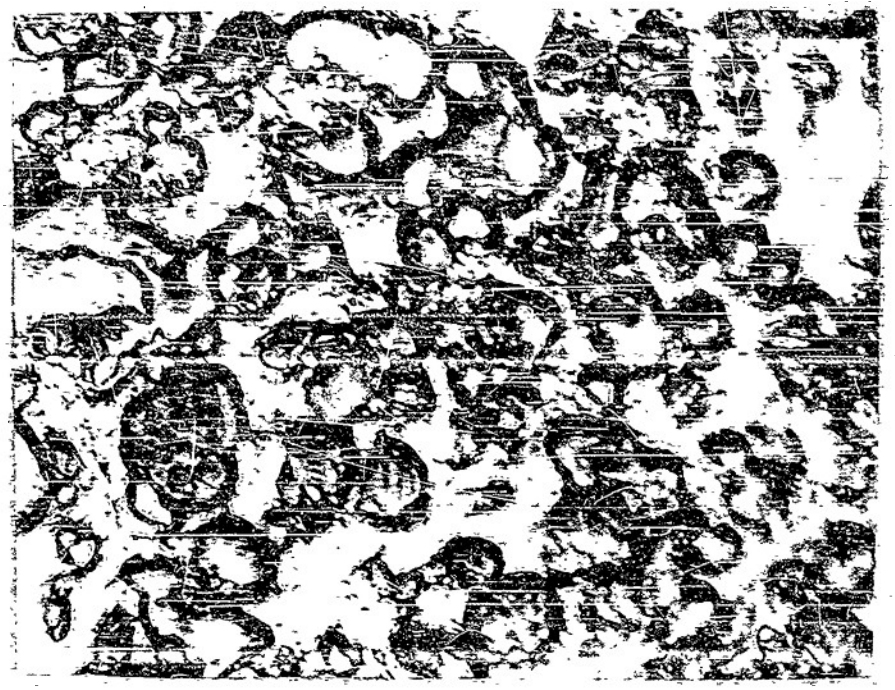

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FIGURE 8

Photomicrographs of Inconel-Impregnated Titanium Carbide Taken at Center of Specimen. Etched with Murakami Reagent.

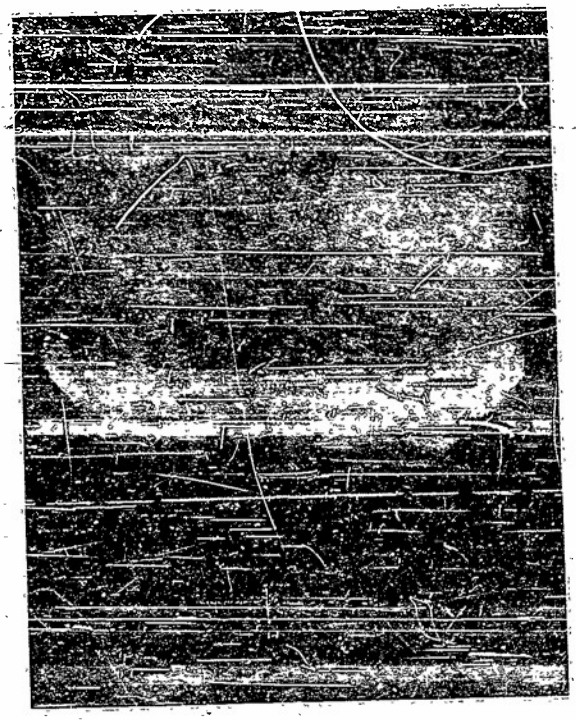

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FIGURE 8

Macrosection of Inconel Impregnated Titanium Carbide. As Polished.

TABLE II

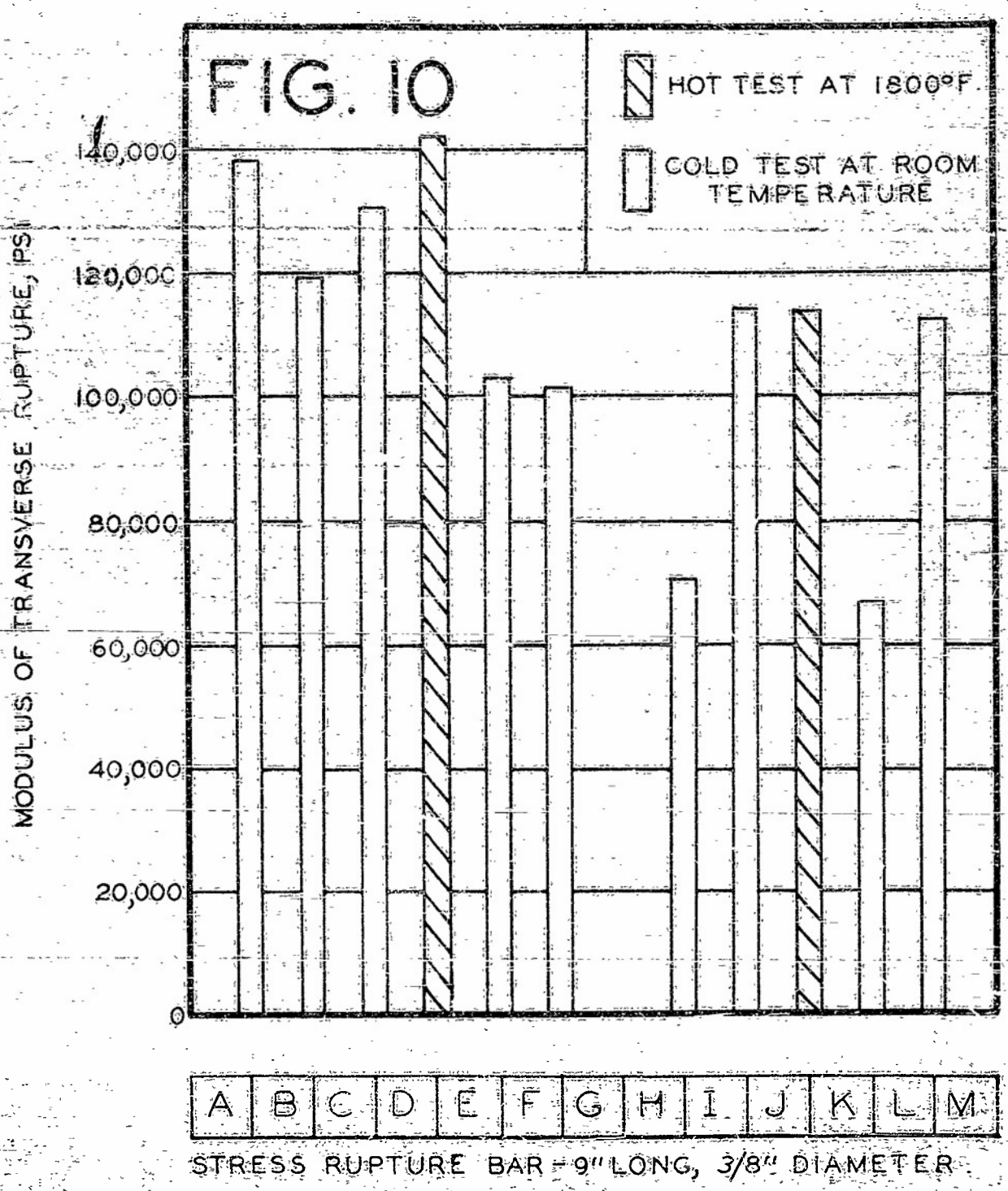
Transverse Rupture Properties of Titanium Carbide

Impregnated in Different Directions with Nickel-Base Alloy Hastelloy "C"

Direction of Infiltration 1/	Testing Direction 2/	Modulus of Transverse Rupture, Psi		Deflection under maximum load at 1800°F, in.	Hardness Rockwell "C" 3/
		Room Temp.	1800°F		
AG	A	117,000-120,000	101,000	0.105	61-63
	B	110,000-108,000	93,500	0.051	61-63
WG	A	118,000-148,000	103,000	0.182	58-65
	B	107,000-134,000	91,000	0.105	54-59
OD	A	120,000-137,000	112,000	0.105	62-68
	B	116,000-132,000	113,000	0.111	60-68
Sintered Titanium Multicarbide (K-151A) 4/		83,000-97,000, 112,000	60,000, 65,500	0.0085, 0.0015	67-71, 69-72

1/ AG, against gravity; WG, with gravity; OD, from two opposite directions
2/ A, fiber of original contact face with molten infiltrant in tension; B, fiber of original contact face with molten infiltrant in compression
3/ Readings taken on 30-N scale on surface of fiber subjected to tension in transverse rupture test, then converted
4/ Specimens originating from Kennametal were tested in this laboratory

VARIATION IN MODULUS OF TRANSVERSE RUPTURE OVER 9-INCH LENGTH OF 3/8-INCH DIAMETER GROUND STRESS RUPTURE BAR BLANK



filtrant by a ceramic case surrounding the bar. All skeletons thus infiltrated showed pronounced variation in the degree of infiltration leading to a practically infiltrant-free structure within one and a half in. of the end in contact with the molten infiltrant, and exhibiting at the ends signs of severe erosion and excessive dissolution of the skeleton.

(3) Horizontal Dip Impregnation Method

This method consisted of preheating the skeleton for 20 to 30 min at a temperature slightly above the melting point of the infiltrant and subsequently pushing it into a bath of molten infiltrant located in close proximity to the skeleton in the same furnace chamber. Although no coring effects were noticed, transverse planes of high density material, which proved to be due to transverse cracks in the skeleton, produced by thermal stresses upon immersion into the molten infiltrant bath, always occurred, thus making the production of isotropic composite bodies impossible.

(4) Pressure Impregnation Method (Transverse)

This method consisted of encasing the skeleton bar horizontally in an investment case which allowed the infiltrant to come in contact with the bar by means of a sprue along the bottom of the skeleton only. The molten infiltrant was conducted to the sprue from a reservoir above the bar, thus forcing the molten metal through the skeleton by the positive pressure of a head of molten infiltrant. Radiographic analysis showed that the infiltrant penetrated the bar along preferred paths, resulting in a non-uniform structure and composition.

(5) Pressure Impregnation Method (Longitudinal)

The procedure in this method was similar to that described in (4) with the variation of allowing the infiltrant to penetrate into the skeleton from one end or both ends only, the other sides being blocked off by a ceramic case

surrounding the skeleton. Fig. 11 shows the variation in modulus of rupture and hardness along the longitudinal direction of the impregnated bar for a unidirectionally impregnated bar. The severe reduction in strength at the entrance end of the skeleton bar was found by radiographic and metallographic study to be due to excessive skeleton erosion and solubility in the molten infiltrant; the equally severe reduction in strength at the opposite end was found to be caused by incomplete infiltrant penetration of the pore structure.

For the reasons of the structural and strength variations cited, it was judged doubtful whether specimens of sufficiently uniform properties could be produced by any of the techniques described, other than the first, which was accordingly adopted for the nine in. test specimen production.

8. Infiltrants Tested

The infiltrants tested were Hastelloy "C", Nichrome V, Inconel, and the intermetallic compound NiAl. Table III shows a representative set of modulus of rupture, ductility, and oxidation resistance data for titanium carbide skeletons impregnated with these infiltrants. On the strength of these data, NiAl-impregnated bodies were eliminated due to their low modulus of rupture and comparatively poor oxidation resistance. However, NiAl-infiltrated skeleton bars consisting of titanium carbide and 10% nickel binder with relative ease. Nickel had to be used as skeleton binder since it was not possible to press pure titanium carbide, and attempts to press skeletons containing aluminum, in addition to nickel, to correspond to the stoichiometric ratio in NiAl, also were not successful. The temperature of infiltration used for NiAl was 3100-3200°F.

Although both Hastelloy "C" and Nichrome V infiltrated the carbide skeletons with greater ease and considerably less gassing and dissolution of the titanium carbide, Inconel was ultimately chosen as infiltrant for the nine in. test bars submitted as specified in the contract, since this infiltrant resulted in composite titanium carbide bodies with a combination of physical and chemical properties superior to those of titanium carbide bodies infiltrated with the other alloys.

FIG. 11

MODULUS OF RUPTURE AND HARDNESS OVER LENGTH OF UNIDIRECTIONAL PRESSURE-IMPREGNATED TITANIUM CARBIDE BAR.

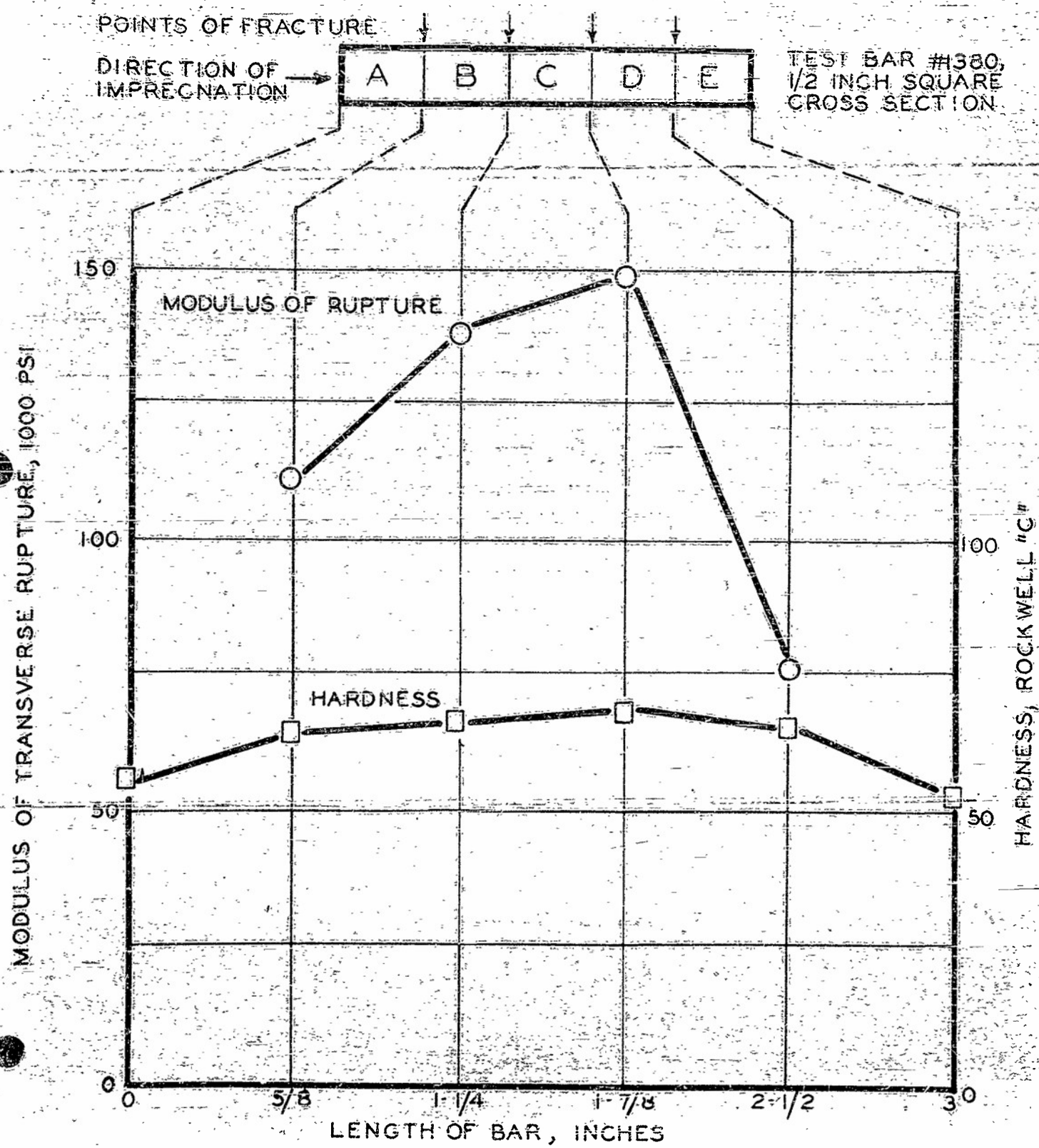


TABLE III

Influence of Type of Nickel Alloy Infiltrant on the Test Bars

Infiltrant Alloy	Modulus of Transverse Rupture, psi		Deflection under maximum load at 1800°F, in.	Oxidation[1/] Resistance at 1800°F mg/cm²
	Room Temperature	1800°F		
Hastelloy C	120,000-175,000	85,000-120,000	0.060-0.180	4.22-8.59
Nichrome	135,000-210,000	80,000-100,000	0.100-0.250	7.05-7.13
Inconel	130,000-200,000	70,000-95,000	0.125-0.350	6.24-6.75
NiAl	20,000-44,000	51,000-60,000	0.013-0.027	23.45

1/ Weight gain after 100 hrs exposure to still air at 1800°F; specimens withdrawn from test furnace, cooled, weighed, and reheated after each 20 hrs (total of 5 cycles).

B. Conventional Powder Metallurgical Methods

1. Hot-Pressing

It was attempted to produce dense, uniform bodies of a composition closely similar to that of infiltrated bodies by hot-pressing a mixture of titanium carbide and metal powders. A typical composition, in weight percent, was titanium carbide 50.6, nickel 40.7, chromium 8.7. The pressing was carried out in conventional graphite molds under a reducing furnace atmosphere at temperatures up to 3500°F. It proved impossible to consistently produce whole specimens in this manner since the excessive amount of binder was squeezed into the mold walls during pressing and caused the bars to break on subsequent cooling and extraction.

Table IV gives the physical properties of a 75:20:5 TiC:Ni:Cr (percent by weight) specimen as produced by hot-pressing. This table also shows physical property values of hot-pressed specimens subsequently infiltrated. It can be seen that the subsequent infiltration improves the strength properties significantly.

The hot-press method was not considered to be of sufficient merit for the production of composite bodies for comparison purposes, due to the production difficulties encountered and the relatively inferior physical properties and density obtained, as compared to infiltrated bodies.

2. Cold-Pressing and Sintering

To facilitate a comparison between the different methods, an investigation was then carried out with composite bodies, consisting of titanium carbide and various metals as binders, of compositions similar to those achieved by infiltration, by cold-pressing and sintering mixtures of the powdered ingredients. All sintering was done under purified atmospheres in graphite tube high frequency furnaces. Various ranges of compositions were investigated, and test bars duplicating the compositions of composite bodies infiltrated with Hastelloy "C", Nichrome V, and Inconel were produced. The most suitable amount of titanium carbide was found to be 60-65% by volume.

TABLE IV

Properties of Titanium Carbide Materials

Containing Nickel and Chromium Produced by Different Methods

Powder Preparation	Test Bar Preparation	Final Density %	Modulus of Transverse Rupture, psi		Maximum Deflection under load at 1800°F, in./2.6 in. span	Hardness Rockwell "C" 1/
			Room Temp.	1800°F		
70-20-5 TiC-Ni-Cr, wet milled 7 days in acetone in carbide mill	Cold-pressed at 70°F, 18 tsi Sintered at 2730°F for 1 hr in desiccated hydrogen	92.8 2/	73,600 82,400	103,000	0.051	64-70
	Hot-pressed at 3180°F, 2500 psi	90.9 2/	74,500 73,200	73,700	0.050	62-66
70-20-5 TiC-Ni-Cr, wet milled 24 hrs in stainless steel mill	Hot-pressed at 2910°F, 2500 psi, to 77.7% density Impregnated with additional Ni-Cr alloy	90.2 3/	137,000 137,000	91,400	0.068	55-62
	Hot-pressed at 3180°F, 2500 psi, to 86.0% density Impregnated with additional Ni-Cr alloy	89.4 4/	60,500 66,600	59,600	0.027	60-66

1/ Readings taken on 30-N scale, then converted
2/ Reference density was taken as 5.8 g/cc
3/ Reference density was taken as 6.6 g/cc
4/ Reference density was taken as 6.2 g/cc

A typical composition in % by weight is 50:40:10 TiC:Ni:Cr. The excessive amount of binder caused, in most cases, the specimen to blister and warp during sintering when carried out according to conventional practices of sintering above the liquefaction temperature of the binder alloy. Ranges of sintering time and temperature were investigated for the purpose of finding the best production procedure. The use of helium, wet hydrogen, or nitrogen, instead of the desiccated hydrogen usually employed as atmosphere during sintering, did not result in significant improvement of the properties of the specimen. Table IV shows the modulus of transverse rupture of a 75:20:5 TiC:Ni:Cr (% by weight) specimen as cold-pressed and sintered. The specimen produced in this manner usually displayed good ductility at elevated temperatures in the transverse bend test and generally modulus of rupture and other physical properties superior to those of hot-pressed test bars, but always significantly inferior to infiltrated specimens. Reduction of the titanium carbide particle size by prolonged milling of the powder prior to pressing did not result in a significant improvement of the physical properties of the test bars. The final bodies which were presintered, shaped, and fully sintered under desiccated hydrogen slightly below the theoretical melting point of the binder alloy to avoid fusion, blistering, and excessive deformation due to overheating, were usually only about 90% dense.

SECTION II

OXIDATION SCREENING TESTS

The oxidation tests performed on test specimens under this contract were carried out in a manner developed and standardized during past research projects. The data obtained is presented in comparison with that compiled for the commercial cemented titanium carbide grades K-138A and K-151A, under the auspices of the Materials Laboratory, Engineering Division, Air Materiel Command [4].

A. Method of Oxidation Testing

The rate of oxidation of the specimens at 1600, 1800, and 2000°F was determined by measuring the weight gain as a function of time. The specimens were heated in a dry-air muffle furnace.

B. Test Results

The primary results obtained from the above mentioned investigation on K-138A and K-151A, e.g., that the oxidation rate is parabolic and that, during oxidation, two distinct layers of oxide are formed on the surface of the specimens, were found to apply, in general, to the composite bodies tested here. The parabolic nature of the oxidation mechanism is attested to by the straight-line relation between the time of exposure and the square of the weight gain divided by the exposed surface area.

The oxidation behavior of titanium carbide skeletons infiltrated with Nichrome V, Hastelloy "C", and Inconel at temperatures of 1600, 1800, and 2000°F is shown in Figs. 12, 13, and 14, respectively. These figures include curves for the oxidation behavior of K-138A and K-151A for comparison purposes. Fig. 15 shows the comparative oxidation behavior of composite bodies of the various compositions. This oxidation study reveals that titanium carbide materials infiltrated with various nickel alloys differ with regard to their susceptibility to oxidation at different temperature levels. It appears that Nichrome V-infiltrated titanium carbide is subject to a more rapidly increasing rate of oxidation with increasing temperature in the range of 1600 to 2000°F than is Inconel-infiltrated titanium carbide. Hastelloy "C"-infiltrated titanium carbide was found to be slightly superior to combinations of titanium carbide with the other two nickel alloy infiltrants in the range of 1600 to 1850°F; its oxidation rate increased rapidly above 1850°F, however, making the material unsuitable for test specimens to be tested at higher temperatures, such as thermal shocking at 2300-2500°F.

The infiltrated materials were found to be considerably superior to the sintered titanium carbide composition cemented with nickel (K-151A) in the range in which they are relatively stable. Table III shows a further comparison of the oxidation behavior of the infiltrated bodies tested, emphasizing, in particular, the relatively poor oxidation resistance of the NiAl-infiltrated titanium carbide bodies.

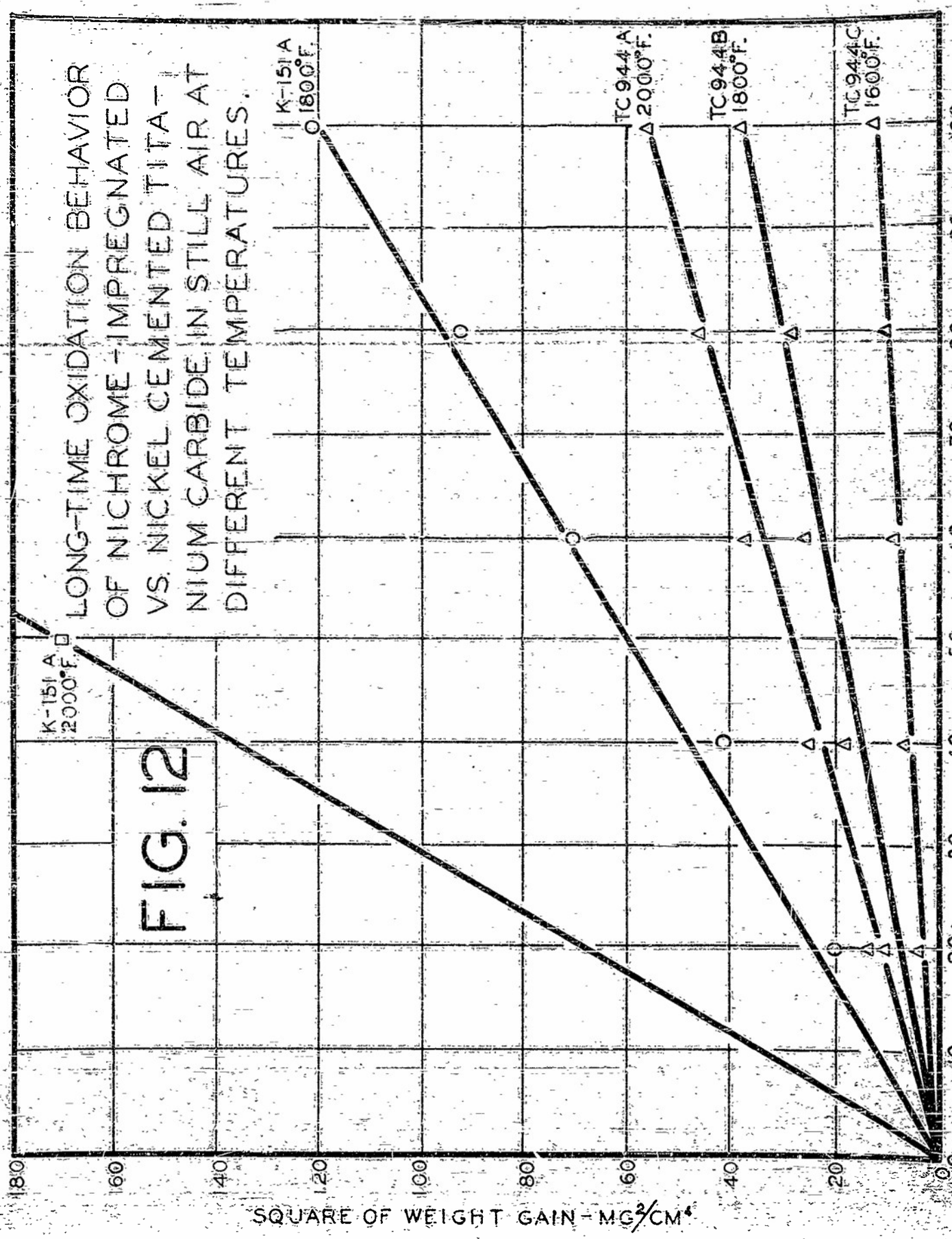


FIG. 12

LONG-TIME OXIDATION BEHAVIOR OF NICHROME-IMPREGNATED VS. NICKEL CEMENTED TITANIUM CARBIDE IN STILL AIR AT DIFFERENT TEMPERATURES.

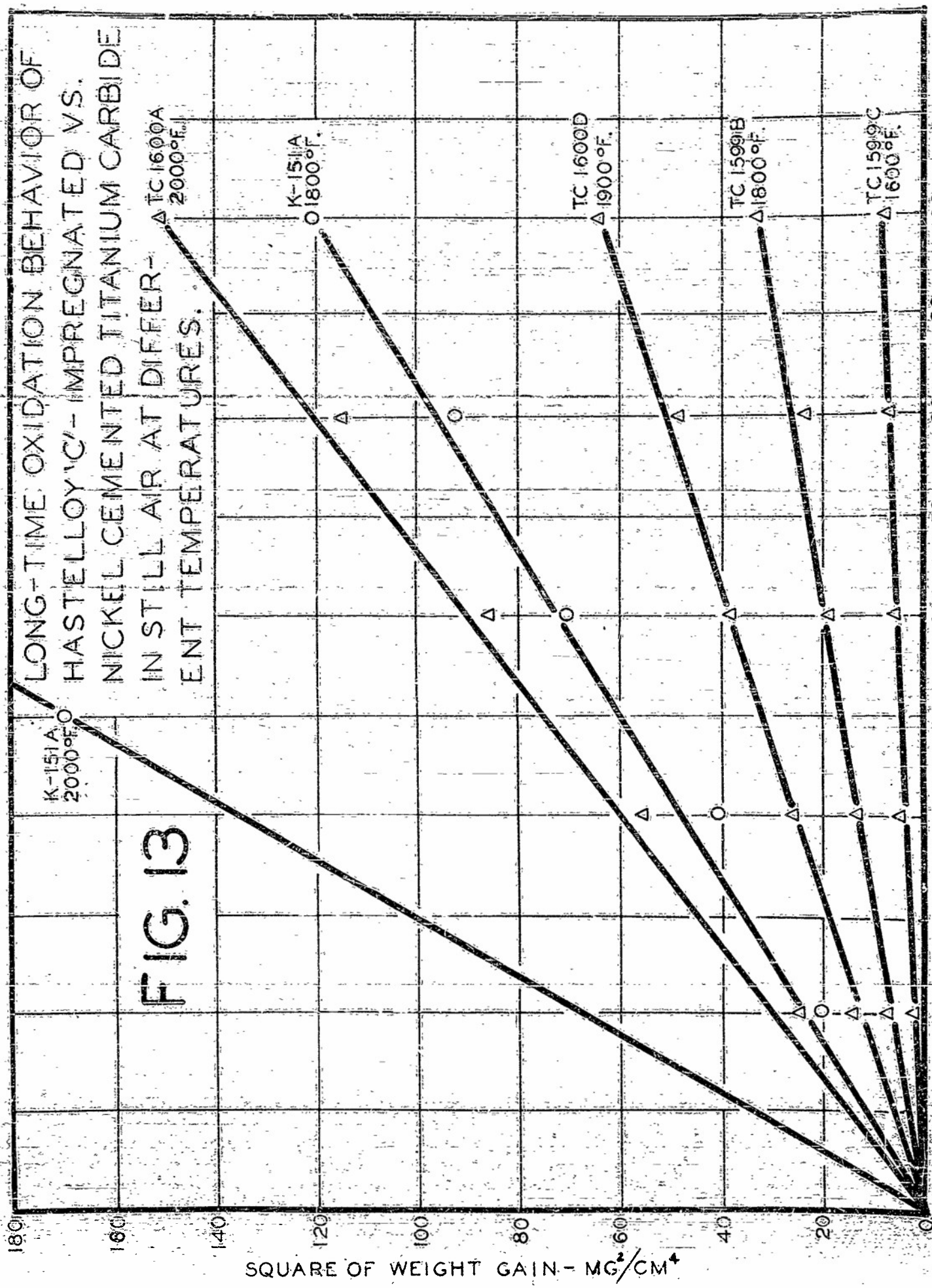

LONG-TIME OXIDATION BEHAVIOR OF
HASTELLOY 'C'-IMPREGNATED VS.
NICKEL CEMENTED TITANIUM CARBIDE
IN STILL AIR AT DIFFER-
ENT TEMPERATURES.
TC 1600A 2000°F.
K-151A 1800°F.
TC 1600D 1900°F.
TC 1599B 1800°F.
TC 1599C 1600°F.
K-151A 2000°F.
TIME - HOURS
SQUARE OF WEIGHT GAIN - MG²/CM⁴
0
10
20
30
40
50
60
70
80
90
100
0
20
40
60
80
100
120
140
160
180

FIG. 13

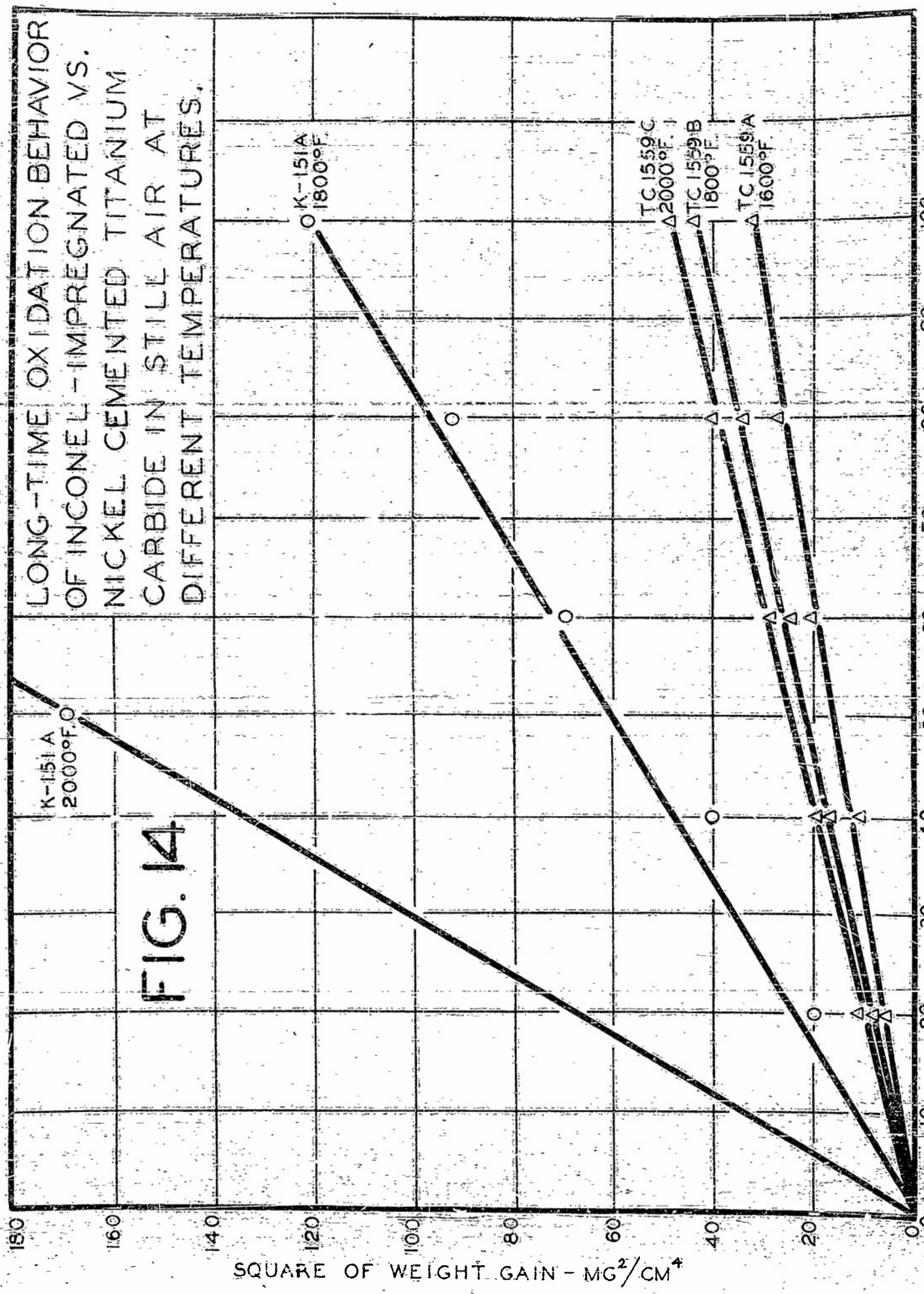

LONG-TIME OXIDATION BEHAVIOR
OF INCONEL-IMPREGNATED VS.
NICKEL CEMENTED TITANIUM
CARBIDE IN STILL AIR AT
DIFFERENT TEMPERATURES.
K-151A
2000°F.
K-151A
1800°F.
TC 1559C
2000°F.
TC 1559B
1800°F.
TC 1559A
1600°F.
TIME - HOURS
SQUARE OF WEIGHT GAIN - MG²/CM⁴
0
10
20
30
40
50
60
70
80
90
100
20
40
60
80
100
120
140
160
180

FIG. 14

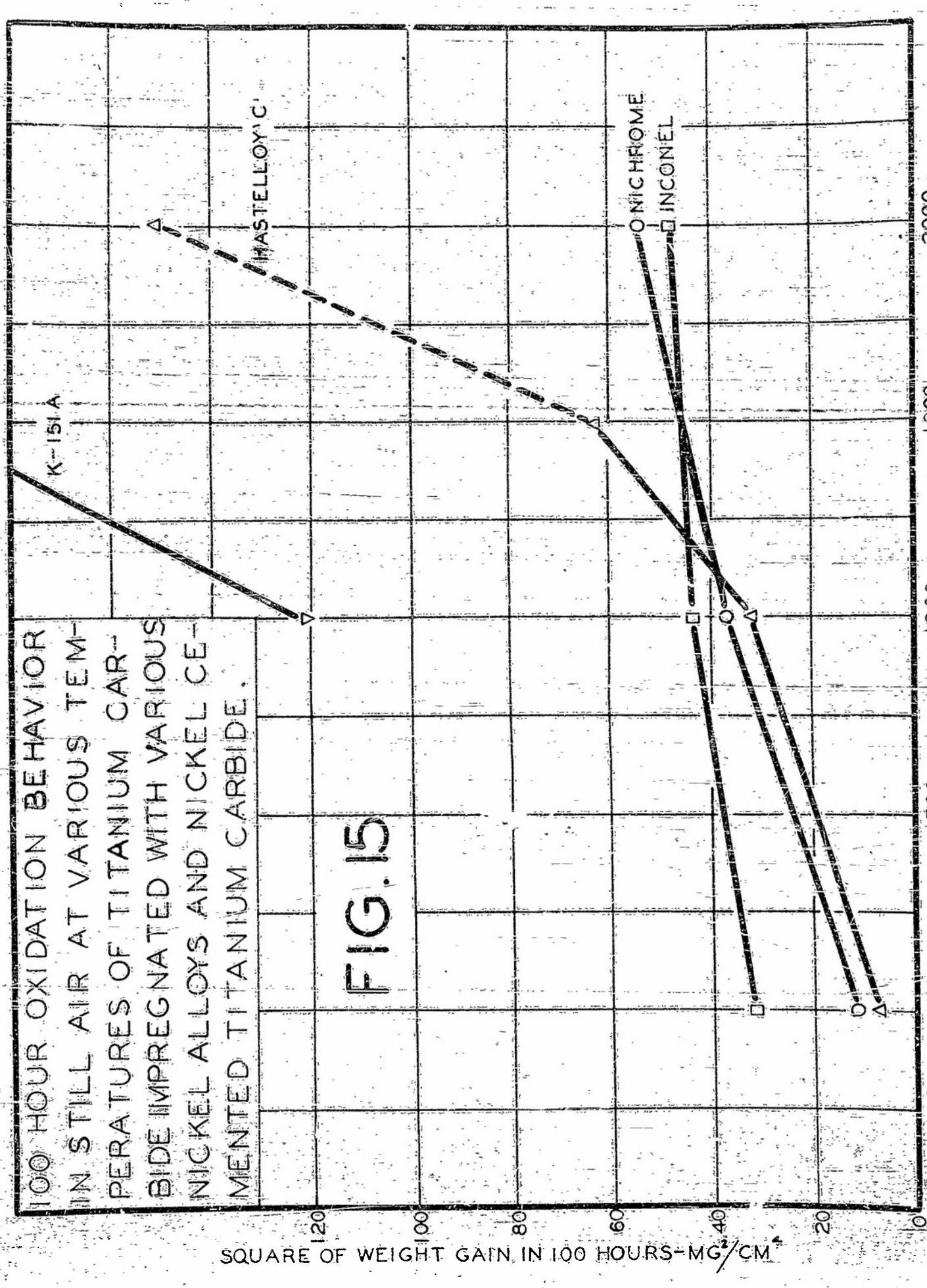


FIG. 15

100 HOUR OXIDATION BEHAVIOR IN STILL AIR AT VARIOUS TEMPERATURES OF TITANIUM CARBIDE IMPREGNATED WITH VARIOUS NICKEL ALLOYS AND NICKEL CEMENTED TITANIUM CARBIDE.

TABLE V

X-ray Diffraction Data (d in Å)

(CuKα radiation, film shielded by aluminum foil, time of exposure, 9 hrs)

Oxidation Product, Inconel-Infiltrated TiC - First Layer	"Synthetic Body" 1/
4.77 *	-
4.10 *	-
3.67 F	3.67 F
3.52 *	-
3.24 R	3.25 R
3.08 *	-
2.93 *	-
2.703 F	2.708 F
2.512 FR	2.515 FR
2.408 N	2.410 N
2.357 *	-
2.293 R	2.290 R
2.201 FR	2.203 FR
2.085 NR	2.087 NR
1.837 F	1.837 F
1.694 FR	1.697 FR
1.626 R	1.624 R
1.600 F	1.600 F
1.483 FR	1.485 FR
1.477 N	1.478 N
1.451 FR	1.452 FR
1.362 *	-
1.350 FR	1.348 FR
1.312 F	1.314 F
1.258 NF	1.258 NF
1.226 F	1.222 F
1.205 N	1.206 N
1.188 F	1.189 F
1.162 FR	1.163 FR
1.150 R	1.150 R
1.138 F	1.135 F
1.102 F	1.101 F
1.085 R	1.090 R
1.075 *	-
1.055 F	1.055 F
1.045 N	1.045 N

N, line belongs to NiO; F, line belongs to Fe_2O_3; R, line belongs to TiO_2 (rutile); *, unidentified.

1/ These lines are common to all six compositions listed in Table VI; based on the relative intensities of representative lines, the first layer oxide product is closest to synthetic body composition No. 5.

SECTION III

OXIDE INVESTIGATION BY X-RAY ANALYSIS

A. Preparation of Specimens

Specimens of titanium carbide infiltrated with Hastelloy "C" and Inconel were oxidized by heating them in a slow stream of oxygen at 2250°F. The resulting oxide was comminuted to -250 mesh and processed into test specimens suitable for X-ray examination. While the Hastelloy "C"-infiltrated material produced an oxide consisting of a fluffy, loose outer layer, and a very tenacious, thin inner layer, the specimens infiltrated with Inconel produced only thin adherent films which increased progressively less in thickness with increasing time of exposure.

B. X-Ray Data Analysis

Analyses of the various oxide products was carried out by X-ray. Table V is a compilation of X-ray data obtained for the oxide layer of an Inconel-infiltrated specimen. Table V also shows the data obtained for a synthetic specimen of apparently similar composition. Table VI gives the analysis of the series of synthetic specimens analyzed for comparison purposes. Accordingly, the major constituents of the oxide film were found to be 30% NiO, 50% Fe_2O_3, 10% TiO_2. The 10% of unidentified compounds did not correspond to TiN, TiC, or Cr_2O_3.

TABLE VI

Synthetic Body	1	2	3	4	5	6
NiO	40	30	30	30	30	20
Fe_2O_3	25	30	40	45	50	60
TiO_2 (rutile)	25	30	20	15	10	10
Inert Material	10	10	10	10	10	10

C. Coefficient of Thermal Expansion

The coefficient of thermal expansion of the Inconel-infiltrated titanium carbide material was found to be 4.44×10^{-6} per °F in the range of 70 to 1200°F.

Attempts to obtain the coefficient of thermal expansion of the oxide material by the methods suggested in the contract specifications, i.e., utilization of a synthetic body of similar composition, or complete oxidation of a thin wafer of the infiltrated material, were abandoned. The use of a synthetic body identical with the natural oxide product seemed unfeasible. No simple way was found to determine the composition, structure and effect on the physical properties of the aggregate of the 10% of unidentified matter. Furthermore, it would seemingly be impossible to duplicate the exact physical structure and phase distribution of the actual oxide film. It is, of course, apparent that the microstructure, in general, and any possible matrix formation, in particular, has a significant influence on the physical properties of the aggregate. A reliable and exact duplication of the expansion characteristics of the natural oxide product in such a synthetic body seems, therefore, not feasible.

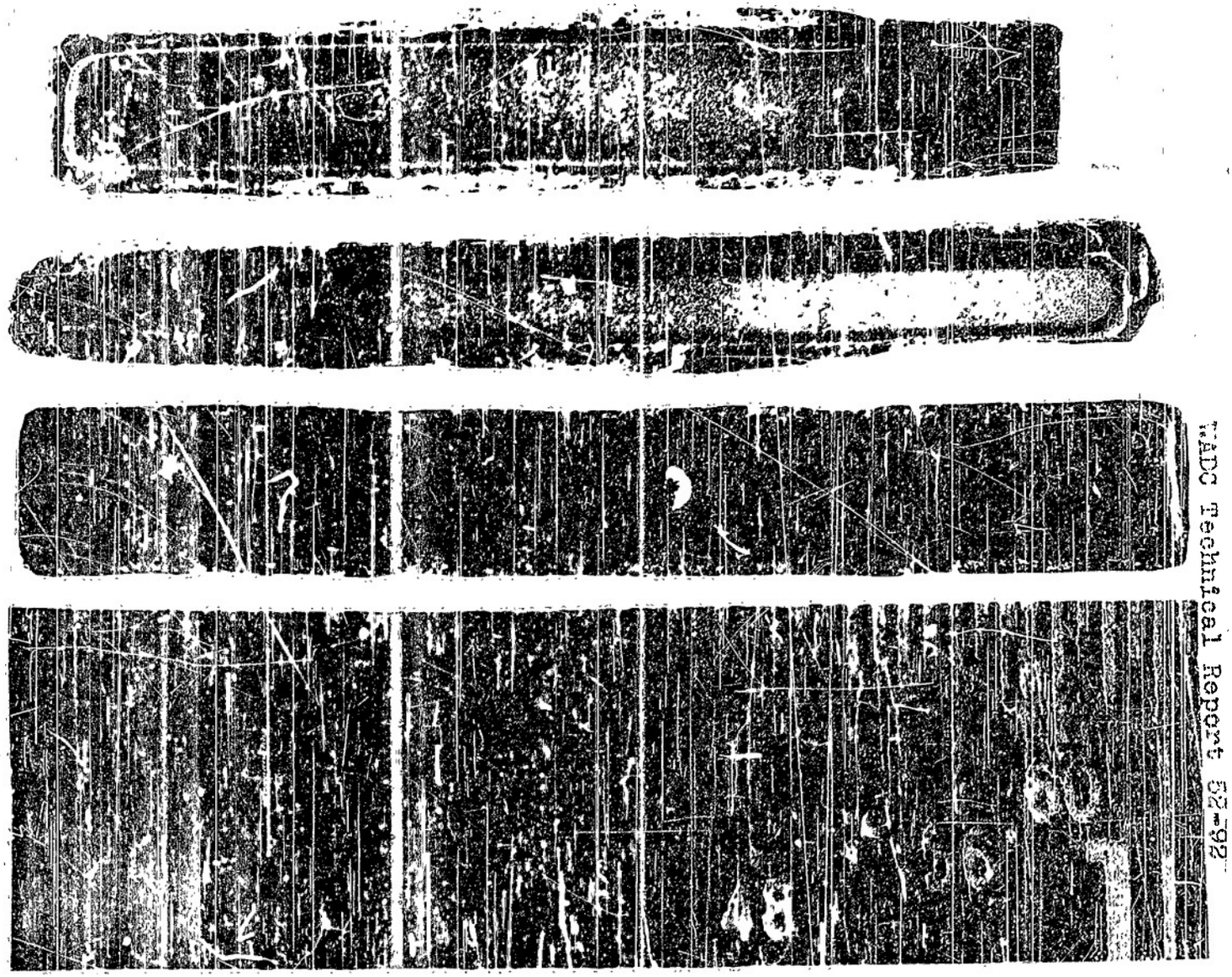
Attempts to prepare test specimens for thermal expansion measurements by a complete oxidation of a thin wafer of infiltrated titanium carbide materials were equally unsuccessful. Fig. 16 shows macrosections of two specimens of 0.090 and 0.060 in. thickness, which were oxidized in air for 100 hrs at 1800°F and a third specimen of 0.090 in. thickness which was oxidized in an oxygen stream for 18 hrs at 2250°F. It is apparent from the thickness of the respective oxide layers (approximately 0.016 to 0.026 in.) that, from a practical point of view, completely oxidized specimens cannot be produced from this type of infiltrated titanium carbide. Since the rate of increase in thickness of the oxide layer decreases with time, the maximum thickness of the layers cannot be expected to exceed approximately 1/32 in. after exposure to the most drastic oxidizing conditions within a reasonable period of time. The production of specimens of a thickness allowing for a complete oxidation throughout the specimen would be prohibitive from a fabricating standpoint, since it would require a metal specimen of a thickness considerably less than the final oxide body of an approximate thickness of 1/16 in.

In fact, scale measurements of the macrosections of the specimens in Fig. 16, showing residual base material thicknesses of 0.079, 0.039 and 0.081 in., respectively, indicate that wafers of 0.011, 0.021 and 0.009 in., respectively, would have to be produced to assure complete oxidation throughout. Methods for the production of wafers of such thicknesses were not available.

MA[illegible] OF [illegible] WA[illegible] OF INCOLOY-INFILTRATED [illegible] CA[illegible]ID [illegible] ON [illegible] PRODUCT SCALES AND [illegible] CORES

Exposed to Still Air for 100 hrs. at 1800 F.

Exposed to Stream of Oxygen for 17 hrs. at 2250 F.



Magnification 10 X

SECTION IV

PRODUCTION AND FABRICATION OF 9-INCH STRESS RUPTURE SPECIMENS

The production method adopted for the preparation of the 9 in. long stress rupture test bars from the infiltrated type of material consisted essentially of (1) mixing the titanium carbide powder with nickel powder sufficient to produce coherent skeleton bodies; (2) hot-pressing the mixture into 9 in. long, 1/2 in. square bar stock; (3) sintering above the melting point of the binder metal to increase the strength of the skeleton bars further; (4) machining the bars into octagonal cross section; (5) infiltration in the transverse direction; (6) finish grinding with abrasive and diamond tools.

The production method adopted for the preparation of the 9 in. stress rupture bars from the sintered type of material consisted of (1) mixing the titanium carbide powder with the metal powder ingredients corresponding to the composition of Inconel; (2) cold-pressing into 20 in. long, 5/8 in. square ingots; (3) cutting to 10 in. long sections; (4) presintering; (5) machining to 7/16 in. diameter bars; (6) sintering; (7) finish grinding with abrasive and diamond tools.

The 9 in. long infiltrated specimens with octagonal cross sections were ground to stress rupture test specimens as per specifications of the contract. The final dimensions were:

Diameter of specimens	- 0.375 in. $^{+0}_{-1/32}$
Length of specimens	- 9 in. $\pm\frac{1}{8}$
Length of test section	- 1.50 in.
Necked down radius of test section	- 4 in.
Smallest diameter of test section	- 0.250 in.

The length of the specimens was extended to 9½ in. by brazing small steel cylinders on both ends. The steel extensions were ground true with the titanium carbide bars. All grinding operations were carried out on a lathe. Fig. 17 shows two finish-ground stress rupture bars.

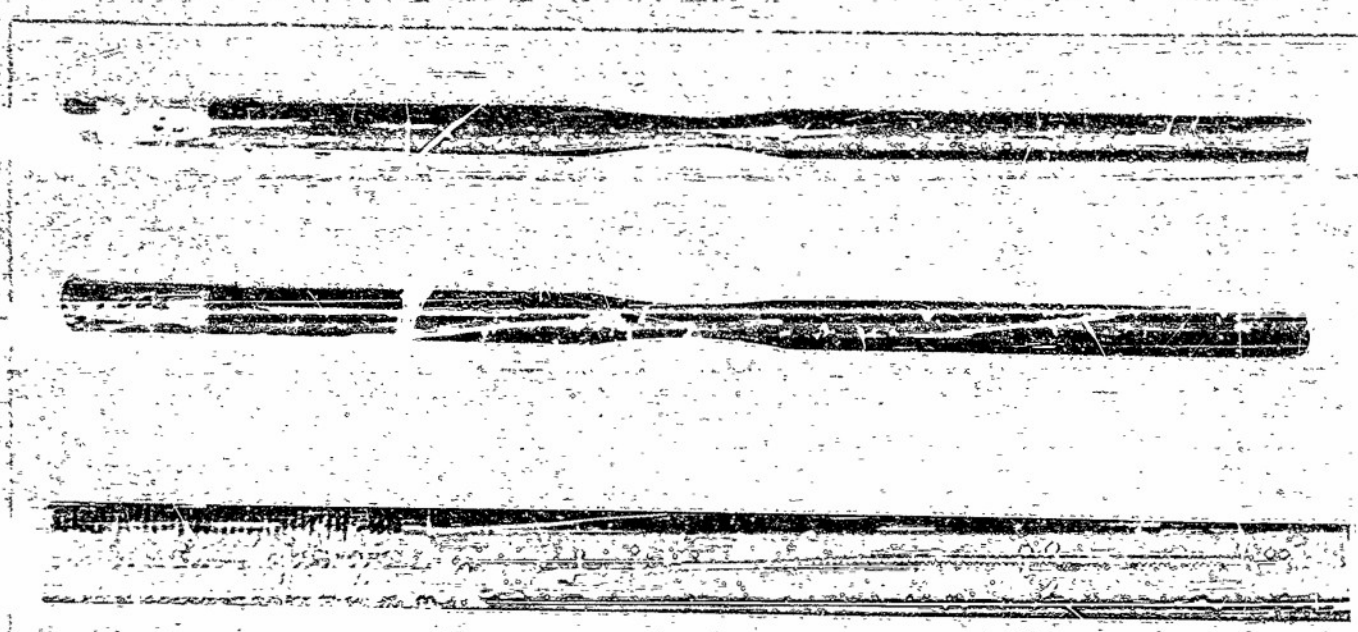


FIGURE 17

Nine-Inch Long Infiltrated Titanium Carbide Bars Completely Finish Ground as per U. S. Air Force Drawing #S49A9491.

SECTION V

THERMAL SHOCK TESTING

A. Testing Procedure

An apparatus was built for the purpose of subjecting stress rupture specimens to thermal shock. Fig. 18 shows the experimental set-up. One side of the center section of the test specimen was subjected to the impingement of a flame produced by the combustion of an oxygen-natural gas mixture in a torch. The temperature was measured by a Pt-Rh thermocouple, at the side opposite the flame impingement. After reaching the testing temperature within 20-30 seconds, the specimen was quenched to room temperature within 30-40 seconds, by directing at the sides of the specimen two streams of nitrogen gas, converging at the hot center of the bar. The test bars were, subsequent to thermal shock testing at the temperatures and for the number of cycles specified in the contract, tested for modulus of transverse rupture at room temperature.

Figs. 19 and 20 show titanium carbide stress rupture bar type specimens containing Nichrome V and Hastelloy "C", respectively, after thermal shock testing. The early and complete breakdown of the Hastelloy "C"-containing specimen due to burning is apparent; this behavior of the material was responsible for its abandonment for further tests.

B. Thermal Shock Testing Data

1. Testing at 2500°F

Modulus of transverse rupture type test bars from Inconel-infiltrated titanium carbide were subjected to successive thermal shock cycling at 2500°F. Fig. 21 shows the deterioration of the specimens at the flame impinged central portion due to melting of the infiltrant phase at this temperature. The brownish wrinkled film appearing on the side of the flame impingement after one cycle developed into large blisters and cracks on both sides of the specimen after eight cycles, which number was taken as the criterion of failure. Subsequently, test specimens were thermally shocked for 1/4, 1/2, and 3/4 of the number of testing cycles which produced failure, i.e., 2, 4, and 6, respectively, and tested for

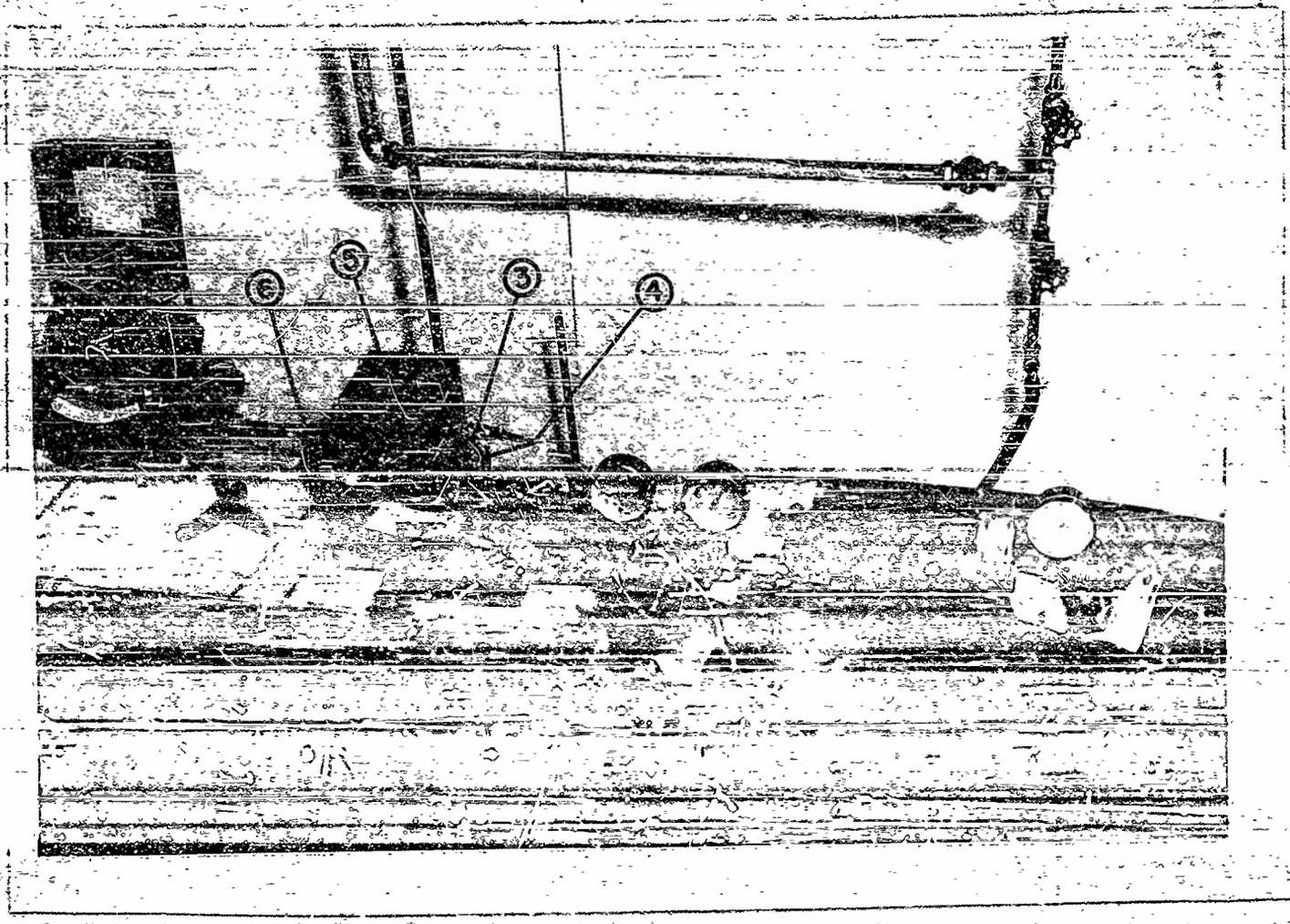


FIGURE 18

Thermal Shock Testing Set-Up

1) Specimen
2) Brick enclosure
3) Oxygen-natural gas torch
4) Nitrogen inlets
5) Exhaust
6) Thermocouple

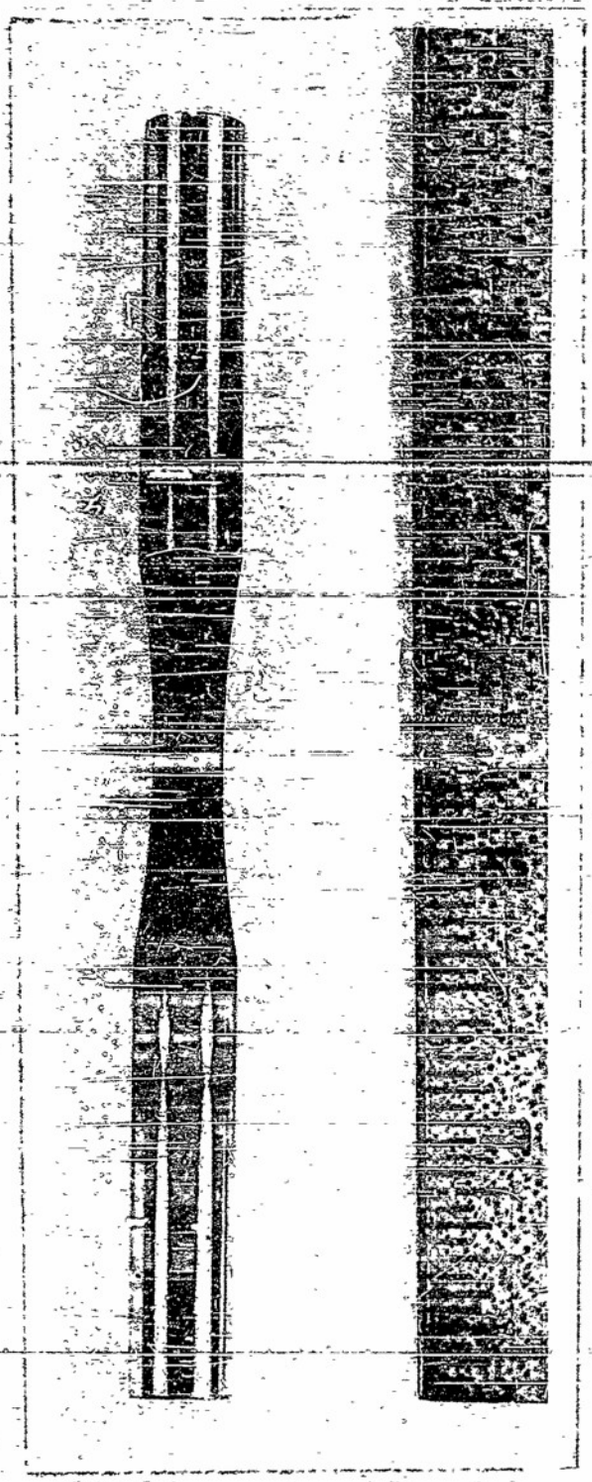


FIGURE 19

Sintered Bar of 50-40-10 TiC-Ni-Cr Mixture
After Thermal Shock Testing for 30 Cycles at 2500°F.

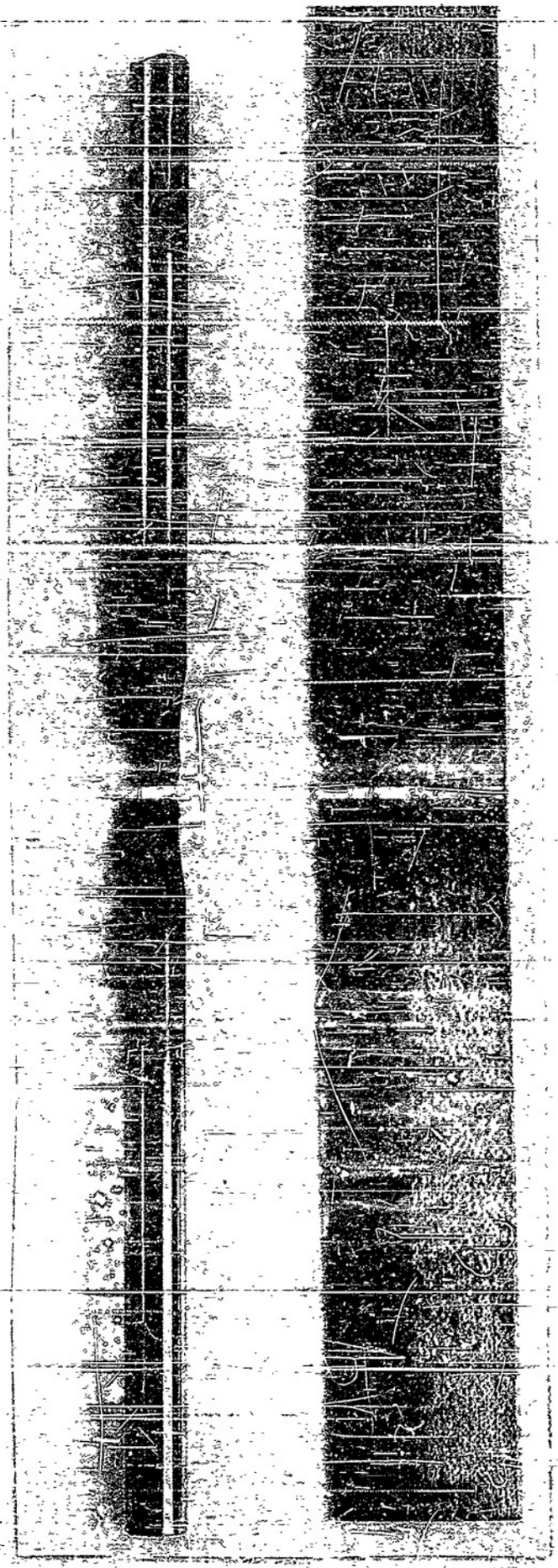


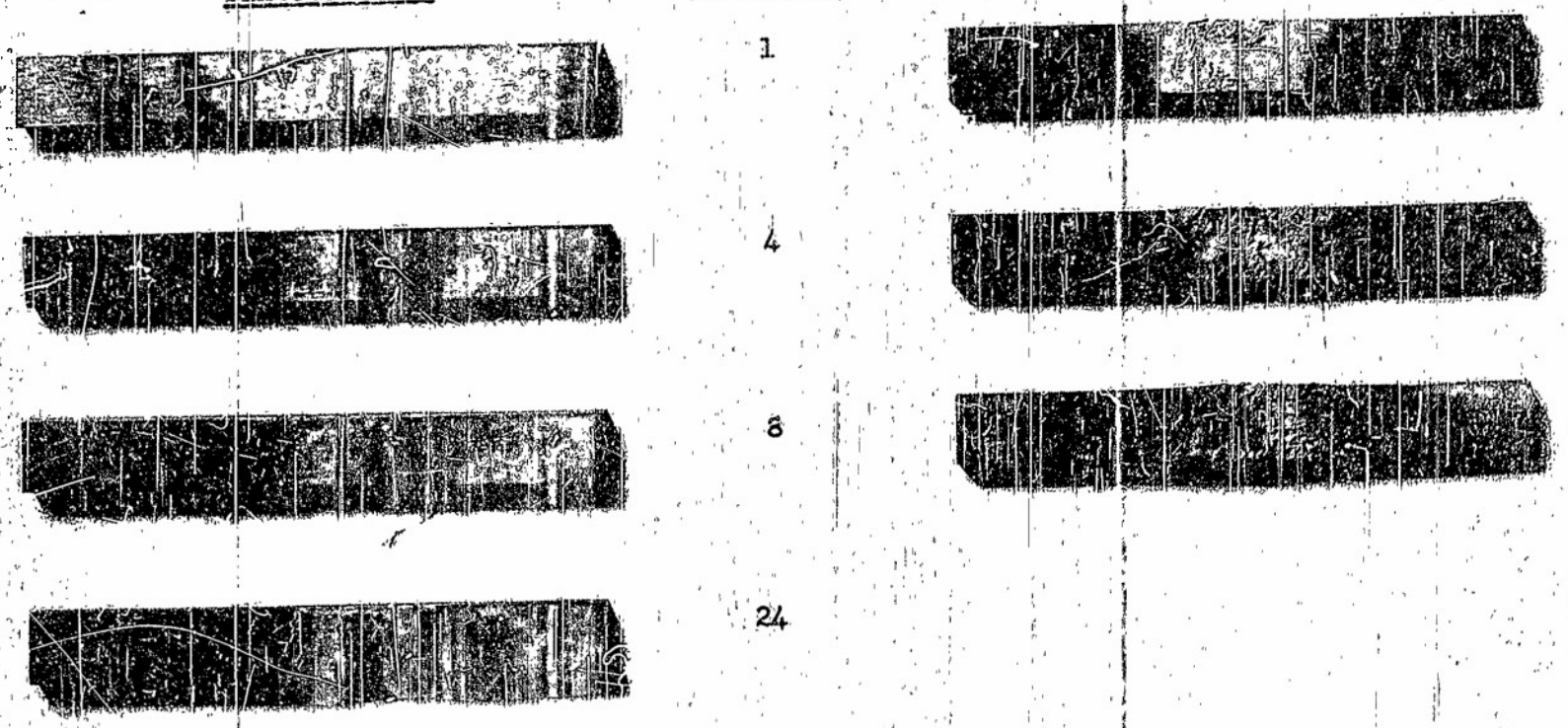
FIGURE 20

Hastelloy "C" Infiltrated TiC Bar Having the Approximate Composition 43-32-10-9-3, TiC-Ni-Mo-Cr-Fe-W After Thermal Shock Testing for 30 Cycles at 2500° F.

FIGURE 21

SURFACE APPEARANCE OF FLAME IMPINGED FACE OF SINTERED VERSUS INFILTRATED TITANIUM CARBIDE INCONEL TEST BARS AFTER VARIOUS NUMBERS OF THERMAL SHOCK CYCLES*

Sintered Bars	No. Shock Cycles	Infiltrated Bars
TNC 3105	1	TC 3005
TNC 3104	4	TC 3003
TNC 3106	8	TC 3004
TNC 3103	24	



*A thermal shock cycle consists of torch heating within 30 seconds to 2500°F. (measured on opposite side of flame impingement) and cooling within 30 seconds at room temperature by nitrogen stream.

modulus of transverse rupture at room temperature. Similar tests on sintered bars resulted in the formation of a reddish film after one cycle at 2500°F and definite erosion along the edges of the specimen after eight cycles. The failure point was taken as eight cycles.

The test results are shown for both types of materials in Fig. 21 exhibiting the condition of the specimens after different numbers of shock cycles. Table VII gives data for the modulus of rupture strength of both types of specimens before and after shocking.

The test results indicated that the testing temperature of 2500°F was too high for this type of material and specimen size (1/4 x 3/8 x 3 in). Fig. 22 shows the deterioration of the physical structure of a test bar shocked for four cycles at 2500°F. The center of the specimen, i.e., the irregularly shaped edge, shows the formation of a porous structure below the surface blister resulting from the melting of the infiltrant.

2. Testing at 2400 and 2300°F

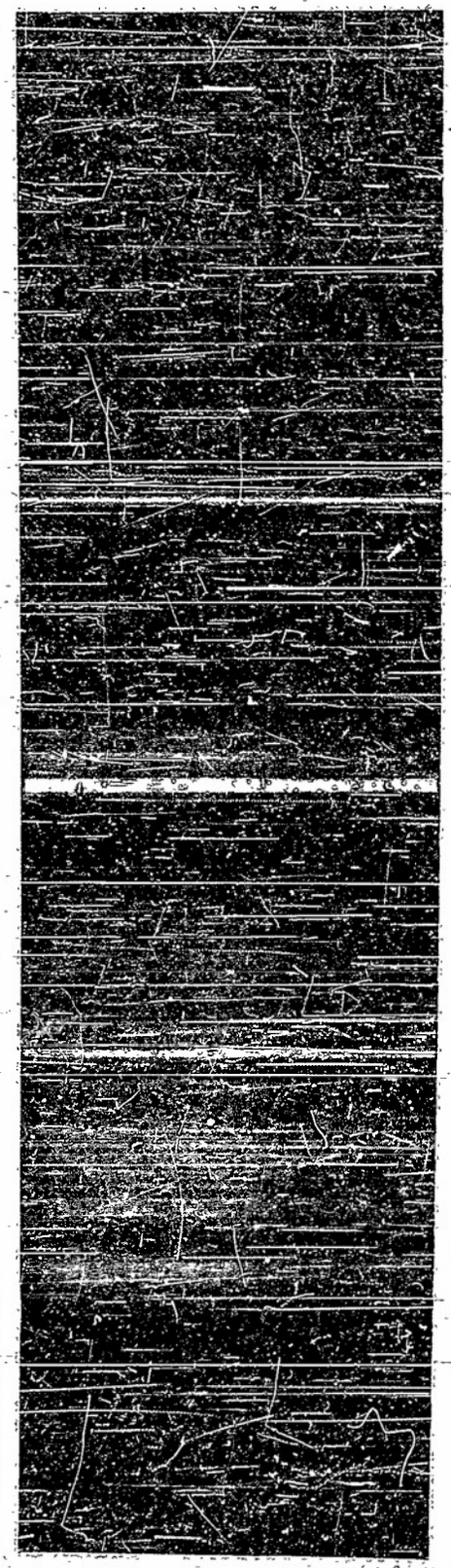
To eliminate the ill effects caused by overheating the specimen beyond the melting temperature of the infiltrant alloy phase, the thermal shocking temperature was successively lowered in 100°F steps. As can be seen from Fig. 23, a shock cycling temperature of 2400°F still resulted in incipient fusion and consequent surface wrinkling and deterioration after a single cycle.

Thermal shocking at the testing temperature of 2300°F, however, did not indicate noticeable surface deterioration until 90 cycles of testing for both the infiltrated and sintered test specimens (see Fig. 23). Definite erosion of the edges, indications of failure, did not occur until 120 testing cycles. Table VII shows the modulus of transverse rupture values for both types of specimens after thermal shocking at 2300°F, while Fig. 23 shows the surface appearance also of the infiltrated specimens after 1 and 60 shocking cycles for 2300°F, as contrasted with specimens subjected to one cycle each at 2400 and 2500°F.

TABLE VII

Modulus of Transverse Rupture at Room Temperature of Inconel-Infiltrated and Cold-Pressed and Sintered Titanium Carbide Specimens as a Function of Number of Thermal Shock Cycles

Temperature, °F	Number of Cycles of Thermal Shock	Average Modulus of Transverse Rupture at Room Temperature, psi	
		Infiltrated Bars	Sintered Bars
Room	None	192,000	92,000
2500	2	103,000	75,500
2500	4	82,000	70,000
2500	8	85,000	69,000
2300	30	171,000	86,000
2300	60	173,500	87,000
2300	90	146,000	85,000



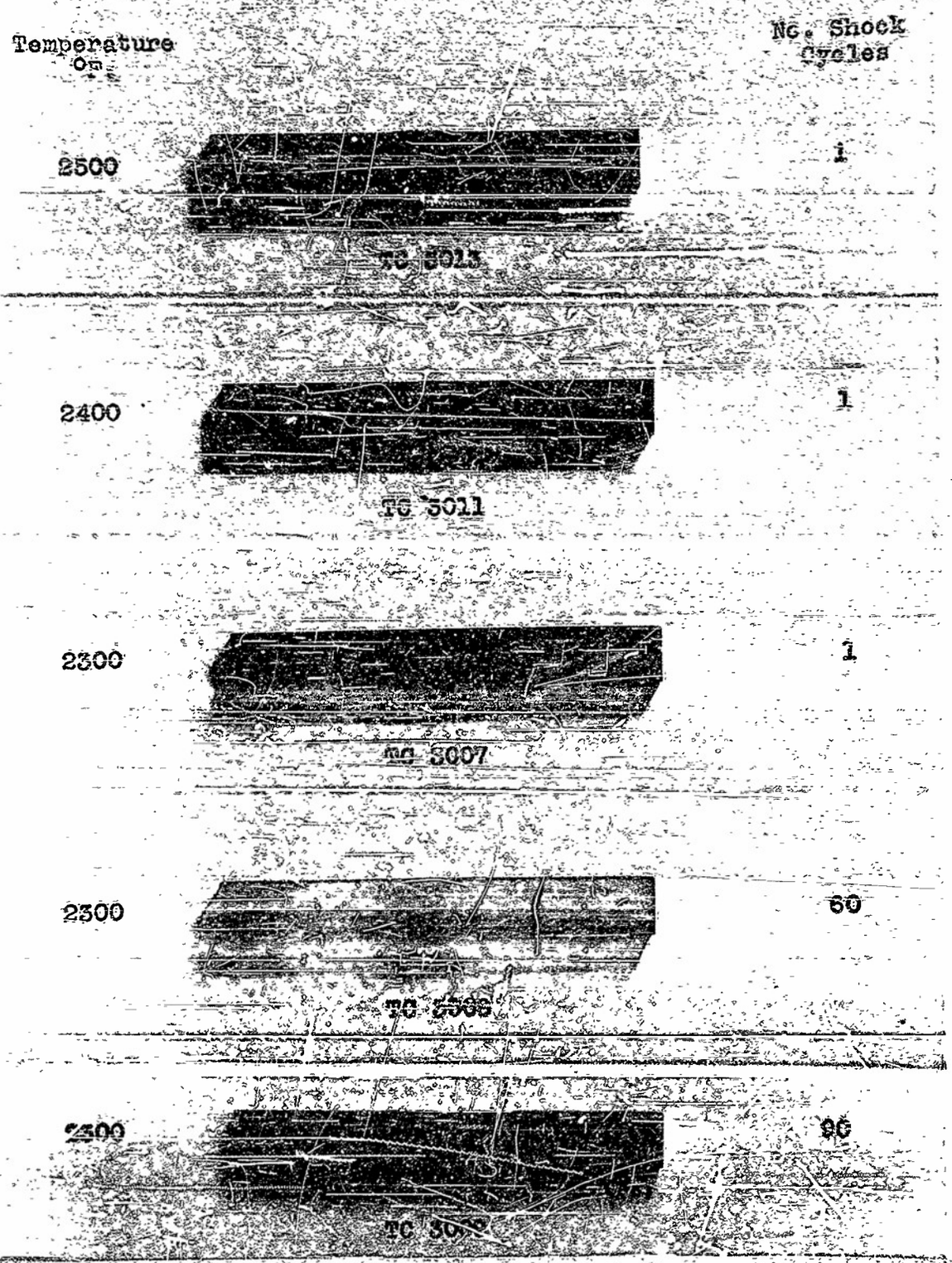
10X

FIGURE 22

Macrosection Through Infiltrated Titanium Carbide Test Bar After Thermal Shock Testing for Four Cycles at 2500°F, Showing Destruction of the Grain Structure by Fusion in Area of Flame Impingement (left).

FIGURE 23

EFFECT OF TEMPERATURE AND TIME OF THERMAL SHOCK TESTS ON INFILTRATED TITANIUM CARBIDE-INCONEL TEST BARS (FLAME-IMPINGED FACE ILLUSTRATED).



C. Discussion of Thermal Shock Testing Results

1. Testing at 2500°F

As shown in Figs. 21 and 22, the testing temperature of 2500°F was definitely too high, causing pronounced deterioration of the physical structure of the specimens, apparent even after one testing cycle.

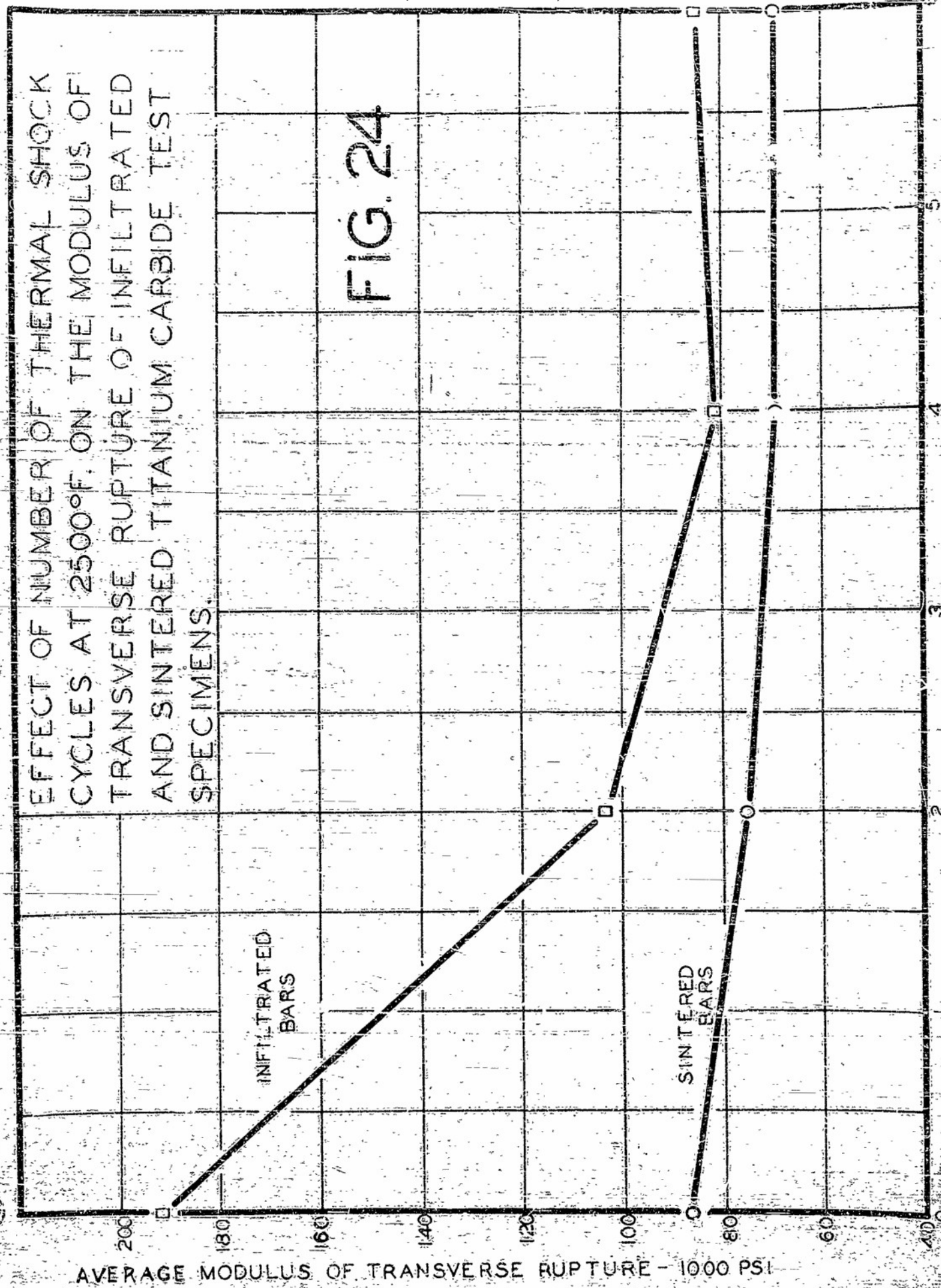
Fig. 24 shows graphically for both infiltrated and sintered specimens the effect of the number of shocking cycles at 2500°F on the modulus of transverse rupture strength at room temperature. The infiltrated specimens exhibit a sharp drop in this property (40% loss) within the first two cycles, followed by a slower reduction in strength within the next two cycles, and a slight recovery after six cycles.

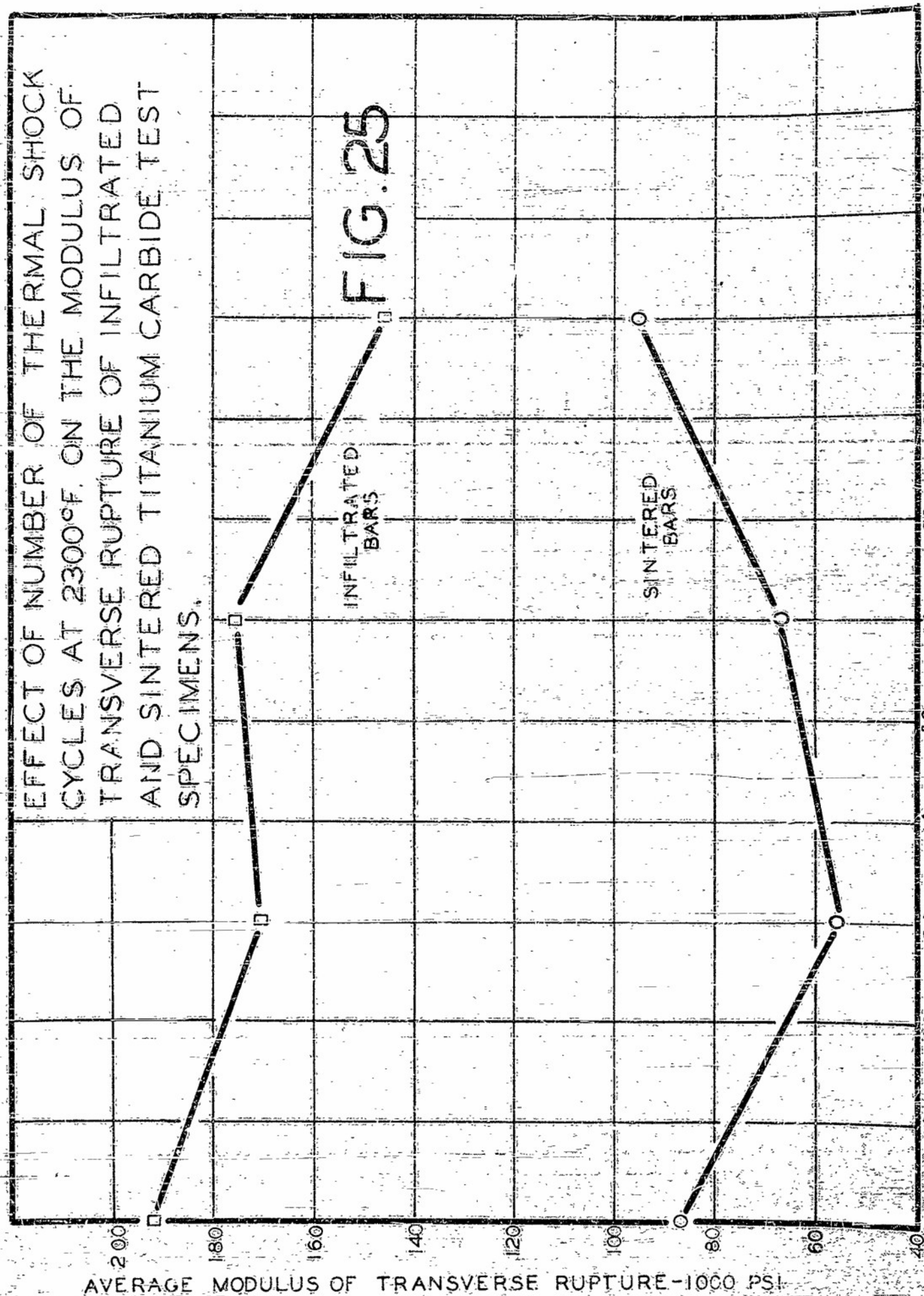
The sintered specimens, starting with an initially much lower modulus of transverse rupture (about ½) as compared to the infiltrated type, show a similar, but less pronounced, behavior. A partial recovery of the strength was only noted after eight shock cycles, when the modulus of rupture value reached 72,000 psi.

2. Testing at 2300°F

Fig. 25 shows that the infiltrated specimens undergo only a moderate loss in modulus of rupture strength (about 10%) up to 30 heat shocking cycles, i.e., much less pronounced than for the 2500°F tests. No drastic change in strength is caused by extending the thermal shock cycles from 30 to 60. After 60 cycles, loss in strength occurs anew, which now manifests itself as a definite commencement of deterioration after 90 cycles, when a loss of 25% of the initial transverse rupture strength can be noted.

The sintered test specimens, starting with strength values one-half below those of the infiltrated ones, experience an additional reduction in modulus of transverse rupture strength after 30 cycles, the values being only one-third of those recorded for the infiltrated type after the same 30 heat shock cycles. The sintered specimens recover part of their strength after the next 30 cycles, and thereafter at an accelerated rate until after 90


EFFECT OF NUMBER OF THERMAL SHOCK CYCLES AT 2500°F. ON THE MODULUS OF TRANSVERSE RUPTURE OF INFILTRATED AND SINTERED TITANIUM CARBIDE TEST SPECIMENS.
FIG. 24
INFILTRATED BARS
SINTERED BARS
NUMBER OF THERMAL SHOCK CYCLES
0
1
2
3
4
5
AVERAGE MODULUS OF TRANSVERSE RUPTURE - 1000 PSI
200
180
160
140
120
100
80
60
40


EFFECT OF NUMBER OF THERMAL SHOCK CYCLES AT 2300°F. ON THE MODULUS OF TRANSVERSE RUPTURE OF INFILTRATED AND SINTERED TITANIUM CARBIDE TEST SPECIMENS.
FIG. 25
INFILTRATED BARS
SINTERED BARS
NUMBER OF THERMAL SHOCK CYCLES
0
10
20
30
40
50
60
70
80
90
100
AVERAGE MODULUS OF TRANSVERSE RUPTURE-1000 PSI
40
60
80
100
120
140
160
180
200

cycles, the initial level of modulus of rupture strength values is regained.

The strength versus number of thermal shock cycles curves exhibit two obvious trends. One is that the strength of both, infiltrated and cold-pressed and sintered types of titanium carbide materials, is affected by the number of thermal shock cycles in approximately the same manner. The apparent initial reduction and subsequent recovery of the strength after progressive thermal shock cycling, is more advanced in the case of the sintered titanium carbide composition, and more retarded, or not at all developed within the number of cycles used for the test series, in the case of the infiltrated material. The trend toward recovery in both cases, however, is most unexpected. It indicates that no direct relation exists between surface deterioration and modulus of transverse rupture strength as long as the structure is not destroyed by fusion under oxidizing conditions.

The other result of significance is that the Inconel-infiltrated type of titanium carbide material yields, under all conditions, modulus of rupture strength values far superior to those obtained under identical conditions for sintered titanium carbide specimens of comparable composition, as produced by the particular method used in this investigation. This superiority is most significant in specimens that were not impaired by overheating.

SECTION VI

SUBMISSION OF SPECIMENS

The specimens submitted, in compliance with the contract stipulations, included:

twelve (12)	finish-ground, nine in. long, stress rupture specimens from titanium carbide infiltrated with Inconel;
three (3)	finish-ground, nine in. long, stress rupture specimens, cold-pressed and sintered titanium carbide-Inconel.

SECTION VII

SUMMARY AND CONCLUSIONS

1. Titanium carbide-base composite bodies were produced by infiltration with nickel-base alloys and a nickel-aluminum compound, as well as by conventional powder metallurgical methods.

2. Among the various methods of infiltration attempted, the transverse capillary infiltration method proved the most favorable for the long and slender type of stress rupture test specimen stipulated by the contract specifications.

3. Among the infiltrants tested, Nichrome V, Hastelloy "C", Inconel, and the intermetallic compound NiAl, Hastelloy "C" proved superior in ease of infiltration.

4. Specimens of compositions similar to those of the infiltrated bars were also produced by cold-pressing and sintering techniques. Hot-pressed specimens proved considerably inferior to cold-pressed and sintered specimens. In general, specimens produced by conventional powder metallurgical techniques were found to have physical and chemical properties which were inferior to those of the infiltrated specimens.

5. The tests conducted at this laboratory were confined to modulus of transverse rupture testing at room temperature and 1800°F, deflection in bending under the test load at 1800°F, oxidation in still air at 1600, 1800 and 2000°F, and in an oxygen stream at 2250°F, and thermal shock cycling at 2300 and 2500°F.

6. Inconel-infiltrated titanium carbide bodies were found to have the highest modulus of transverse rupture values and best oxidation resistance. They also showed good ductility, as manifested by pronounced bending under load, at 1800°F. This material was therefore selected for the stress rupture and thermal shock test specimens.

7. The number of specimens submitted to the Materials Laboratory, Research Division, Wright Air Development Center for stress rupture testing were limited, due to difficulties in production, to twelve, nine in. long, Inconel-infiltrated, finish-ground, titanium carbide specimens and three specimens similar in composition, produced by cold-pressing and sintering.

8. While it was possible to produce by the infiltration technique three in. long specimens which showed great uniformity in structure and properties, it was not possible to achieve the same degree of uniformity in nine in. long bars.

9. An X-ray investigation of the oxide scale of oxidized Inconel-infiltrated titanium carbide specimens was carried out, and the composition of the oxide product determined to be primarily ferric oxide, nickel oxide, and titanium dioxide.

10. The coefficient of thermal expansion of the Inconel-infiltrated titanium carbide was found to be 4.44×10^{-6} per °F in the range of 70 to 1200°F.

11. It was not possible to obtain the coefficient of thermal expansion of the oxide scale since it was found not to be feasible to produce test specimens by any of the available methods.

12. Comparison showed a clear-cut superiority of about 2 to 1 of infiltrated to cold-pressed and sintered material in modulus of transverse rupture and thermal shock testing properties; this is probably due, at least in part, to the higher density of the infiltrated type of material, but may also be partly due to the continuity of the binder (infiltrant) phase.

13. The effect of thermal shock, below the melting point of the infiltrant, on infiltrated specimens, shows an approximately 25% decrease in the transverse rupture strength of the material after prolonged shock cycling.

14. Thermal shocking of material made by cold-pressing and sintering caused a 35% initial drop in transverse rupture strength followed by a partial recuperation after continued shocking at 2300°F. At temperatures above 2300°F only minor recuperation of strength after continued shocking was observed.

15. The encouraging test results in modulus of transverse rupture, thermal shock, and oxidation testing of Inconel-infiltrated titanium carbide and similar materials suggest that these materials may be well suited for use as structural materials in high temperature engine components. It is necessary, however, to carry out further development work with the object of producing such composite bodies free of microporosity, surface erosion effects, etc. The accomplishment of a completely uniform microstructure would undoubtedly result in further improvement in the physical and chemical properties of such infiltrated titanium carbide material.

16. The utilization of this improved type of material for power plant components depends on further engineering and development work relative to methods of shape generation, infiltration and finishing.

REFERENCES

1. Air Force Contract AF 33(038)-6841; Report No. 8, Final Summary Report, October 31, 1950.

2. Air Force Contract W 33-038 ac-19710 (19354); A. F. Technical Report 5892, May 1949.

3. J. C. Redmont and E. N. Smith, Trans. Am. Inst. Mining Met. Engrs., 185, 987 (1949).

4. Air Force Contract AF 33(038)-16911, Bimonthly Report No. 2, Section III, May 1, 1951.

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