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TITANIUM CARBIDE-NICKEL CERMETS: PROCESSING AND JOINING

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MARCH 1952

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Statement A
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WRIGHT AIR DEVELOPMENT CENTER

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Massachusetts Institute of Technology

March 1952

Power Plant Laboratory
Contract No. AF 33(038)-8879
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Executive [illegible]
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Wright Air Development Center
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United States Air Force
Wright-Patterson Air Force Base, Ohio

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FOREWORD

This report was prepared by John Wulff, James E. Cline, Amos J. Shaler, Malcolm Basche, Romeo G. Bourdeau, and Edward R. Stover at the Massachusetts Institute of Technology. The work was accomplished under Contract No. AF33(038)-8879 which is identified with Expenditure Order No. R-506-67, Ceramic Components for Aircraft Power Plants. Presented herein are the results of experiments involving the processing of titanium carbide-nickel cermets, the diffusion welding of these cermets to alloy metals, and a study of phase relationships and properties in the titanium-carbon-nickel system. The technical phases of the contract are administered by the Power Plant Laboratory at the Wright Air Development Center, with Mr. B. L. Paris acting as Project Engineer and 2/Lt. C. K. Russell as Asst. Project Engineer.

ABSTRACT

Titanium carbide-nickel cermets can be sintered to have transverse rupture strengths over 250,000 pounds per square inch. To do so, four principal processing variables must be controlled: the powder must be as free as possible from oxygen and nitrogen; the powder must be milled in carbon tetrachloride and pressed without intermediate drying; sintering must be done in vacuo or possibly in an inert gas, but not in hydrogen; heating-up must be slow enough to allow thorough degassing.

Joining titanium-carbide cermets to high-temperature alloys has been accomplished by vacuum diffusion, and gives a weld stronger than the cermet.

Fabrication of cermet articles of graded composition has been successfully accomplished by fragmentation-forming, i.e. cold forging the powder in a ductile envelope and subsequently sintering.

PUBLICATION REVIEW

FOR THE COMMANDING GENERAL:

NORMAN C. APPOID
Colonel, USAF
Chief, Power Plant Laboratory
Aeronautics Division

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I. INTRODUCTION

The research described in this report has had as its main purpose the determination of the proper processing techniques necessary to achieve optimum room temperature properties in titanium carbide-nickel compositions. Various titanium carbide and nickel powders available on the open market were employed as starting material. Ball milling, mixing and pressing procedures were the subject of the first phase of the study. This was followed by an investigation of sintering procedures and of testing techniques. In order to determine proper sintering temperatures, thermal, magnetic and X-ray diffraction experiments were made to ascertain phase relationships in and near the TiC-Ni section of the ternary diagram nickel-titanium carbon. Related to this research were auxiliary studies on joining titanium carbide cermets to such heat-resistant alloys as vitallium and 18:8 stainless steel. The fabrication of graded composition to achieve better physical properties in the root sections of turbine blades was also attempted. Results obtained indicate that titanium carbide-nickel cermets of substantially greater strength than has been heretofore reported can be made. Further work on joining and on graded compositions is worthwhile, in our opinion.

II. EXPERIMENTAL APPARATUS AND PROCESSING TECHNIQUES

A. Powder Selection

Various titanium carbide powders are available in the United States. The type which is perhaps at present most reliable is made from a high frequency melt of iron, titanium and carbon, crushed and leached to separate iron from the cold powder. A second kind of powder is made by leaching electric-arc-furnace high-carbon ferro-titanium. A third type is made by arc-melting titanium sponge and carbon. Still another powder comes from the reduction and carburization of titanium dioxide in a carbon-tube resistance furnace at a temperature of 1900-2000°C under hydrogen or helium. All these powders possess shortcomings, for titanium carbide is a highly reactive material. It is extremely difficult to manufacture a carbide of 20% carbon content, corresponding to the formula TiC. A carbon content of 17.5 - 19.5 is usually obtained, or else the carbon exceeds 20% and some free graphite is present.

Most of the titanium carbide made at present contains some oxygen and nitrogen atoms which replace carbon atoms in the TiC structure. Neither the subject matter nor the financial support of this research permitted much experimentation towards the development of a carbide fabrication method which would give a carbide of stoichiometric composition free of graphite, oxygen, nitrogen and other impurities. Although the importance of such an endeavor was not underestimated, we soon came to the decision to purchase carbide powder on the open market. The powder which gave us the optimum mechanical properties in the sintered state was made by adding titanium dioxide to a high-temperature high-carbon-iron melt, subsequently crushed and leached. The analysis of such powders yielded the range of compositions 79.22% titanium, 19.66 total carbon, 0.60 free carbon, 0.01-0.5 chromium, vanadium and iron; there were spectroscopic traces of many other metals. To be sure this was not pure titanium carbide, yet it contained less oxygen, nitrogen, and free carbon than other powders that we were able to purchase; above all, its composition varied least from lot to lot, so that we could be reasonably sure of obtaining reproducible results with it.

Nickel powders investigated for use as binder phase were electrolytic powder, reduced oxide powder, and reduced formate powder, all available on the open market. Crushed electrolytic powder pre-annealed in hydrogen was finally selected. It contained 98.43% combined nickel and cobalt.

B. Ball Milling

In carbide powder-metallurgy it is common practice to ball mill the carbide with the binder material. Sometimes ball milling to reduce powder size precedes milling with binder. Ball milling practice varies from plant to plant not only in the kind of balls and mill lining used but also in the vehicle employed. Some mills are evacuated, others are partly filled with alcohol or carbon tetrachloride. In this research we found that the only milling technique that can be trusted involves using carbon tetrachloride. The mill we used was lined with austenitic stainless steel and the balls were made of martensitic stainless steel or tungsten carbide. Milling was continued until all the powder was finer than 10 microns. The operation lasted from 24 to 72 hours depending on the charge.

C. Pressing

In preliminary sintering experiments with pressed compacts, the use of such lubricants as carbowax or camphor to aid pressing gave such deleterious results that these materials were abandoned. Likewise, dyring the powder after ball-milling in carbon-tetrachloride proved less efficacious than merely decanting the liquid and pressing the powder wet. Compacts pressed from wet powder from one side had cracks which did not heal in sintering. Consistently good results were obtained by pressing from both sides. Low-nickel compositions were best pressed under 10,000 pounds per square inch; high-nickel compositions (over 30%) were most satisfactory at a maximum of 30,000 pounds per square inch. For complicated shapes not reported in this paper, hydrostatic pressing leads to a more uniform distribution of density.

D. Fragmentation Forming

Since one of the purposes of this investigation was to form shapes similar to turbine blades but of composition varying from tip to root, a process developed by one of us elsewhere for making cigarette lighter flints and other articles was employed. The process has been termed fragmentation forming. It is based on the fact that brittle powders, or mixtures of brittle and ductile powders, can be compacted in an envelope of ductile metal such as iron, nickel, or stainless steel by repeated impact. Densities ranging from 80 to 98% of theoretical density are obtained, whereas with customary pressing methods the maximum densification is about 50%. The fragmentation-forming technique was applied to the present work, using nickel and stainless steel envelopes. These were filled with titanium-carbide-rich mixes increasing in nickel content toward one end and sealed with a force-fitted plug. The tubes were then cold-forged to shape, and sintered. The envelope was either removed before sintering or allowed to infiltrate the compact. By this technique a density of 93% of theoretical was achieved during pressing. Sintering and infiltration raised the density to 100% of theoretical.

E. Sintering Atmospheres and Temperatures

Experiments in which compacts pressed in the conventional way were sintered in hydrogen, purified helium, or vacuum indicated that sintering at a pressure of 10^{-4} mm, or less gave material of maximum mechanical strength at minimum sintering temperatures and times. In all our sintering studies a minimum temperature of 1310°C for very high nickel compositions and a maximum sintering temperature of 1400°C for low nickel compositions were employed. Higher sintering temperatures for compacts containing less than 10% nickel are no doubt advisable, but in employing such temperatures one always comes up against the hazard of a reaction between the carbide and its support. Among the latter, zirconia, graphite and metallic carbides were tried out. The first of these leads to gas formation, the second to graphite transport, and the third to defiltration. Lower temperatures of sintering in all cases reduce deleterious effects of this kind.

Sintering times employed in this research ranged from 15 minutes to 4 hours at optimum temperature and pressure. Additional time was used to bring the compacts slowly to the sintering temperature in order to preserve at all times a pressure of 5×10^{-4} mm. or less. The temperature was raised only as fast as residual gases were removed. Copious gas evolution always took place at about 1310°C. This necessitated a dwell of at least 10 minutes at this temperature; for compacts made of inferior quality powder the dwell lasted sometimes over one hour. In later sections of this report, the temperature 1310°C is shown to be just above the temperature at which the binder is completely melted.

F. Diffusion Joining

In vacuum-sintering studies, using various substrates to support the carbide compact, it was found that welding of the compact to the substrate often took place. Since some results had been reported to us by the Air Materiel Command concerning the brittleness of the root sections of titanium carbide cermet blades, it was decided to attempt vacuum diffusion welding of cast high-temperature alloy roots to TiC-Ni cermet blade sections. Using 18:8 stainless steel and vitallium roots, it was found necessary to insure mechanical mating of abutting members by first assuring good fits and then using slight pressure. In a vacuum of 5×10^{-4} mm. or better and at minimum temperatures of 1300°C (for 18:8) and 1290°C (for vitallium) it was found possible to make strong and ductile joints. Figure 1 indicates in that test the alloy becomes deformed whereas the cermet and the joint do not.

In diffusion welding of the kind described here, the cermet takes up metal from the root. A graded joint is thus formed. If diffusion is allowed to proceed for any appreciable time, the cermet swells by infiltration (Figure 1).

III. PHASE RELATIONSHIPS AND PROPERTIES

A. Physical Properties

Data obtained throughout this research on the transverse rupture strength and density of titanium-carbide nickel cermets as a function of nickel content is plotted in Figure 2. All the results obtained for vacuum sintering are included; they represent a wide variety of techniques. The scatter of results is primarily due to controlled variations in compaction pressure, in heating-up rate, and in sintering temperature, time, and residual gas pressure.

Maintenance of optimum properties requires control of size and purity of powder before pressing. Powders containing appreciable amounts of oxygen, nitrogen, or hydrogen give lower values of transverse rupture strength than those reported here. All transverse test specimens used in obtaining these results had the dimensions 1/8" x 1/8" x 1". Because the highest transverse rupture strength measured (260,000 pounds per square inch) is well beyond that reported elsewhere in the literature, various other sizes of specimens were tested. No size effect was observed. As a double check, commercial bar stock was purchased and ground to 1/8" size. The values found for this bar were equal to those reported by the maker for 1/4" test piece from the same lot.

The maximum transverse strength was found for compositions in the region 20-40% nickel. Beyond 50% nickel the transverse rupture strength falls rapidly; in such specimens, elastic deformation before fracture was observable, and for nickel contents exceeding 70%, some elastic deformation occurred before fracture.

A second curve in Figure 2 indicates the change of density with nickel content. The hardness and density of the 10% nickel compositions is lower than that reported by other workers.[1,2] The presence of porosity in these compacts may be attributed to the low sintering temperature (1400°C) employed. On the other hand the transverse rupture strength is higher than that reported by others.[1,2]

Figure 3 indicates the particle size of the carbides, the shape of the grains, and the nature of the network in compositions containing 21% to 51.7% nickel at a magnification of 1500 x. For comparison, a photomicrograph of an arc cast cermet containing 20% nickel is also shown. In all specimens, careful polishing and etching reveal the presence of a faint precipitate in the binder. In cast material of similar composition a coarser precipitate of titanium carbide particles is observed in some regions of the binder.

B. Binder Phase Relationships

In order to provide a background for proper compounding and sintering of titanium carbide cermets, work was begun on the nickel-titanium-carbon ternary diagram. Cooling and heating curves were obtained from compacted powder mixtures induction-melted in zirconia under argon. Metallographic, magnetic and X-ray studies were made of powder-metallurgy and arc-cast materials. The TiC end of the system has not been completely examined, but the data obtained on high-purity nickel-rich compositions warrant a preliminary description of the vertical ternary sections at and near the nickel-titanium carbide plane.

Three of these sections are shown in Figure 4. The dotted lines represent more tentative conclusions than the solid ones. The top diagram of Figure 4 is a section running from pure nickel to stoichiometric titanium carbide (20 atomic per cent carbon). Some of the carbon from the titanium carbide always occurs as free graphite if the carbide has stoichiometric composition. A ternary eutectic between this graphite, the alpha phase, and titanium carbide occurs at 1270 ± 4°C. Commercial titanium carbide also contains oxygen and nitrogen, which displace carbon from interstitial solution in titanium carbide (their solubility in the carbide becomes greater with increasing pressure and decreasing temperature). If cermets are sintered under conditions such that this oxygen and nitrogen remain in the alloy, free graphite may be precipitated from the binder during cooling. Figure 4 thus shows the phase fields present in contaminated cermets. The solidus is probably also lowered by the contaminating elements in solution. When oxygen and nitrogen are present in sufficiently large amounts, new phases appear, characterized by dark gray and rose colors in the cermet microstructure.

The best cermets are obtained by sintering in the highest possible vacuum. During such sintering, the gaseous contaminates are removed, and decarburization occurs by the formation of carbon monoxide. The middle section of Figure 4 is representative of cermet compositions decarburized to a certain extent by vacuum sintering. The solidus ranges between 1270°C and 1307 ± 4°C. At 1307°C, a eutectic reaction occurs between titanium carbide and an alpha-solution containing 0.24 weight per cent carbon and 8.4 weight per cent titanium. The lower section in Figure 4 indicates the cermet structures present when the pure titanium carbide is still more deficient in carbon than in the preceding diagram. The lamellar compound $TiNi_3$ is not readily precipitated from alpha solid-solutions and has not been identified in cermet compositions.

The equilibrium diagrams of Figure 4 show several features of interest in cermet manufacture. First, binder solidification occurs over a range of temperatures; it is possible, as a general rule, to sinter at a temperature at which only a part of the binder is molten. The best temperature (between 1270 and 1307°C) accordingly varies with alloy content and sintering conditions.

Second, the tendency for graphite precipitation increases with increasing binder content (although such precipitation may be reduced by rapid cooling after sintering). Third, the addition, in small amounts, of transition-element alloying agents may produce a slight increase in the freezing range of the binder; such an addition also lowers the carbon content and the solidus of the system.

The diagram does not show one important feature of the commercial titanium-carbide cermets: the finely dispersed structure (resembling a precipitate) often found in the binder. Since no unusual phase exists above 1200°C in these compositions, any such precipitate must be TiC, $TiNi_3$, graphite, or a mixture of these. It may be formed as a result of the stresses due to the differences in thermal contraction of the binder and the surrounding TiC on cooling. The fact that the Curie point of a vacuum-sintered cermet is about the same as that of the nickel powder used in its manufacture indicates that such stress-induced precipitation may have resulted in a binder composition corresponding to a very low temperature on the equilibrium diagram.

IV. DISCUSSION AND CONCLUSIONS

Our work thus far with titanium carbide nickel cermets has impressed us with the fact that the transverse rupture strength is in the first instance dependent on the quality of the carbide powder used. Here minimum nitrogen and oxygen content are of prime importance. Unless carbide formers are added to the nickel matrix, excess free graphite can also lower the transverse rupture strength.

Lack of control of other fabricating variables, such as ball milling vehicle and the state of the powder during pressing, can lead to oxidation and otherwise suitable powders. Oxidation can be avoided by milling in carbon tetrachloride followed by wet pressing. Active adsorption of the tetrachloride appears to take place and to prevent subsequent oxidation of the micron-size particles.

Sintering temperatures need not exceed 1400°C for cermets containing more than 20% nickel, and times need not exceed 4 hours; excellent properties can, however, be achieved by vacuum sintering at lower temperatures above the solidus and in shorter times. The heating-up period must be long enough to allow thorough de-gassing. Hydrogen sintering we have found extremely harmful, but helium or argon sintering cannot be ruled out as a practical method.

Diffusion joining of titanium carbide-nickel (20%) cermets to austenitic stainless steel and vitallium appears possible at temperatures of about 1300°C in vacua of 5×10^{-4} mm. or better. It is rapid and gives a ductile joint of graded composition. Fracture occurs in the cermet beyond the weld. Grain-growth in the cast or wrought material at 1300°C can become quite severe. Its effect, if any, needs still to be ascertained. If such grain-growth is harmful, it is intended in future research to experiment with manganese-nickel and nickel-chromium-boron brazing powders. In vacuum-brazing these show promise of success at temperatures some 200°C lower.

The apparent success of vacuum diffusion joining has led us to temporarily discontinue our work on the fabrication of cermets of graded composition. But in preliminary work, it was found possible to make a blade of high titanium carbide content (80%) at the tip and of lower content (20%) at the root. A suitable method for so doing involves cold-forging powder contained in a ductile metal envelope. The envelope may be removed after forging. It may also be left on and allowed during sintering to infiltrate the compact; allowances must then be made for it in the composition of the original fill.

The plot of transverse rupture strength versus nickel content shown in Figure 2 indicates that optimum properties are obtained for binder contents from 20 to 40%. From a curve published by Kennametal[2] and data by others[1], it was found that for lower strengths even 50% Ni binder is perhaps tolerable. Such a binder content brings the material almost into the vacuum-casting range. The grain size of cast material, however, is excessively high. Transverse rupture tests are now being made on such material.

The TiC-Ni sections of the ternary diagram Ni-Ti-C presented in Figure 4, although not completed in all details should serve as a guide to workers in this field. The different sections indicate what trends might be expected for different impurity content of the carbide. They also show the effect of the presence of excess graphite and of carbide formers. How the diagram is changed by alloying additions such as chromium carbide or chromium is at present being worked on from a metallographic as well as a strength standpoint.

Finally, it must be emphasized again that, as we learned in this research, good housekeeping is even more important in the titanium carbide-nickel cermet field than in the fabrication of cemented tungsten carbide.

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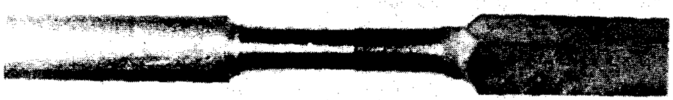
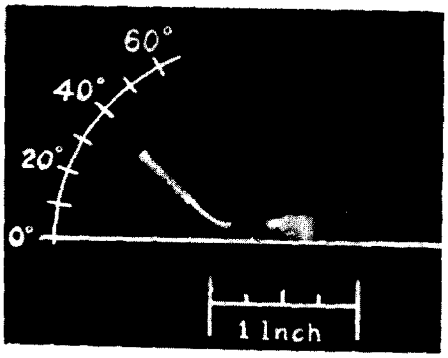
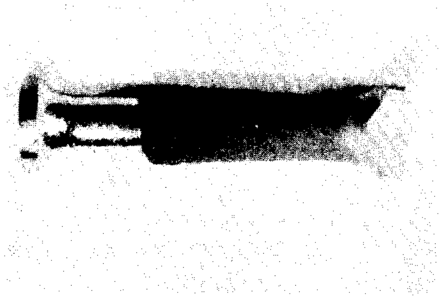




CARBIDE: 20% NICKEL - 80% TITANIUM CARBIDE (RIGHT)
ALLOY: 18-8 STAINLESS STEEL (LEFT)
WELDING TECHNIQUE: 1310 C FOR ONE HOUR
TRANSVERSE RUPTURE STRENGTH: 183,200 PSI
FRACTURE IN CARBIDE



CARBIDE: 20% NICKEL - 80% TITANIUM CARBIDE (RIGHT)
ALLOY: 18-8 STAINLESS STEEL (LEFT)
WELDING TECHNIQUE: 1400 C FOR ONE HOUR
STAINLESS SHRINKS; CARBIDE GROWS

FIGURE I TOP: PHOTOGRAPH OF JOINT BETWEEN CERMET AND HIGH TEMPERATURE ALLOY AFTER BEND TEST.
BOTTOM: SPECIMEN WELDED AT HIGH TEMPERATURE SHOWING EXPANSION OF CERMET.
PHOTOGRAPHS REDUCED APPROXIMATELY 2X FOR REPRODUCTION.

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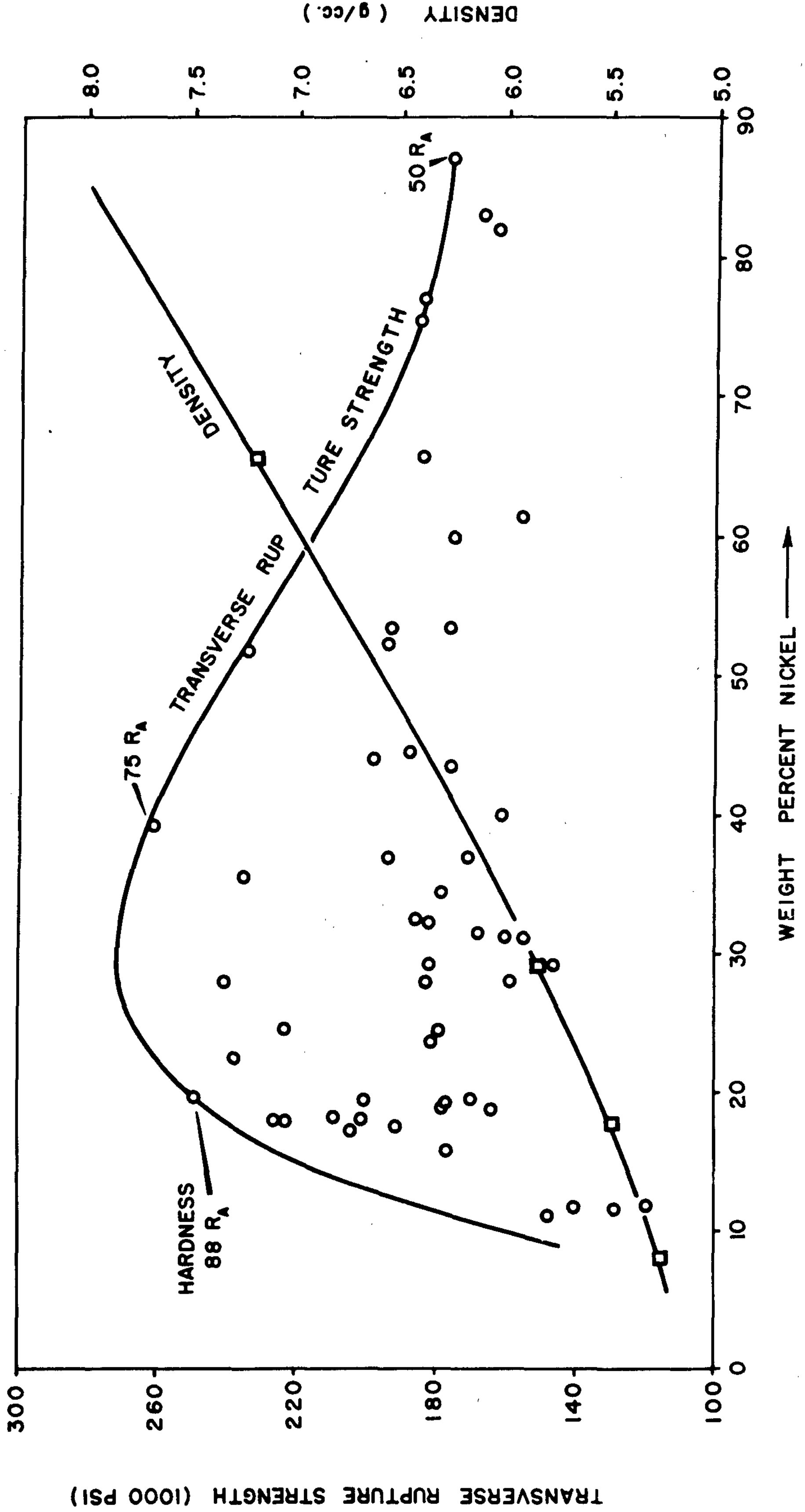
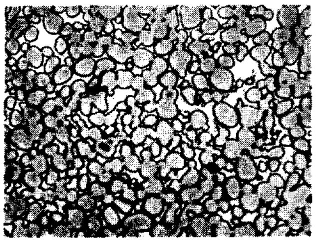
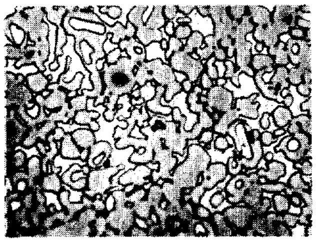


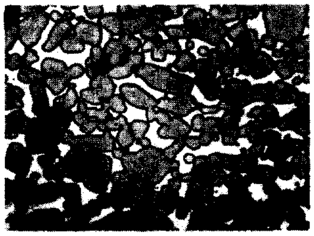
FIGURE 2. TRANSVERSE RUPTURE STRENGTH AND DENSITY VS. WEIGHT PERCENT NICKEL.



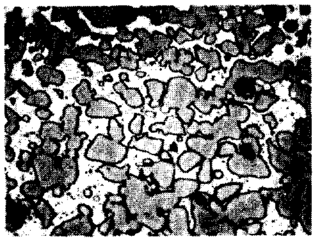
21% NICKEL ; R_A : 85 ; DENSITY : 5.59
TRANSVERSE RUPTURE STRENGTH : 249,600 PSI
HEAT UP AND DEGASSING TIME : 3 1/4 HOURS
SINTERING SCHEDULE : 1350 C FOR 4 HOURS



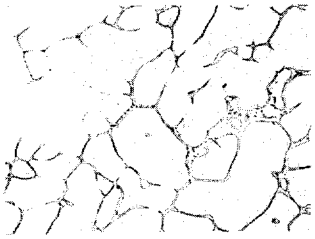
28% NICKEL ; R_A : 82 ; DENSITY : 5.79
TRANSVERSE RUPTURE STRENGTH : 241,000 PSI
HEAT UP AND DEGASSING TIME : 2 1/2 HOURS
SINTERING SCHEDULE : 1500 C FOR 1 MIN
1250 C FOR 1 HR



39.4 % NICKEL ; R_A : 75 ; DENSITY : 6.175
TRANSVERSE RUPTURE STRENGTH : 262,500 PSI
HEAT UP AND DEGASSING TIME : 3 HOURS
SINTERING SCHEDULE : 1320 C FOR 10 MIN
1280 C FOR 20 MIN



51.7% NICKEL ; R_A : 59 ; DENSITY : 6.645
TRANSVERSE RUPTURE STRENGTH : 234,000 PSI
HEAT UP AND DEGASSING TIME : 3 1/6 HOURS
SINTERING SCHEDULE : 1320 C FOR 15 MIN
1280 C FOR 2 HRS



ARC-CAST SPECIMEN ; 20% NICKEL. 300 X

FIGURE 3 TOP: PHOTOMICROGRAPHS OF SINTERED TITANIUM CARBIDE-NICKEL CERMETS. 1500 X
BOTTOM: ARC-CAST SPECIMEN; 20% NICKEL. 300 X
ETCH: 5% $FeCl_3$, 2% HCl, 93% METHYL ALCOHOL.
PHOTOGRAPHS REDUCED APPROXIMATELY 2X FOR REPRODUCTION.

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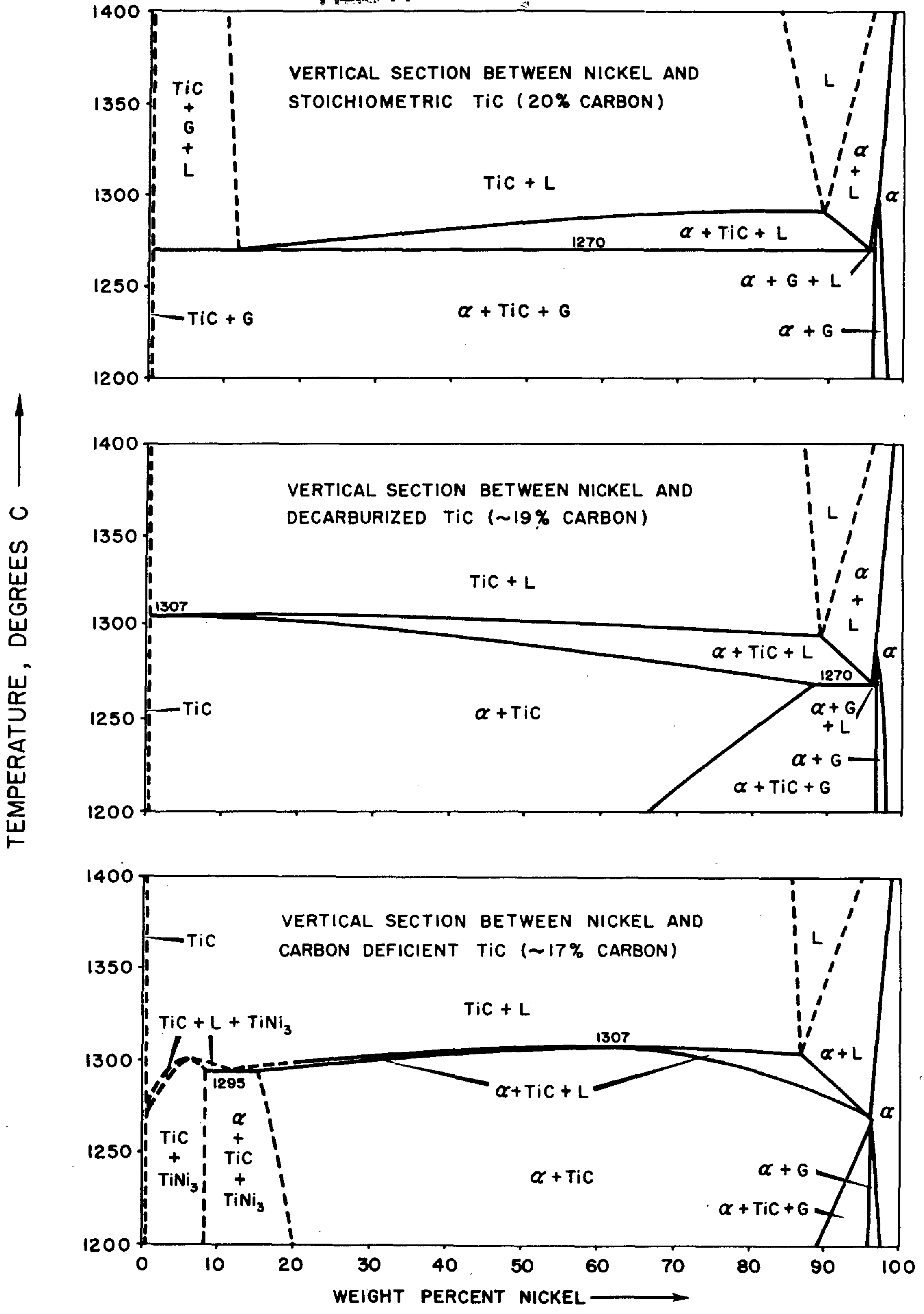

FIGURE 4. VERTICAL SECTIONS THROUGH TERNARY Ni-Ti-C DIAGRAM AT AND NEAR THE TiC-Ni PLANE.

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