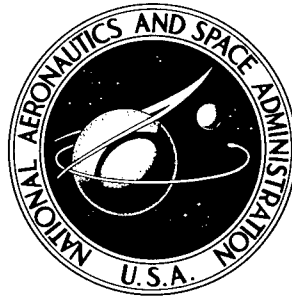


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MECHANICAL PROPERTIES, OXIDATION CHARACTERISTICS, AND WELDABILITY OF TWO UNCOATED AND COATED VANADIUM-BASE ALLOYS

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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SUMMARY

An experimental investigation was made to evaluate two vanadium-base sheet alloys for aerospace applications in the 1,800° F to 2,400° F (1,260° K to 1,590° K) temperature range. The investigation consisted of mechanical property tests both at room and elevated temperatures and oxidation tests on coated and uncoated material. Three silicide diffusion coatings were evaluated by continuous and cyclic oxidation testing to determine their protective qualities. An X-ray study, a metallographic examination, and hardness measurements were made to identify and locate the elements and compounds present in the coating and substrate before and after elevated-temperature exposure. Fabrication and joining of the alloys are discussed. A description of the equipment and procedures utilized in performing the evaluation tests is included.

INTRODUCTION

Recently, vanadium-base alloys have attracted considerable attention as potential structural materials for aerospace application in the 1,800° F to 2,400° F (1,260° K to 1,590° K) temperature range because of their high strength-weight ratios. Vanadium alloys can be strengthened by solid-solution alloying with other metals including most refractory metals. In addition, small amounts of carbon have been shown to dispersion strengthen vanadium alloys. (See ref. 1.) Since vanadium forms an oxide that has a melting point of 1,250° F (950° K), the vanadium alloys must be protected with a high-temperature oxidation-resistant coating when subjected to elevated temperatures in an oxidizing atmosphere.

Two recently developed vanadium alloys were made available to NASA for a preliminary investigation to determine their capabilities and limitations for use in aerospace applications. The study included determination of tensile properties at room temperature and 1,800° F (1,260° K) to 2,400° F (1,590° K), investigation of oxidation properties of coated and uncoated material from 1,800° F (1,260° K) to 2,700° F (1,760° K), as well as a metallographic and X-ray study. Three silicide-base coatings were studied to determine their resistance to oxidation. Diffusion characteristics of the coating and substrate were also studied to determine the possible need for diffusion barriers in

aerospace applications. Fabrication studies included machining of the alloys and joining by several welding techniques. The results of the investigation are presented herein.

The units used for the physical quantities appearing in this paper are given in both the U.S. customary units and the International System of Units (SI) (ref. 2). An appendix is included to explain the relationship between these two systems of units.

MATERIALS AND SPECIMENS

The sheet material utilized in this investigation had a nominal thickness of 0.05 inch (1.3 mm). The nominal composition of the two alloys was:

- (1) 60 weight percent ^Vcolumbium, 1 percent ^{V, Ti}titanium, and the remainder vanadium
- (2) 20 weight percent columbium, 4 percent titanium, 1 percent zirconium, 0.075 percent carbon, and the remainder vanadium

These two alloys will hereinafter be referred to as V-60Cb and V-20Cb, respectively. No attempt was made to make an interstitial analysis of the alloys either prior to or after completion of the evaluation tests. Both the V-60Cb and V-20Cb alloys were prepared from vacuum arc melted 100-pound (45.4-kg) ingots. These ingots were then extruded, hot rolled to within 30 percent of the desired sheet thickness, and then cold rolled to approximately 0.050 inch (1.3 mm). The alloys and the process used in their manufacturing are described in reference 3.

The configuration of tensile specimens used in this investigation is shown in figure 1. Two types of bend specimens were used; one was a strip 2 inches (5.08 cm) long by 1/2 inch (1.27 cm) wide, and the second was a weld specimen in which two of these strips were welded together along a 2-inch (5.08-cm) edge. Oxidation specimens 0.60 inch (1.52-cm) square were machined from the 0.050-inch (1.3-mm) sheet material.

Vanadium forms the liquid vanadium pentoxide (V_2O_5) when subjected to temperatures above 1,250° F (950° K) in air; therefore, an oxidation-resistant coating must be applied to the vanadium alloys for high-temperature application in an oxidizing atmosphere. Since very little is known about protective coatings for vanadium-base alloys, it was decided to evaluate several commercially available coatings. In this investigation three coatings were studied for both the V-60Cb and V-20Cb alloys. They were reported by the coating manufacturers to be:

- (1) A pure ^Vsilicide
- (2) A modified silicide containing Cr, ^{V, Ti, C, Ta}Ti, C, and Ta
- (3) A modified silicide containing Cr, ^{V, Fe, B}Fe, and B

Two coating thicknesses were investigated for the pure silicide, 0.0028 inch (0.071 mm) thick and 0.0015 inch (0.038 mm) thick. These coatings will subsequently be referred to as "first group" and "second group," respectively.

The National Aeronautics and Space Administration provided tensile, bend, and oxidation specimens to the coating suppliers for application of the coatings. Prior to shipping the uncoated specimens to the three coating suppliers, the test specimens were tumbled in a ball mill using a dry mixture of pebbles and 180 grit silicon carbide for approximately 150 hours. The tumbling produced rounded edges and corners for better application of the coating in these areas. Figure 2 shows photomicrographs of sections taken through edges of coated specimens selected at random from each coating supplied. Of particular interest are the relative uniformity of the coating around the edge of each specimen and the thickness of the coatings.

The following material conditions are defined to help the clarity of this report. "As-received" material refers to the uncoated V-60Cb and V-20Cb material. "As-coated" material refers to the coated V-60Cb and V-20Cb material which has not been exposed to elevated temperatures. "Base" material refers to the V-60Cb and V-20Cb substrate.

TEST PROCEDURE

Mechanical Properties

Tensile tests.-- Tensile tests were conducted in a 10,000-pound (44.5-kN) capacity screw-powered testing machine at a head speed corresponding to a strain rate of 0.005 per minute (0.000083 per second) to the yield stress and at an increased head speed corresponding to a strain rate of 0.05 per minute (0.00083 per second) from yield to failure. All specimens were loaded parallel to the longitudinal grain direction.

Room-temperature stress-strain tests were made on each alloy in the as-received condition using two linear differential transformers mounted back to back on knife edges to determine the modulus of elasticity and yield stress. Load was recorded autographically against strain in these room-temperature stress-strain tests and against extension as determined by the relative separation of the testing machine heads for all subsequent tests.

The elevated-temperature tensile specimens were resistance heated and held at temperature for approximately 1 minute before testing. Temperatures on coated specimens were measured at the center of the test section with an optical pyrometer; no emittance corrections were made. Uncoated material was protected from oxidizing during the elevated-temperature tests by the use of a protective shield of argon gas enclosing the test specimen. A thermocouple was used to measure temperatures on the uncoated specimens.

Bend tests.-- Bend tests were made at room temperature in the 10,000-pound (44.5-kN) capacity screw-powered testing machine, utilizing the fixture shown in figure 3. The bend specimens were simply supported on 1/8-inch- (3.2-mm)

diameter pins, 1 inch (2.54 cm) apart, and were centrally loaded by a ram with a contact radius of one t. Bending loads were applied at a head speed of 0.01 inch per minute ($4.2 \mu\text{m/s}$). Bend tests were also made on butt-welded specimens joined by tungsten-inert-gas and electron-beam welding. The ram loaded the specimen normally to the direction of the weld to determine the characteristics of the weld as well as the heat-affected zone and base material.

Oxidation Tests

Continuous tests.- Continuous oxidation tests were made in static air at atmospheric pressure in both vertical and horizontal tube furnaces at discrete temperatures from $1,800^\circ \text{F}$ ($1,260^\circ \text{K}$) to $2,500^\circ \text{F}$ ($1,650^\circ \text{K}$). The vertical tube furnace was equipped with a continuous weighing system. The specimens were placed in a zircon boat, which was suspended in the furnace by a platinum wire. The weight change of the specimen during exposure was autographically recorded on a sensitive balance system. In addition, all specimens were weighed before and after each test as a means of checking the automatic balance record. On several tests, it was not possible to weigh the specimens after exposure due to catastrophic failure of the test specimens. For the coated specimens, the tests were terminated after a 10-percent weight gain.

Cyclic tests.- Cyclic exposure of silicide-coated specimens usually results in a sharp reduction in coating performance. (See ref. 4.) To determine the lifetime of the coated vanadium alloys, cyclic oxidation tests were made in static air in an open-ended horizontal tube furnace at discrete temperatures from $2,000^\circ \text{F}$ ($1,370^\circ \text{K}$) to $2,700^\circ \text{F}$ ($1,760^\circ \text{K}$). Test specimens were inserted in the furnace at the test temperature, exposed to temperature for 1 hour, removed from the furnace, air cooled, and weighed. The cycle was then repeated until a 10-percent weight loss occurred which was taken as failure. A visual examination was made to determine the onset of vanadium oxide formation.

Metallurgical Studies

X-ray diffraction, emission techniques, hardness measurements, and metallographic examination were used as an aid in evaluating the mechanical and oxidation properties of the uncoated and coated vanadium-base alloys.

X-ray analysis.- The procedure used for X-ray analysis on the as-received and coated material is given in reference 5. The diffraction patterns were indexed by using the American Society for Testing Materials (ASTM) card data file for all compounds except VSi_2 and $\text{TiO}_2 \cdot \text{Cb}_2\text{O}_5$. The VSi_2 was identified from a known structure and a lattice parameter taken from reference 6. The $\text{TiO}_2 \cdot \text{Cb}_2\text{O}_5$ was identified on the basis of information given in reference 7.

Metallographic and hardness examination.- Sheared sections were removed from the tested specimens and as-received material. The edges of these sections were wet ground to remove any effect of cold work left from the shearing operation. The sections were then mounted and mechanically polished. After the polishing procedures, the specimens were etched by using a solution of 60 cm^3 of

lactic acid, 5 cm³ nitric acid, and 5 cm³ hydrofluoric acid. Photomicrographs were taken at various magnifications from 50 to 1000 diameters to show the detailed structure in the sectioned specimens. Hardness measurements were made on the coated sections before and after exposure to elevated temperatures. All hardness measurements were made on a microhardness tester by using the Knoop indenter with a 100-gram mass.

Diffusion study. - The behavior of the coating-metal interface after elevated temperature exposures was studied on the V-60Cb specimens. To do this, thickness measurements were made on the specimens in the as-coated condition. These specimens were then exposed at 2,400° F (1,590° K) for various time periods up to 160 hours. The coated specimens were then sectioned, and coating and substrate thickness measurements were made by using a filar micrometer stage on a bench microscope at a magnification of X150. The extent of diffusion on the coating and substrate was determined by plotting change in total specimen and coating thickness with time.

Fabrication Study

Some fabrication characteristics of the vanadium alloys were studied to determine possible difficulties in machining and joining the material into structural components. These studies included machining of the test specimens from the sheet material and the joining of the material by tungsten inert-gas (hereafter referred to as TIG welding), and electron-beam welding (hereafter referred to as EB welding). The parameters for TIG and EB welding for both the V-60Cb and V-20Cb material were determined through experimentation with small specimens and are given in table I. The description of the weld bend tests was given previously.

RESULTS AND DISCUSSION

Mechanical Properties

Tensile tests. - The results of the tensile tests on the 0.050-inch- (1.3-mm) thick V-60Cb and V-20Cb alloys are presented in tables II and III and are shown in figures 4 to 10. Figure 4 shows the room-temperature stress-strain curves for the as-received V-60Cb and V-20Cb alloys. Figure 5 shows the stress-extension curves for the as-received material tested at temperatures of 1,800° F (1,260° K) and 2,200° F (1,480° K). A comparison between the room-temperature ultimate strength (table II) and elevated-temperature ultimate tensile strength (fig. 5) of the V-60Cb material shows that at 1,800° F the tensile strength was reduced approximately 51 percent and at 2,200° F the strength was reduced 87 percent. The same comparison for the V-20Cb alloy indicates a reduction in strength of 56 percent at 1,800° F and 85 percent at 2,200° F.

Figure 6 shows the room-temperature tensile data for the V-60Cb alloy with a pure silicide coating and the modified Cr-Ti silicide coating. Prior to testing, the tensile specimens were preoxidized for 24 hours in air in a vertical-tube furnace at a temperature of 2,000° F (1,370° K). The low value

of elongation for the first group pure silicide indicated embrittlement of the V-60Cb substrate. Considerable reduction of elongation for the second group was also noted but some ductility was retained. There was little or no degradation of material strength due to the application of the pure silicide (second group) or the modified Cr-Ti silicide coating on the V-60Cb substrate.

Tensile specimens coated with the modified Cr-B silicide coating and pre-oxidized for 24 hours at 2,000° F (1,370° K) failed catastrophically during the exposure cycle. Figure 7 shows the three different diffusion coatings on tensile specimens after 24 hours exposure at 2,000° F. The outside specimens coated with the pure silicide and the modified Cr-Ti silicide coatings appeared not to be affected by preoxidation. The modified Cr-B silicide-coated specimen in the center of the figure shows the typical failure experienced with this coating. Failure of the coating occurred around the edge of the specimen and exposed the substrate which oxidized and flowed through the damaged coating.

Results shown in figure 8 and given in table III indicated that the first group pure-silicide-coated specimens appeared to be embrittled below 2,000° F (1,370° K) but higher values of elongation and strength values to 43,000 psi (296 MN/m²) were recorded at 2,200° F (1,480° K). Because of the limited number of second group pure-silicide-coated specimens, no elevated-temperature tests were made. The material coated with the modified Cr-Ti silicide coating retained strength values to 45,000 psi (310 MN/m²) at 2,200° F. The specimens coated with modified Cr-B silicide appear to have embrittled the substrate at 2,000° F but no definite conclusion can be reached because of the limited number of tests.

The strength of the coated V-60Cb alloy is compared with that of two coated refractory alloys, a chrome composite, and dispersion-strengthened nickel on a strength-weight basis in figure 9. In view of the temperature-measuring methods used in this study, that is, optical pyrometer with no corrections for emittance, the following comparison may not be exact but the data for the V-60Cb alloy are on the conservative side. The refractory alloys used for comparison are Mo-0.5Ti-0.08Zr-0.03C molybdenum alloy (ref. 8) and Cb-28Ta-10W-1Zr columbium alloy (ref. 9). The data for the chromium composite and dispersion-strengthened nickel also used for comparisons are found in references 10 and 5, respectively.

On a strength-weight basis (fig. 9) the V-60Cb material appears to retain a higher ratio over the temperature range (1,800° F to 2,400° F; 1,260° K to 1,590° K) than any of the other materials cited. Dispersion-strengthened nickel and the chromium composite do not require a protective coating at the temperatures investigated whereas the refractory alloys which show good elevated-temperature strength do require a protective coating because of their oxidation characteristics. The vanadium alloy also requires a high-temperature oxidation-resistant coating. The increased use of vanadium alloys at elevated temperatures will probably depend on the coating reliability. As will be shown in a subsequent section, a coating failure can be very sudden, and thereby can cause degradation of the substrate material.

Figure 10 shows the elongation in 1 inch (2.54-cm) for the as-received V-60Cb and V-20Cb alloys and also the effect of preoxidation on the elongation

of the V-60Cb coated alloy tested at room temperature. ^{V-60Cb} The as-received material is taken as the standard and shows good room-temperature elongation. The specimens coated with the pure silicide and the modified Cr-Ti silicide retained at least 8-percent elongation over a 1-inch- (2.54-cm) gage length after 24 hours exposure at 2,000° F (1,370° K) whereas the first group pure silicide was badly embrittled.

Bend tests.- Bend tests were made on coated V-60Cb alloy in the as-coated condition as well as after 24 hours exposure at 2,000° F (1,370° K) to supplement tensile elongation data. Two bend tests were made on the as-received sheet with the resulting average bend angle that the specimens withstood (a 115° bend) taken as the reference angle for the tests on the preoxidized materials. The results of testing shown in figure 11 and given in table IV indicate that the bend ductility was decreased by coating the specimens but that the second group pure silicide and modified Cr-Ti coated material retained at least 30 percent bend ductility in the as-coated condition as well as after exposure at 2,000° F. The first group pure silicide and the modified Cr-B coated specimens appeared to be very brittle. During the 2,000° F preoxidizing exposure, the Cr-B coated specimen failed. These results indicate the same trend as those found in the tensile testing of the V-60Cb material. *60Cb*

Oxidation Properties

Continuous tests, uncoated.- The results of continuous oxidation tests on as-received specimens of both vanadium alloys subjected to elevated-temperature exposures in both the vertical and horizontal tube furnace are shown in figures 12 and 13. These tests were made at 1,800° F (1,260° K) and 1,950° F (1,340° K) at exposure times ranging from 3 to 15 minutes. Figure 12 shows the loss of material due to oxidation for the two alloys. At both temperatures the V-20Cb alloy lost more material due to oxidation than did the V-60Cb alloy. This loss is probably due to the higher vanadium content in the V-20Cb alloy which readily caused formation of V₂O₅ at temperatures above 1,200° F (920° K). (See ref. 11.) The V-60Cb alloy also forms V₂O₅ but at a somewhat reduced rate.

Figure 13 shows the V-60Cb and V-20Cb alloys after exposure to 1,950° F (1,340° K) for 15 minutes. The oxide formed on the surface of the V-60Cb alloy is approximately 72 mils thick (1.8 mm) whereas the oxide on the V-20Cb alloy is approximately 15 mils (0.4 mm) thick, even though figure 12 shows a larger material loss to oxidation for the V-20Cb. The thinner oxide indicates that molten V₂O₅ ran off the specimen during elevated-temperature exposure for the alloy containing the high content of vanadium.

Continuous tests, coated.- The results of continuous oxidation tests on the coated vanadium alloys are presented in figures 14 and 15 and table V. The oxidation tests were terminated at various times to 192 hours unless coating failure had occurred. When the tests were terminated, some of the specimens were utilized in a metallographic investigation. Table V indicates that the oxidation protection of the pure silicide was essentially the same for both the first and second group. Figure 14 shows curves of weight change in percent

plotted against time at various temperatures ranging from 2,000° F to 2,500° F (1,370° K to 1,650° K) for the V-60Cb alloy. The curves in figure 14(a) show that the pure silicide coating appears to offer good oxidation protection at 2,200° F (1,480° K) for 192 hours and for 100 hours at 2,400° F (1,590° K). At 2,500° F (1,650° K) the coating protected the material for 80 hours. The data for the modified Cr-Ti silicide coating shown in figure 14(b) indicate that at 2,000° F and 2,200° F the coating offers good oxidation protection for at least 168 hours. At 2,400° F there is considerable scatter in the data (table V), but the coating offered protection for approximately 50 hours. The data for the modified Cr-B silicide coating are not shown in the figure because coating failure occurred in the first few hours of testing at temperatures of 2,000° F and above. The results for the continuous oxidation data on the V-20Cb alloy presented in figure 15 indicate that the modified Cr-Ti silicide affords oxidation protection up to 2,200° F. On an average the three coatings did not protect the V-20Cb alloy as well as they did the V-60Cb alloy.

Coating failure and subsequent oxidation of the substrate are indicated on the two figures by a sudden increase in weight. It was noted previously that exposure of uncoated vanadium to elevated temperatures produces a pentoxide, V₂O₅, which has a melting point of 1,250° F (950° K). (See ref. 11.) As a result, molten V₂O₅ will run off a specimen into the zircon boat at temperatures above 1,250° F. This condition makes it impossible to weigh the specimens and the zircon boats separately; therefore, the weight change shown in figures 14 and 15 represents the change in weight of the test specimen and the boat. The sudden increase of weight on the two figures probably indicates the formation of the V₂O₅. Even though the specimen lost weight, the combined weight of specimen and zircon boat saturated with V₂O₅ is a weight gain.

Cyclic tests.—One-hour cyclic tests were made on the coated vanadium alloys to study the types of coating failures that occur and to determine the coating lifetime under more severe test conditions. The test specimens were weighed before and after each 1-hour cycle. Since the zircon boat was not weighed with the test specimens, only the change in weight of the specimens is plotted against time for the cyclic oxidation data in figures 16 to 18. The cyclic tests data are presented in table VI. The cyclic tests were terminated after a 10-percent weight loss which was taken to be specimen failure. Figure 16 shows the cyclic oxidation data for the three silicide coatings investigated on the V-60Cb at 2,300° F (1,540° K). The average lifetime of the pure silicide coating was approximately 33 hours whereas the modified Cr-Ti silicide had an average lifetime of approximately 50 hours. The specimens that failed after two cycles apparently had a defective coating. The specimens with the modified Cr-B silicide coating failed after a very few cycles. The cyclic oxidation data for the V-60Cb alloy at 2,500° F (1,650° K) are shown in figure 17. It is evident from the data that the coatings failed after a few cycles and the only specimens lasting longer than 30 hours were the ones coated with the pure silicide. Cyclic tests were also made on the pure-silicide-coated material at temperatures of 2,000° F (1,370° K) and 2,700° F (1,760° K). At 2,000° F the tests were terminated after 120 cycles with no apparent damage to the coating. At 2,700° F no specimen lasted longer than three cycles.

The cyclic data for the V-20Cb coated specimens exposed to temperatures ranging from 1,800° F (1,260° K) to 2,200° F (1,480° K) are presented in figure 18. There is scatter in the data for the pure silicide coating at 2,000° F (1,370° K), one test being terminated after 171 cycles with no apparent coating damage. The modified Cr-Ti silicide coating had an apparent lifetime of 38 and 32 cycles at 1,800° F and 2,000° F, respectively, whereas the modified Cr-B silicide had a very short lifetime at 2,000° F. Because of the small quantity of V-20Cb material available, very few tests were made. Figure 19 shows the progression of failures for the three silicide coatings investigated on the V-60Cb. These specimens were cycled at 2,300° F (1,540° K). The pure silicide coating generally failed on the edge or corner as did the modified Cr-Ti silicide with the exception of a few specimens that failed on the flat surface. The modified Cr-B silicide coating always exhibited edge failures.

Metallurgical Study

X-ray, metallographic, and microhardness examination.— A metallurgical study was made on the vanadium-base alloys to identify oxidation products formed during elevated-temperature exposure and to determine why the pure silicide and the modified Cr-Ti silicide protected the material to 2,400° F (1,590° K) whereas the modified Cr-B silicide failed in a few hours at 2,000° F (1,370° K). This study correlates the results of an X-ray analysis, microstructure examination, and microhardness measurements.

Figures 20 to 24 show photomicrographs of the base material and the three coatings before and after elevated-temperature exposure. The microstructures of the as-received V-20Cb and the V-60Cb material are shown in figure 20. The remaining photomicrographs showing the coatings are for the V-60Cb alloy since only a limited number of the V-20Cb alloy specimens were available for the metallographic study. The X-ray diffraction study showed that the "d" spacings for each reflecting plane of the V-60Cb are about midway between the "d" spacing of the pure vanadium and pure columbium for the same reflecting plane. Since no other X-ray peaks were found, the V, Cb, and Ti materials appear to be completely soluble in the solid state. The lattice parameters for the V-60Cb and the V-20Cb were determined to be 3.17 Å and 3.08 Å (31.7 and 30.8 nm), respectively.

The X-ray study on the as-received material that was exposed to elevated temperatures identified the following oxidation products: V₂O₅, TiO₂·Cb₂O₅, CbO₂, and VO₂.

The pure silicide coating on the first and second group of V-60Cb specimens is shown in figures 21 and 22 after various exposure times to 192 hours at a temperature of 2,400° F (1,590° K). This coating provided a reference for the reactions which occur at elevated temperatures in the other two modified silicide coatings. A metallographic examination of the pure silicide coating (fig. 21) shows the microstructure of the coating before and after 2,400° F exposure. After elevated-temperature exposure, the coating appears to have a number of phases present. The X-ray diffraction study on the pure silicide

coating indicates that the coating "as-coated" was a solid-solution vanadium-columbium disilicide $(V-Cb)Si_2$. Microhardness values of 1,097 Knoop and 1,040 Knoop were recorded for the coating "as-coated." These values are in agreement with reference 12 which gives similar values for VSi_2 and $CbSi_2$ with a 100-gram mass. After specimen exposure of 192 hours at 2,400° F, the X-ray study identified the outer layer as [the crystalline form of SiO_2] alpha cristobalite. Below the SiO_2 layer, a higher order vanadium-columbium silicide $(V-Cb)_5Si_3$ was found which gradated into a thin layer of untransformed $(V-Cb)Si_2$ and finally the substrate. The hardness measurements of 495, 1,230, and 1,100, respectively, were found for the three above-mentioned layers. Reference 13 gives a hardness value of 820 for SiO_2 and reference 14 indicates that the hardness of V_5Si_3 runs from 1,300 to 1,500 Knoop. Figure 21(d) shows porosity in the outer layer which could account for the low hardness value for SiO_2 .

The specimen with the modified Cr-Ti silicide coating is shown in figure 23. After elevated-temperature exposure, the photomicrograph depicts three regions above the coating metal interface. The X-ray study identified the coating "as-coated" as a solid-solution vanadium-columbium disilicide $(V-Cb)Si_2$. After elevated-temperature exposure, the outside was identified as the crystalline form of SiO_2 , alpha cristobalite, followed by high-order $(V-Cb)_5Si_3$ which gradated into the untransformed $(V-Cb)Si_2$ just above the coating metal interface. These phases appear to be identical to those found in the pure silicide. No evidence of the Ti or Cr was noted in the X-ray data; thus, they were probably in solid solution with the vanadium and columbium. Hardness measurements made in the coating after exposure showed values of 374, 1,389, and 1,161 Knoop in the three areas. The 1,389 Knoop corresponds to the $(V-Cb)_5Si_3$ identified by X-ray analysis as does the 1,161 Knoop for $(V-Cb)Si_2$ and the value of 374 Knoop is low for SiO_2 .

The specimen with the modified Cr-B silicide is shown in figure 24. The metallographic examination shows two distinct regions above the metal-coating interface. Although diffraction studies showed no specific iron compounds, emission clearly showed significant amounts in the as-coated condition. Reference 15 states that Si and Cr form Cr_3Si on the outside of columbium during coating deposition. The X-ray study confirmed such a compound Cr_3Si had also formed on the surface of the vanadium-base alloys and below this the separate vanadium VSi_2 and columbium $CbSi_2$ disilicides. Hardness measurements indicated a region with a Knoop hardness of 1,690 and another of 1,306. These values are considerably above the recorded values of 1,005 Knoop (ref. 14) for Cr_3Si and 1,090 Knoop for VSi_2 but interstitial contaminates, not noted in the X-ray data, could increase the hardness a considerable amount. After elevated-temperature exposure, the coatings appear to show two separate areas. X-ray analysis indicates an iron chromium oxide $FeCr_2O_3$ has formed on the surface

followed by the $(V-Cb)_5Si_3$ which gradated into the uncombined VSi_2 and $CbSi_2$. Hardness measurements showed two areas, one of 274 Knoop and the other of 1,050 Knoop. The 1,050 Knoop hardness value indicates probable formation of vanadium or columbium disilicides.

The results of this study combined with the oxidation results indicate that the coatings that formed SiO_2 after elevated-temperature exposure offered good oxidation protection on the V-60Cb alloy while the coating that formed $FeCr_2O_3$ on the surface did not protect the substrate. Thus the pure silicide and modified Cr-Ti silicide both of which formed essentially the same compounds protected the V-60Cb substrate about equally whereas the modified Cr-B silicide which formed different compounds afforded poor oxidation protection. In this case the boron addition to the silicide apparently does not aid in forming the borosilicate glass which had been found to provide good oxidation protection on silicide-coated molybdenum. (See ref. 16.)

Diffusion study.- A study of the coating-metal interface was undertaken on the V-60Cb alloy to determine the rate at which the coating diffused into the substrate and whether diffusion barriers would be required on thin sheets. Figure 25 shows the increase in total specimen thickness and decrease in substrate thickness. The coating on the pure silicide first group is shown to be relatively thick on the specimens. The pure silicide coating second group as well as the modified Cr-Ti silicide coating appear to be very similar in all respects. The rate of substrate loss as well as increase in coating thickness is nearly identical for both coatings. This similarity is an indication that the diffusion phenomenon encountered is mainly one involving the diffusion of silicon into the solid-solution vanadium-columbium alloy and that small additions of Cr and Ti do not appreciably affect diffusion. At 2,400° F (1,590° K) the coating appears to diffuse into the substrate about 0.003 inch (0.076 mm) on a side after 160 hours; therefore, diffusion barriers will probably be needed if the vanadium-base alloys are utilized for high-temperature structural use in thin gages.

Fabrication Study

Fabrication characteristics of the two vanadium-base alloys were studied by machining and by various joining methods such as EB and TIG welding.

Welding.- The weld bend specimens were tested to determine weldability of the alloy by the methods described in the procedure. The test results are plotted in figure 26 and presented in table VII. Load is plotted against bend angle and the bend strength of the welded V-60Cb appears to be about 50 percent stronger than the bend strength of the welded V-20Cb. The weld was normal to the contact radius of the ram. The test on both materials indicated that very good welds could be made on vanadium-base alloys without causing embrittlement by using ordinary welding techniques. Both materials exhibited good bend ductility, but the welds on the V-20Cb showed slight cracking, and the welds on V-60Cb did not (fig. 27) even at a total bend of 100° (1.74 radians).

Microstructures of the electron-beam-welded specimens are shown in figures 28 to 31. Figure 28 shows a cross section and a top view of a V-60Cb specimen welded by electron-beam techniques at 30 inches per minute (1.27 cm/s). The next three figures compare the electron-beam welds made at 20, 30, and 40 inches per minute (0.85, 1.27, and 1.69 cm/s) on the V-20Cb alloy. The microstructure shows no noticeable difference in the three specimens and the bend strengths of the welded specimens were nearly identical. The V-20Cb-EB curve in figure 26 is indicative of the bend strength of the three weld speeds. This result is an indication that satisfactory welding can be done on a production basis at speeds to 40 inches per minute (1.69 cm/s).

Machining. - Difficulty was encountered in the machining of the V-60Cb sheet. Milling operations were limited to 33 revolutions per minute (0.55 revolutions per second) at a table feed of 0.37 inch per minute (0.016 cm/s) and depth of cut of 0.005 inch (0.13 mm). Tungsten carbide milling cutters were found to work satisfactorily. The V-20Cb was machined at a cutting speed of 100 revolutions per minute (1.66 revolutions per second), table feed of 0.75 inch per minute (0.032 cm/s), and depth of cut of 0.015 inch (0.38 mm) with no difficulty. Tungsten carbide cutters also performed very well on this material.

CONCLUSIONS

An integrated mechanical property and metallurgical study was made on the V-60Cb and V-20Cb vanadium alloys to determine their capabilities and limitations as structural materials. Three commercially available silicide-base coatings were investigated for these alloys. The following conclusions on the vanadium alloys are based on the results found in the present investigation:

1. The coated vanadium-base alloys appear to be very promising as structural materials on a strength-weight basis in the temperature range of 1,800° F (1,260° K) to 2,400° F (1,590° K).
2. The material loss to oxidation for the uncoated material in an oxidizing atmosphere is very rapid at elevated temperatures and the need for an oxidation protective coating is noted.
3. The pure silicide coating and the modified Cr-Ti silicide coating protected the V-60Cb alloy up to 2,400° F (1,590° K) for at least 50 hours under continuous exposure conditions. Coated material that was exposed to cyclic oxidation showed a reduction in coating lifetime.
4. As expected, the formation of silicon dioxide on the surface of the pure silicide and modified Cr-Ti silicide coatings appears to account for their good oxidation protective characteristics.
5. A study of the diffusion characteristics of the pure silicide coating and the vanadium-alloy substrate indicates the possible need for diffusion barriers if the material is to be used in thin sheets.

6. Fabrication studies of the material indicate that machining and forming operations can be accomplished with relative ease on the V-20Cb alloy. Considerable difficulty is encountered in machining the V-60Cb alloy. Both alloys could be tungsten-inert-gas and electron-beam welded with relative ease.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Station, Hampton, Va., September 11, 1964.

APPENDIX

CONVERSION OF U.S. CUSTOMARY UNITS TO SI UNITS

The International Systems of Units (SI) was adopted by the Eleventh General Conference on Weights and Measures, October 1960. (See ref. 2.) Conversion factors for units used herein are given in the following table:

	U.S. customary unit	SI unit	Conversion factor
Temperature	$^{\circ}\text{F}$	$^{\circ}\text{K}$	$\frac{5}{9}(\text{^{\circ}\text{F}} + 459.67)$
Length	inches	centimeter	2.54
	Angstrom	nanometers	10
Force	pound	Newton	4.448
Mass	pound	kilogram	0.4536
Volume	cubic feet	meters ³	2.832×10^{-2}
Stress	pounds/square inch	Newton/meter ²	6.894×10^3
Angle	degree	radians	1.745×10^{-2}
Stress- density	inches	J/kg	0.249089

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TABLE I.- PARAMETERS FOR THE BUTT-WELDING OF VANADIUM ALLOYS

(a) Tungsten-inert-gas

Machine type	a-c--d-c inert arc and metal arc welder
Back-up gas	Argon
Flow rate	6 cu ft/hr (47.2 cm ³ /s)
Torch type	Heliarc
Torch size	M 50A
Electrode	3/32 thoriated tungsten (0.238 cm)
Shielding gas	Argon
Flow rate	20 cu ft/hr (157.3 cm ³ /s)
Travel speed	35 (low speed) in./min (1.48 cm/s)
Polarity	Direct current, straight polarity
Current	120 A

(b) Electron beam

Accelerating potential	120 kV
Beam current	2.8 mA
Welding speed	20, 30, and 40 in./min (0.85, 1.27, and 1.69 cm/s)
Beam deflection	0
Vacuum	10 ⁻⁴ mm Hg (13.3 mW/m ²)

TABLE II.- TENSILE-TEST DATA FOR V-Cb SHEETS AT ROOM TEMPERATURE

Material condition	Yield strength		Tensile strength		Elongation in 1 inch, percent (2.54 cm)	Modulus of elasticity	
	psi	MN/m ²	psi	MN/m ²		psi	GN/m ²
V-60Cb:							
Uncoated	124,500	859	147,300	1016	33	17.5 × 10 ⁶	121
Uncoated	123,400	851	145,500	1004	32	18.5	128
V-20Cb:							
Uncoated	77,000	53.1	101,500	700	28	17.2	119
Uncoated	72,600	50.1	101,500	700	29	17.8	123
V-60Cb:							
Pure silicide, first group:							
As-received			119,000	821	0		
24 hours at 2,000° F (1,370° K)			63,600	43.9	0		
24 hours at 2,000° F (1,370° K)			36,250	25.0	1		
Pure silicide, second group:							
24 hours at 2,000° F (1,370° K)			128,200	885	12		
24 hours at 2,000° F (1,370° K)			127,100	877	7		
Modified Cr-Ti silicide:							
24 hours at 2,000° F (1,370° K)			153,700	1061	8		
24 hours at 2,000° F (1,370° K)			146,200	1009	7		

TABLE III.- TENSILE-TEST DATA FOR V-Cb SHEETS AT ELEVATED TEMPERATURE

Material	Temperature		Tensile strength		Elongation in 1 inch, percent (2.54 cm)
	°F	°K	psi	MN/m ²	
V-60Cb					
Uncoated	1,800	1,260	71,500	49.3	12
Uncoated	2,200	1,480	18,370	12.7	17
V-20Cb					
Uncoated	1,800	1,260	44,700	30.4	19
Uncoated	2,200	1,480	14,800	10.2	21
V-60Cb					
Pure silicide, first group . . .	1,800	1,260	61,400	42.4	3
	2,000	1,370	58,800	40.6	4
	2,200	1,480	43,700	30.2	12
	2,400	1,590	22,610	15.6	--
Modified Cr-Ti silicide	1,800	1,260	92,600	63.9	4
	2,000	1,370	71,400	49.3	6
	2,200	1,480	45,500	31.4	10
Modified Cr-B silicide	2,000	1,370	60,000	41.4	3
	2,000	1,370	61,100	42.2	4

TABLE IV.- BEND TEST DATA FOR THE AS-RECEIVED AND COATED V-60Cb ALLOY

Specimen	Test conditions	Bend angle at failure	
		degrees	radians
As-received:			
1	No exposure	115	2.007
2	No exposure	115	2.007
3	No exposure	115	2.007
Pure silicide:			
4, first group . . .	No exposure	7	0.122
5, first group . . .	No exposure	10	0.175
6, first group . . .	Preoxidized ¹	8	0.140
7, first group . . .	Preoxidized ¹	30	0.524
8, second group . . .	No exposure	57	0.995
9, second group . . .	Preoxidized ¹	39	0.680
10, second group . . .	Preoxidized ¹	37	0.645
Modified Cr-Ti silicide:			
11	No exposure	51	0.890
12	Preoxidized ¹	32	0.559
Modified Cr-B silicide:			
13	No exposure	8	0.140
14	Preoxidized ²	---	-----

¹Preoxidized - exposed for 24 hours at 2,000° F (1,370° K) in air.

²Coating failed during the preoxidizing exposure.

TABLE V.- CONTINUOUS OXIDATION TEST DATA FOR THE COATED V-Cb ALLOYS

Specimen	Test temperature		Hours to 10-percent weight change
	°F	°K	
V-60Cb alloy			
Pure silicide:			
1	2,200	1,480	a ₁₉₂
2	2,200	1,480	b ₁₇₅
3	2,200	1,480	a ₇₂
4	2,200	1,480	a ₇
5	2,400	1,590	b ₁₆₈
6	2,400	1,590	98
7	2,400	1,590	140
8	2,400	1,590	a ₇
9	2,400	1,590	a ₇₂
10, second group . . .	2,400	1,590	a ₁₆₈
11, second group . . .	2,400	1,590	a ₇₂
12, second group . . .	2,400	1,590	a ₇
13	2,500	1,650	124.5
Modified Cr-Ti silicide:			
1	2,000	1,370	a ₇₂
2	2,000	1,370	a ₁₆₈
3	2,000	1,370	a ₇
4	2,200	1,480	a ₇₄
5	2,200	1,480	a ₁₆₈
6	2,400	1,590	11
7	2,400	1,590	a ₁₃₂
8	2,400	1,590	b ₄₃
9	2,400	1,590	a ₄₈
10	2,400	1,590	b ₈₀
11	2,400	1,590	a ₇
Modified Cr-B silicide:			
1	2,000	1,370	7
2	2,400	1,590	1.5
V-20Cb alloy			
Pure silicide:			
1	1,800	1,260	a ₁₆₈
2	1,800	1,260	b ₄₈
3	1,800	1,260	a ₇
4	2,000	1,370	26
5	2,000	1,370	43
6	2,000	1,370	b ₄₂
7	2,000	1,370	a ₁₆₈
8	2,200	1,480	18.5
Modified Cr-Ti silicide:			
1	2,000	1,370	b ₇₂
2	2,000	1,370	a ₁₆₈
3	2,200	1,480	97
Modified Cr-B silicide:			
1	1,800	1,260	81
2	1,800	1,260	25
3	2,000	1,370	16

^aTest terminated for metallurgical investigation.

^bFailures started, terminated prior to 10-percent weight change to investigate type of failure.

TABLE VI.-- CYCLIC OXIDATION TEST DATA FOR THE COATED V-Cb ALLOYS

Specimen	Test temperature		Hours to 10-percent weight change
	°F	°K	
V-60Cb alloy			
Pure silicide:			
1	2,000	1,370	^a 120
2	2,300	1,540	28
3	2,300	1,540	48
4	2,300	1,540	32
5	2,300	1,540	25
6	2,500	1,650	^a 35
7	2,500	1,650	34
8	2,500	1,650	^b 28
9	2,500	1,650	6
10	2,500	1,650	2
11	2,500	1,650	2
12, second group	2,500	1,650	5
13, second group	2,500	1,650	18
14, second group	2,500	1,650	27
15	2,700	1,760	3
16	2,700	1,760	3
17	2,700	1,760	3
18	2,700	1,760	3
Modified Cr-Ti silicide:			
1	2,000	1,370	^a 151
2	2,000	1,370	^a 151
3	2,300	1,540	^b 48
4	2,300	1,540	12
5	2,300	1,540	52
6	2,300	1,540	60
7	2,300	1,540	76
8	2,500	1,650	^b 6
9	2,500	1,650	9
Modified Cr-B silicide:			
1	2,000	1,370	^a 5
2	2,300	1,540	3
3	2,300	1,540	2
4	2,500	1,650	2
5	2,500	1,650	1
V-20Cb alloy			
Pure silicide:			
1	2,000	1,370	59
2	2,000	1,370	10
3	2,000	1,370	^a 171
4	2,300	1,540	^b 80
5	2,300	1,540	44
6	2,300	1,540	25
7	2,300	1,540	19
Modified Cr-Ti silicide:			
1	1,800	1,260	41
2	2,000	1,370	36
3	2,000	1,370	19
4	2,000	1,370	^a 129
5	2,000	1,370	43
6	2,300	1,540	13
Modified Cr-B silicide:			
1	2,000	1,370	7
2	2,000	1,370	7
3	2,300	1,540	3

^aTest terminated for metallurgical investigation.

^bFailures started, terminated prior to 10-percent weight change to investigate type of failure.

TABLE VII.- BEND TEST DATA FOR WELDED UNCOATED V-Cb SHEET MATERIAL

Material	Type of weld	Weld speed,		Ultimate load		Bend angle at ultimate load		Load at failure		Bend angle at failure,	
		in./min	cm/s	lb	N	deg	radians	lb	N	deg	radians
V-60Cb	Tungsten inert gas			660	2937	20	0.350	(a)	(a)	(a)	(a)
V-20Cb	Tungsten inert gas			407	1811	22	.385	273	1215	40	0.70
V-20Cb	Electron beam	40	1.69	420	1869	22.5	.394	(a)	(a)	(a)	(a)
V-20Cb	Electron beam	30	1.27	420	1869	22.5	.394	(a)	(a)	(a)	(a)
V-20Cb	Electron beam	20	.85	420	1869	22.5	.394	(a)	(a)	(a)	(a)

^aSpecimen did not fail.

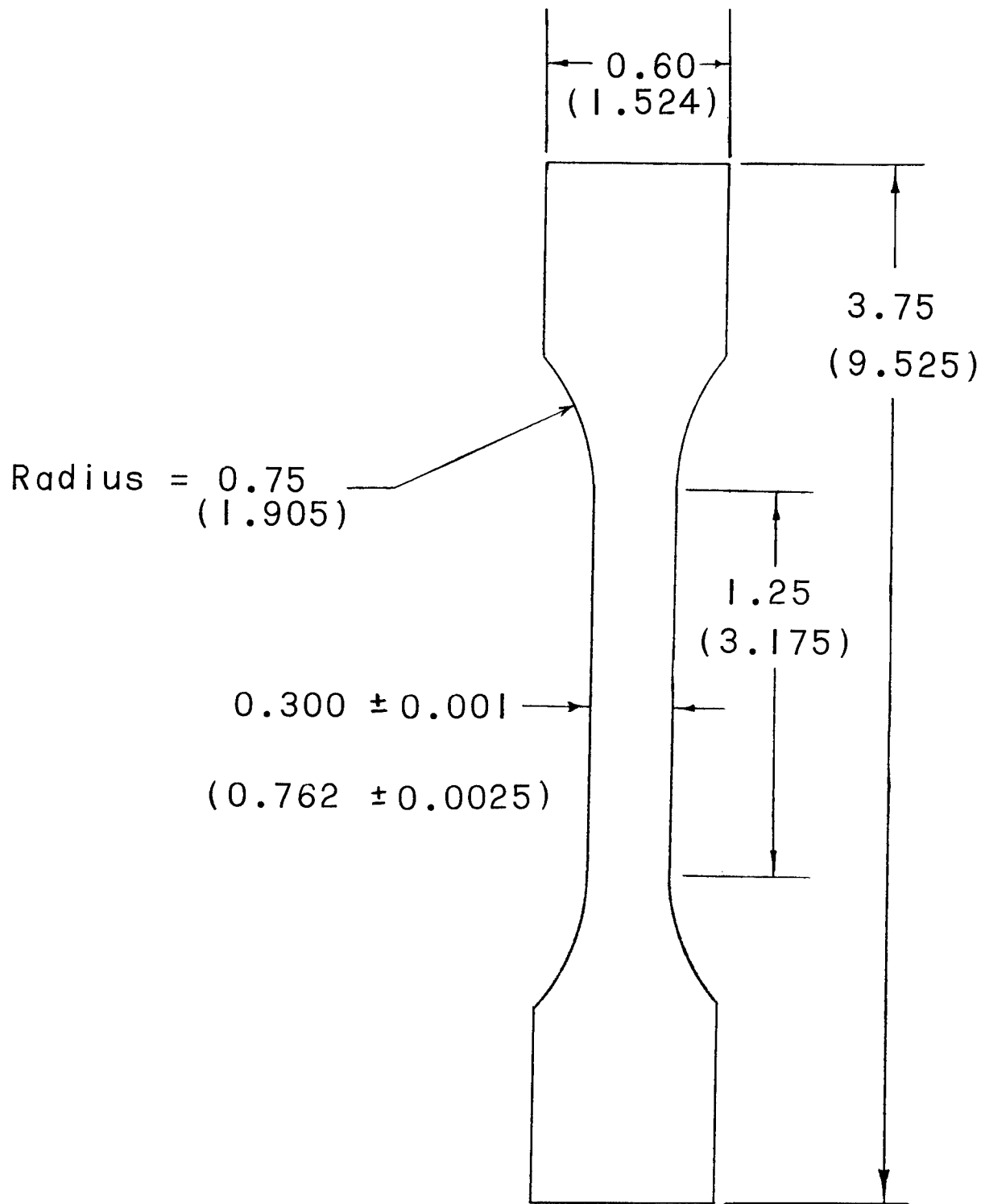
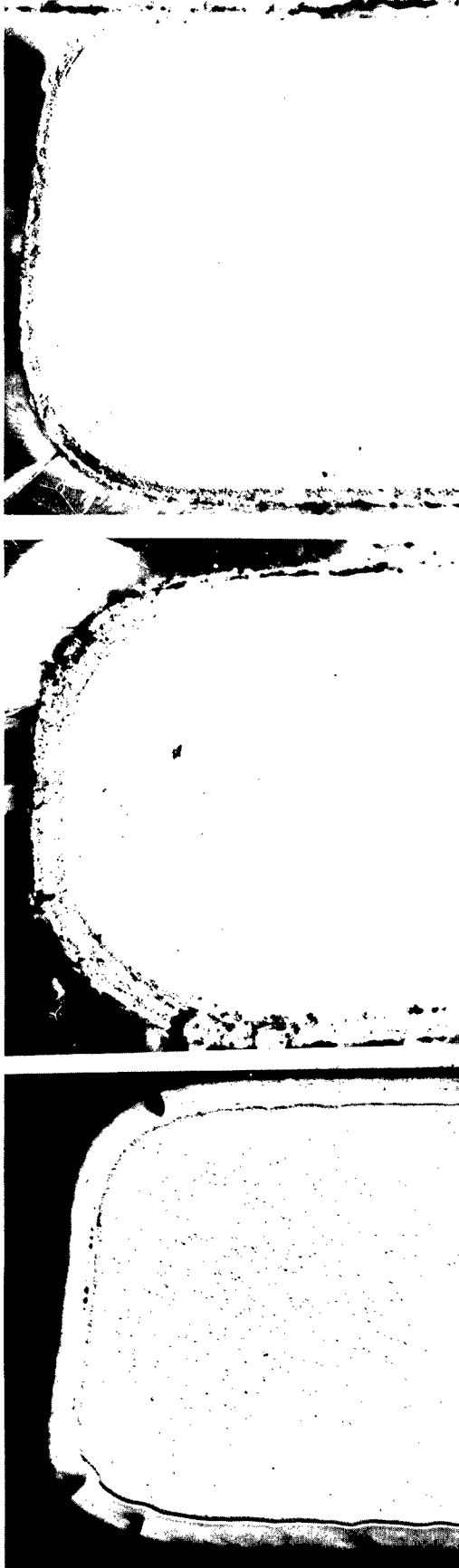


Figure 1.- Tensile specimen. All dimensions are in inches. (Numbers in parentheses are in equivalent centimeters.)



(a) Pure silicide.

(b) Modified Cr-Ti.

(c) Modified Cr-B.

Figure 2.- Photomicrographs of a sectioned edge for each of the three silicide coatings on the V-60Cb alloy L-64-8328 in the as-coated condition. X50.

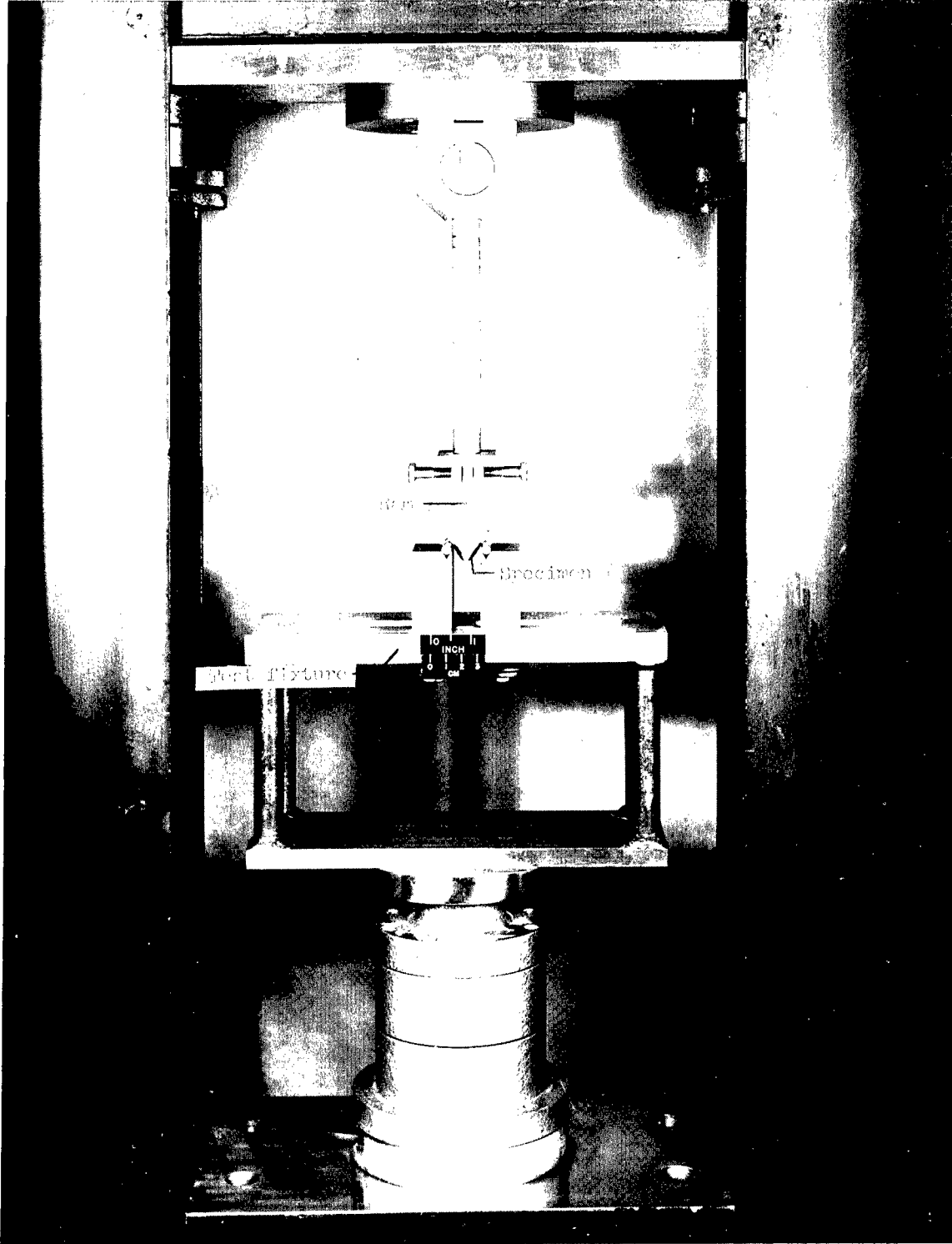


Figure 3.- Test fixture for determination of bend ductility.

L-64-725.1

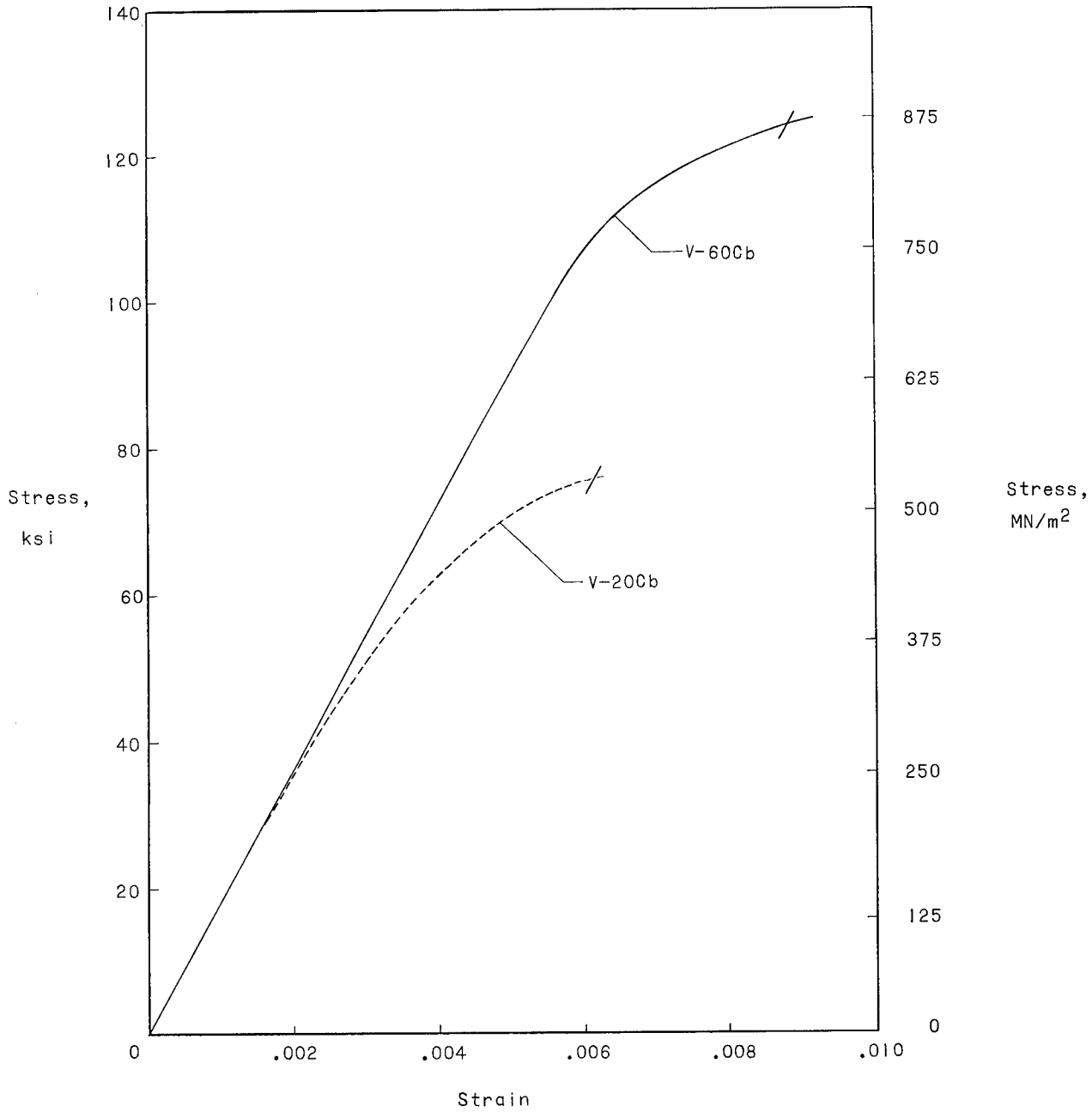


Figure 4.- Tensile stress-strain curves for as-received V-60Cb and V-20Cb alloys at room temperature. Ticks indicate yield stress.

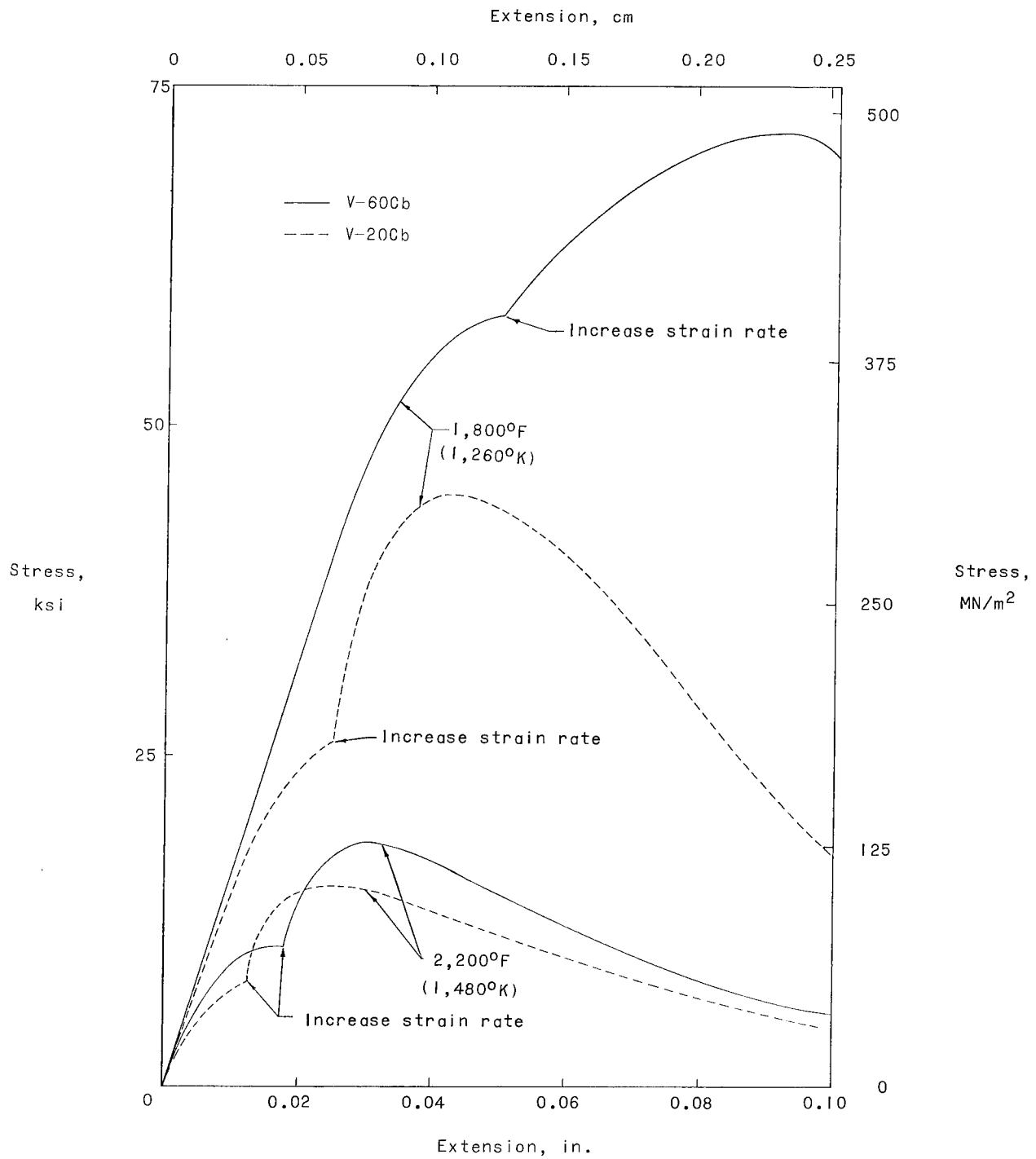


Figure 5.- Stress-extension curves for as-received V-60Cb and V-20Cb alloys at elevated temperatures.

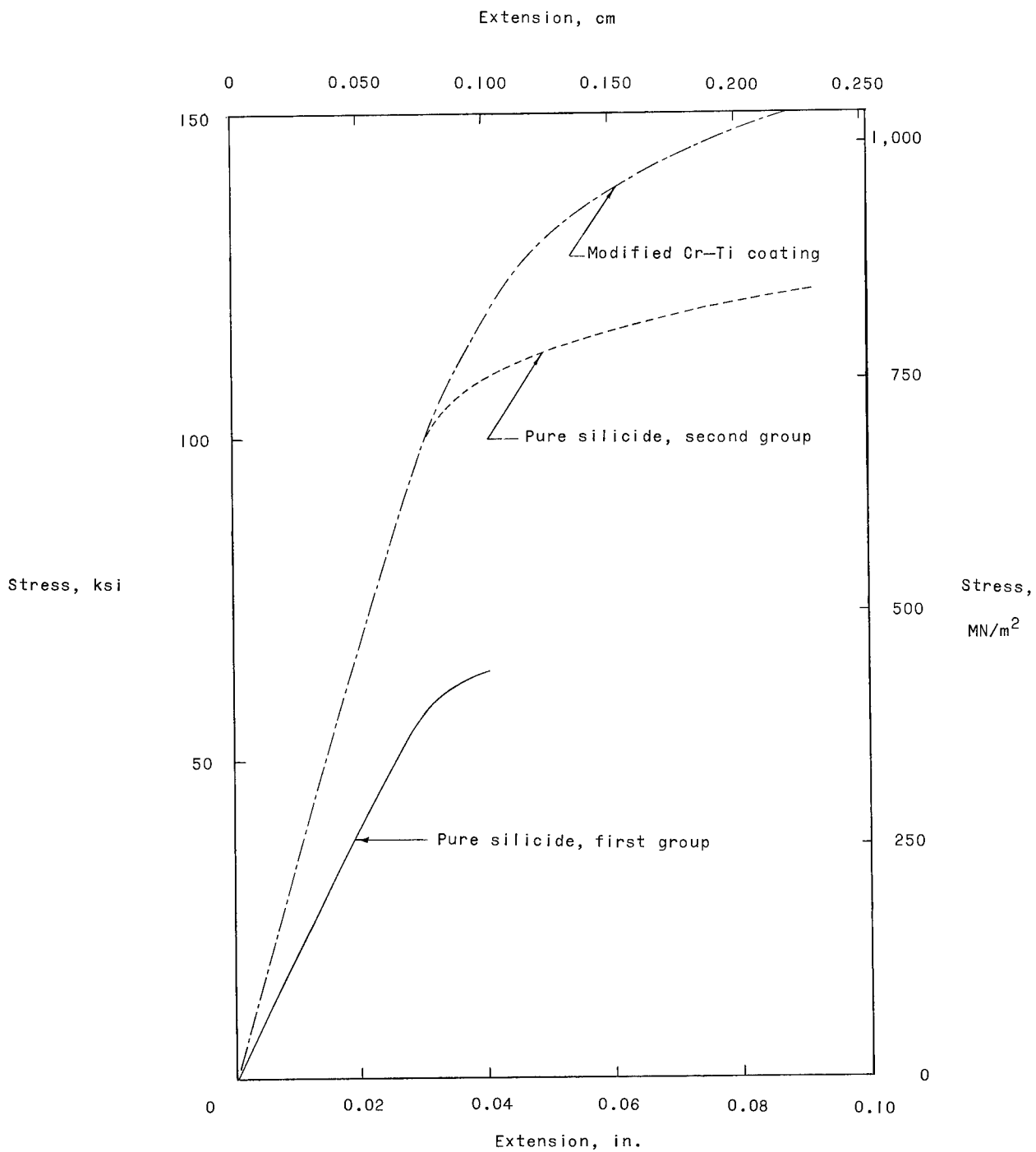
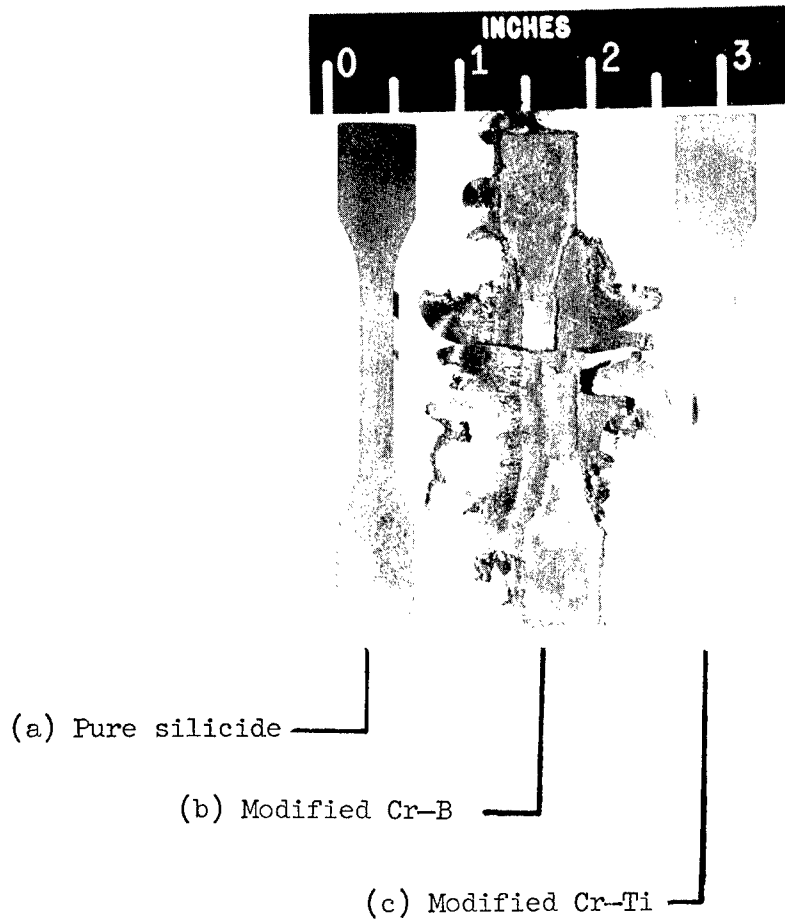
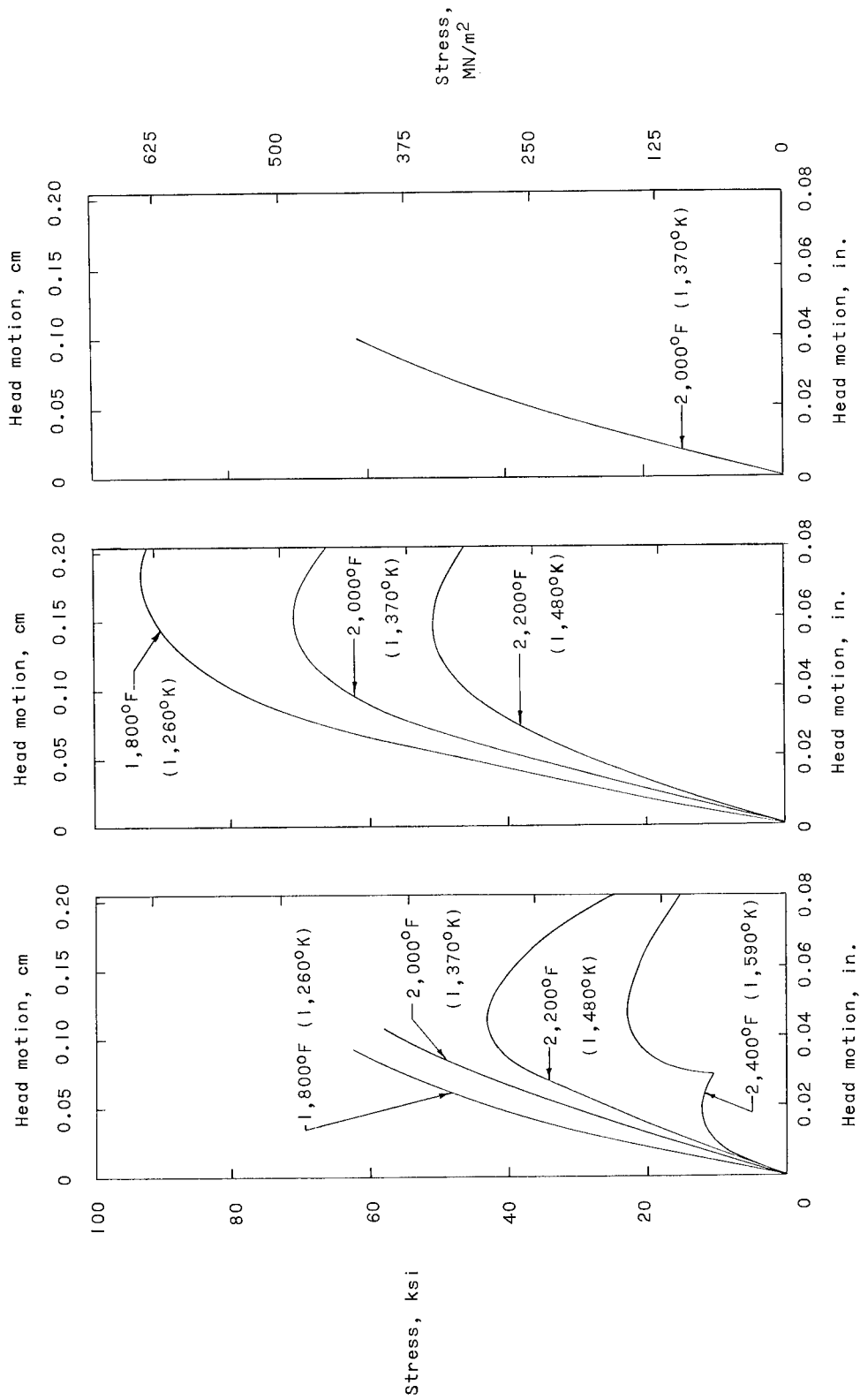


Figure 6.- Selected room-temperature stress-extension curves for preoxidized (24 hours at 2,000° F (1,370° K)) V-60Cb alloy.



L-64-8329
Figure 7.- Coated V-60Cb specimens after 24 hours at 2,000° F (1,370° K). The pure silicide specimen is representative of both the first and second groups.



(a) Pure silicide coating, first group. (b) Modified Cr-Ti coating. (c) Modified Cr-B coating.

Figure 8.- Stress-extension curves for coated V-60Cb alloy at elevated temperatures.

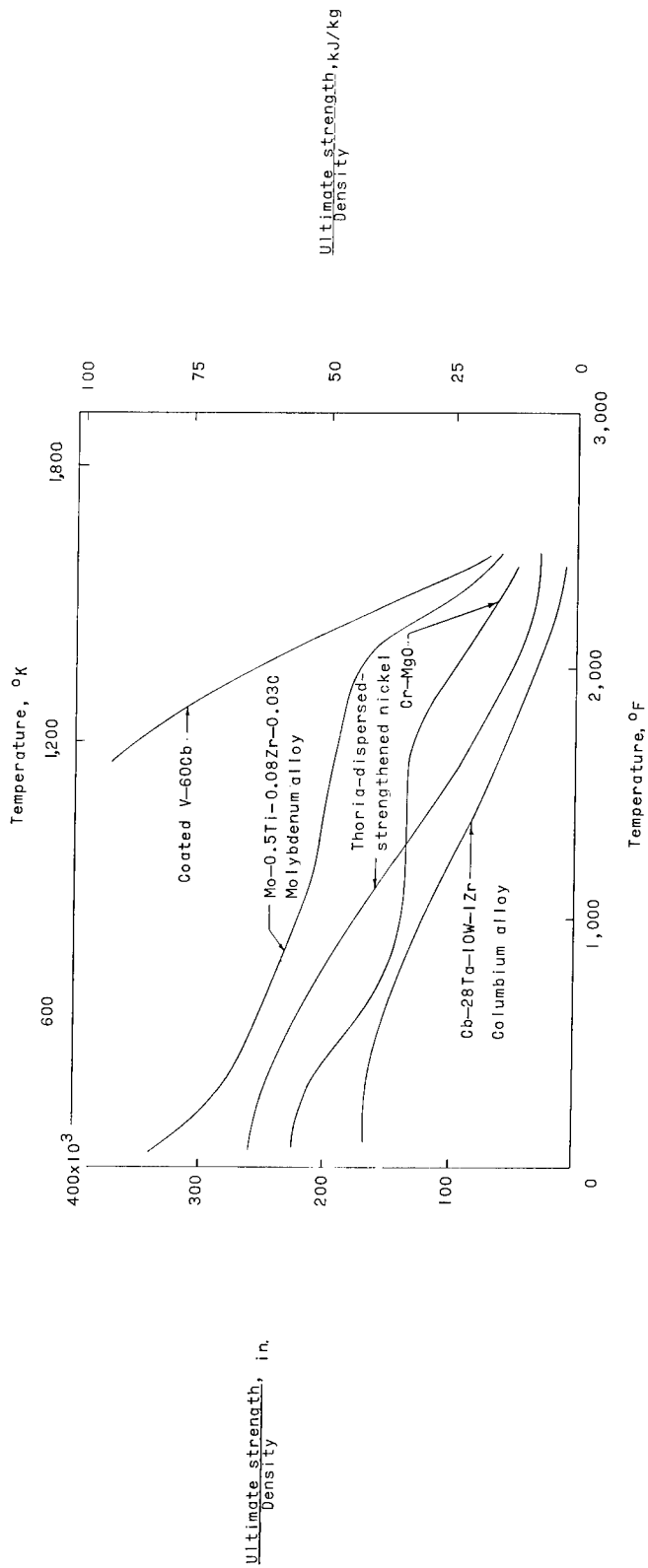


Figure 9.- Comparison of coated V-60Cb alloy data based on uncorrected optical pyrometer readings with two refractory-metal alloys, thoria-dispersed-strengthened nickel and a Cr-MgO composite on a strength-weight basis.

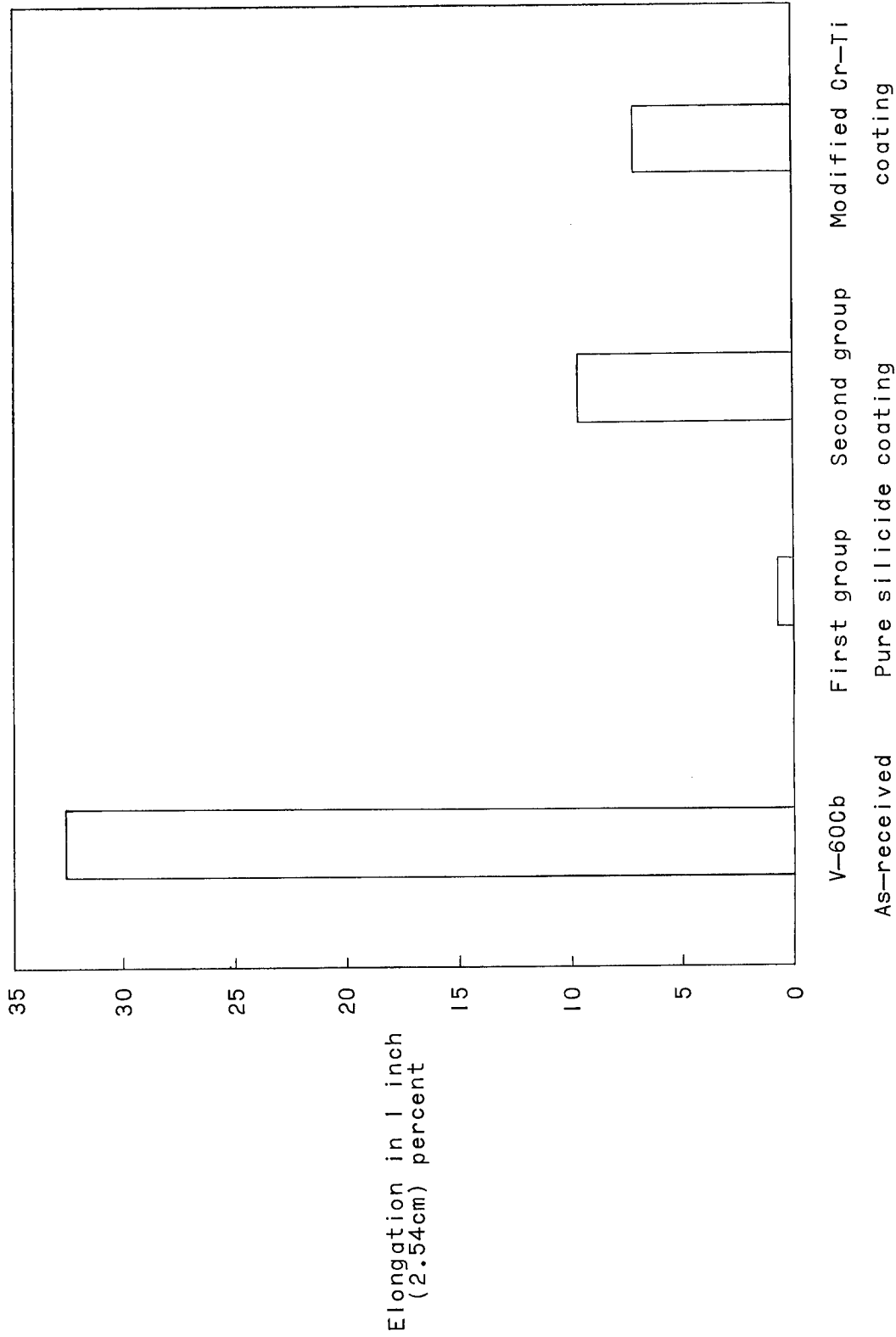


Figure 10.- Room-temperature elongation data for as-received and coated V-60Cb exposed for 24 hours at 2,000° F (1,370° K).

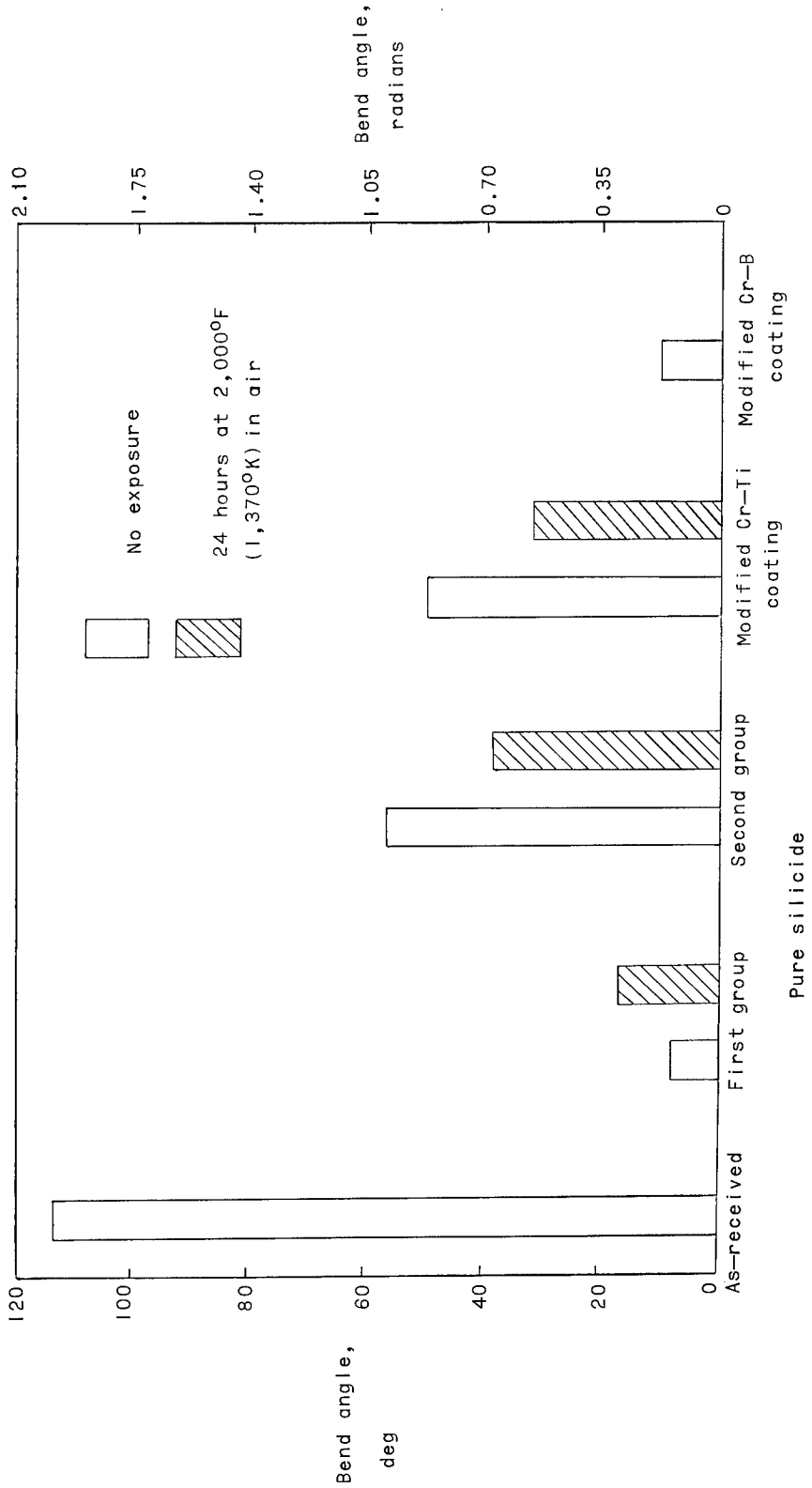


Figure 11.- Bend ductility for the as-received and coated V-60Cb alloy.

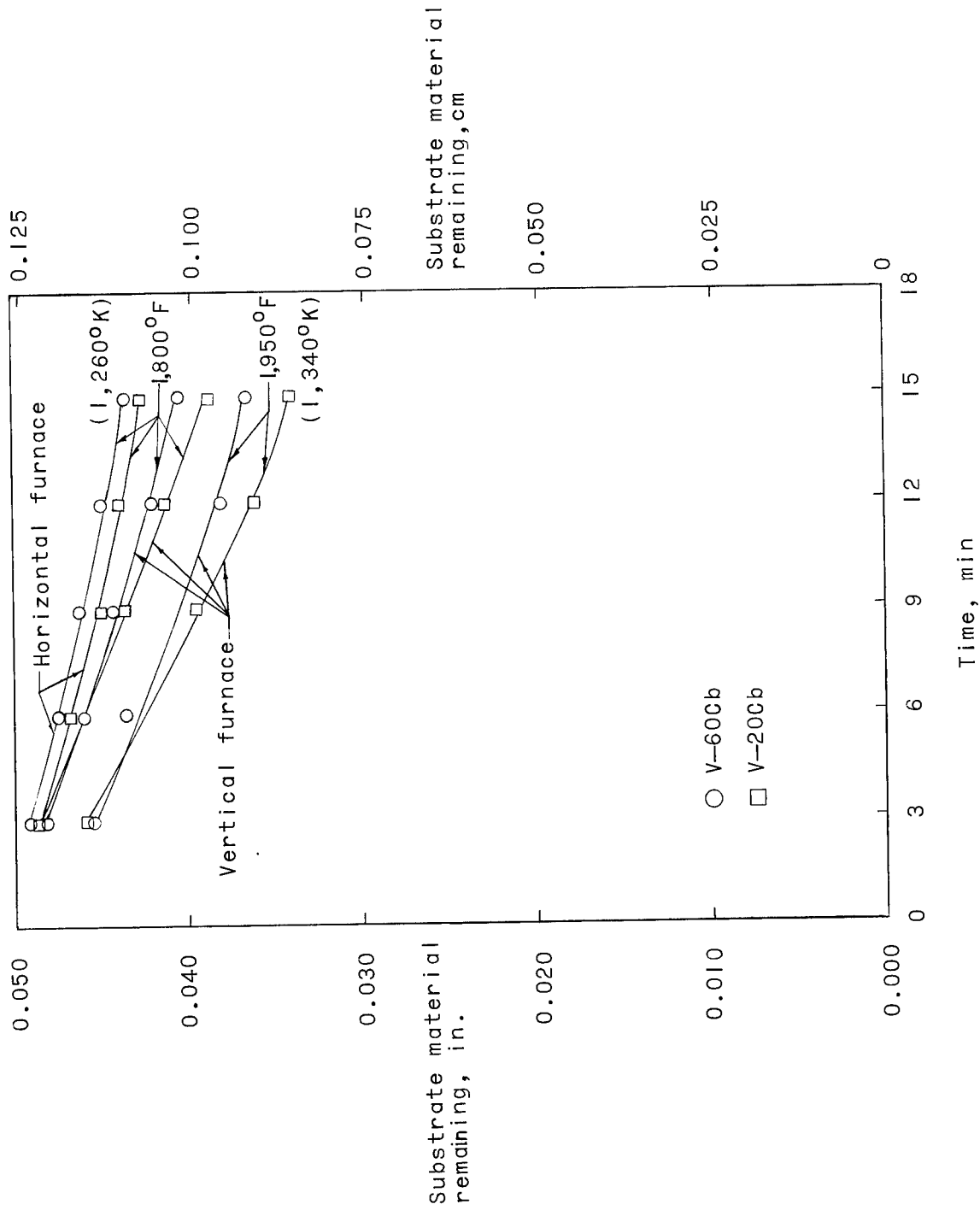
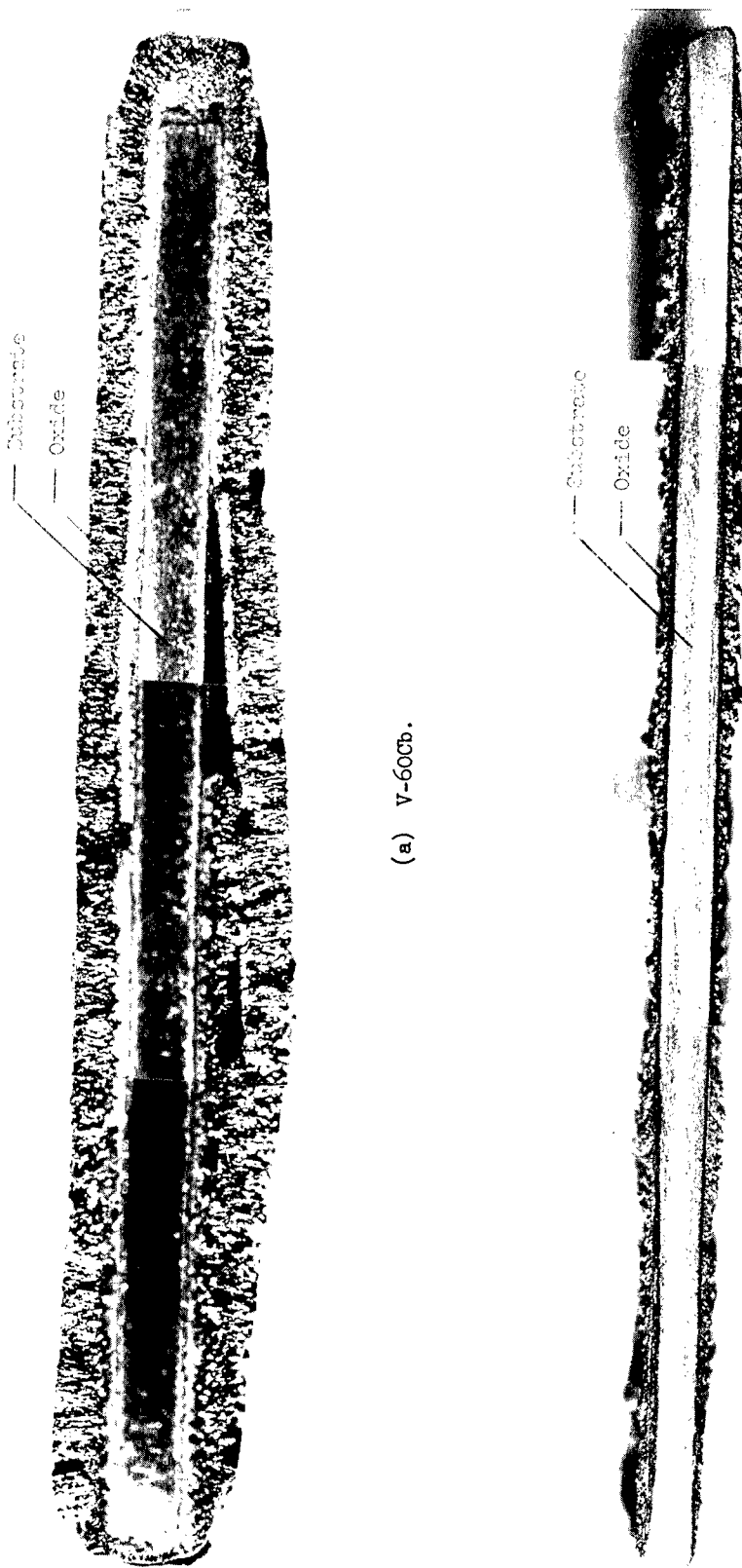


Figure 12.- Material remaining as a function of time for the oxidation characteristics of the as-received V-60Cb and V-20Cb alloys at temperatures of 1,800° F (1,260° K) and 1,950° F (1,340° K).



(a) V-60Cb.

(b) V-20Cb.

Figure 13.- Oxide formation on as-received V-60Cb and V-20Cb alloys exposed for 15 minutes at 1,950° F (1,340° K). X8.3.

L-64-8330

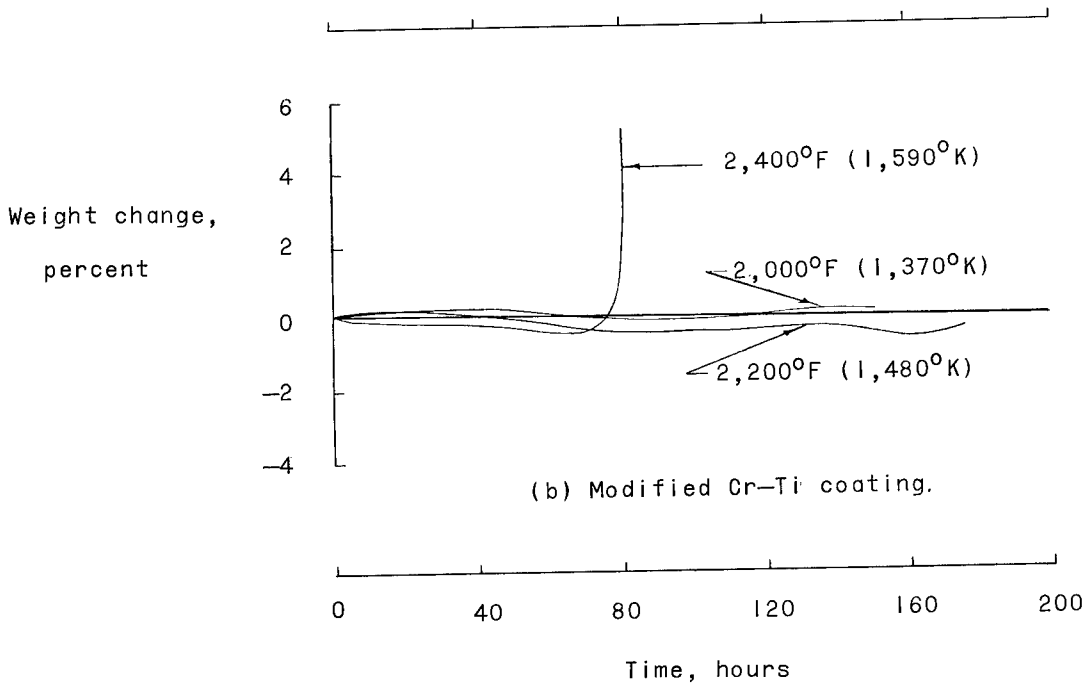
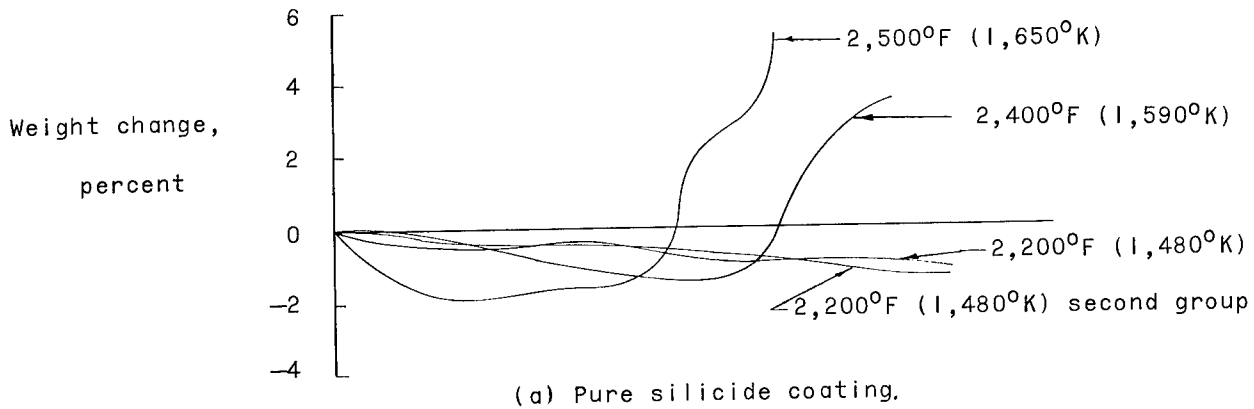


Figure 14.- Continuous oxidation curves for the coated V-60Cb alloy showing weight change as a function of time.

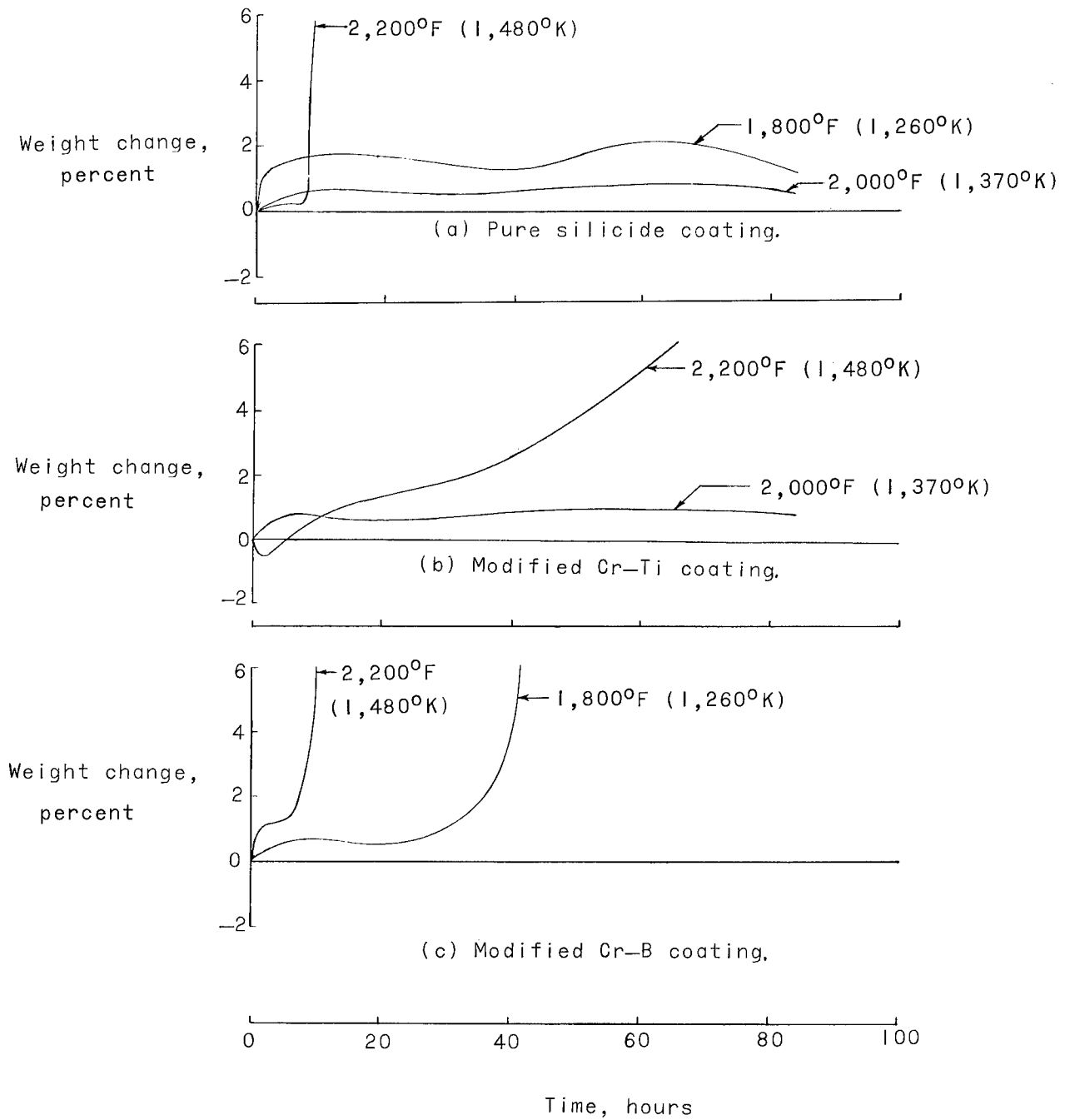


Figure 15.- Continuous oxidation curves for the coated V-20Cb alloy showing weight change as a function of time.

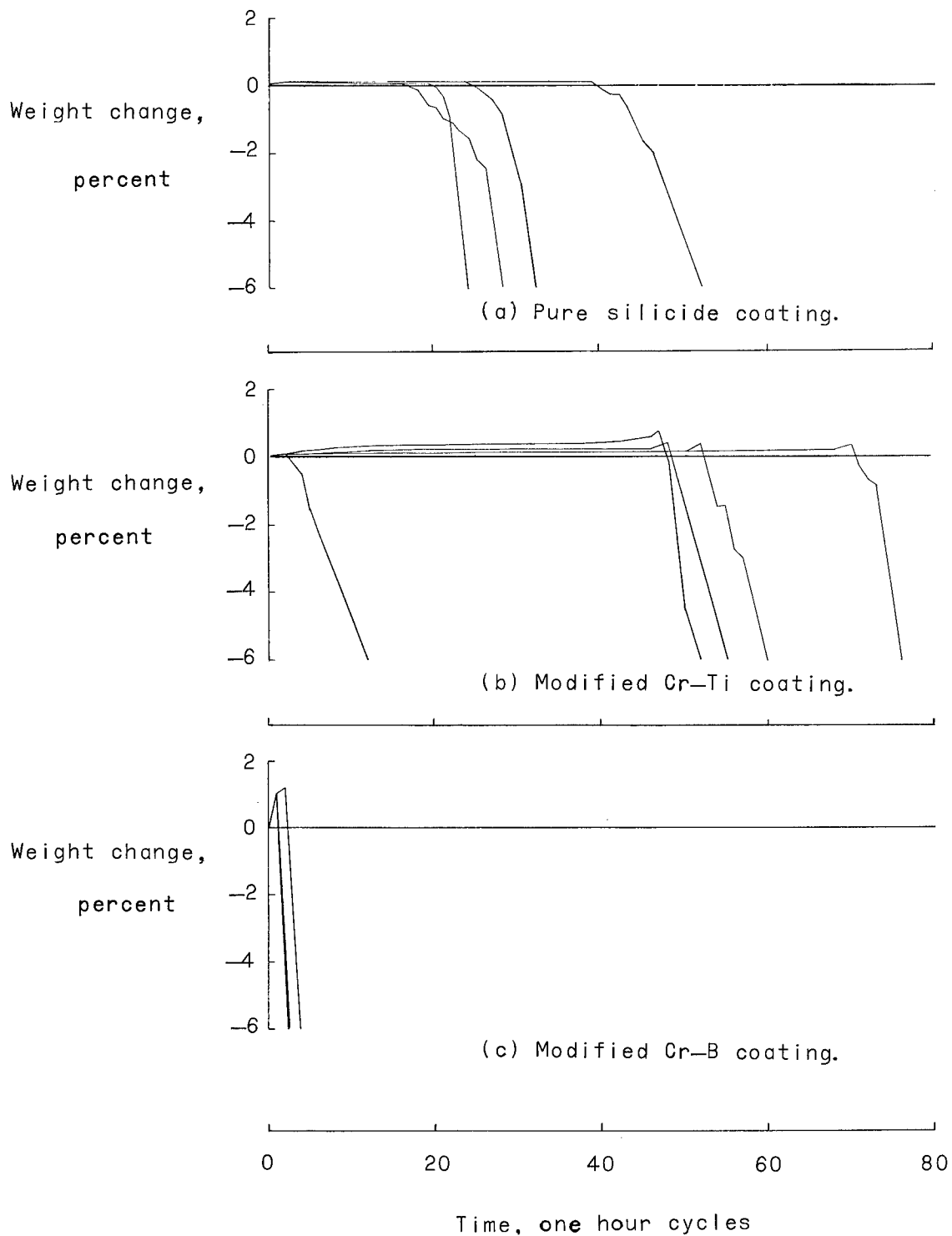


Figure 16.- Cyclic oxidation curves for the V-60Cb alloy at 2,300° F (1,540° K) showing weight change as a function of time.

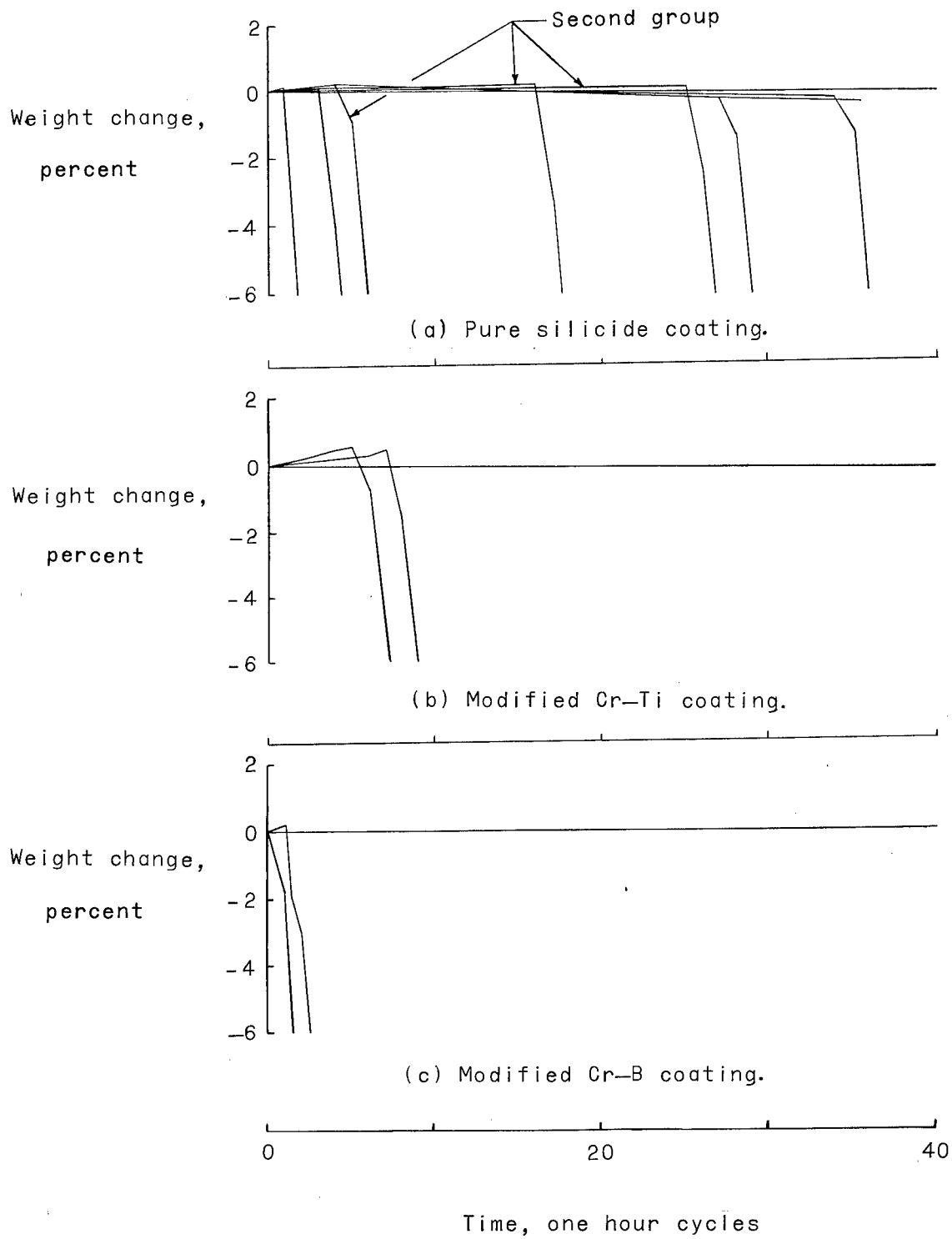


Figure 17.- Cyclic oxidation curves for the V-60Cb alloy at 2,500° F (1,650° K) showing weight change as a function of time.

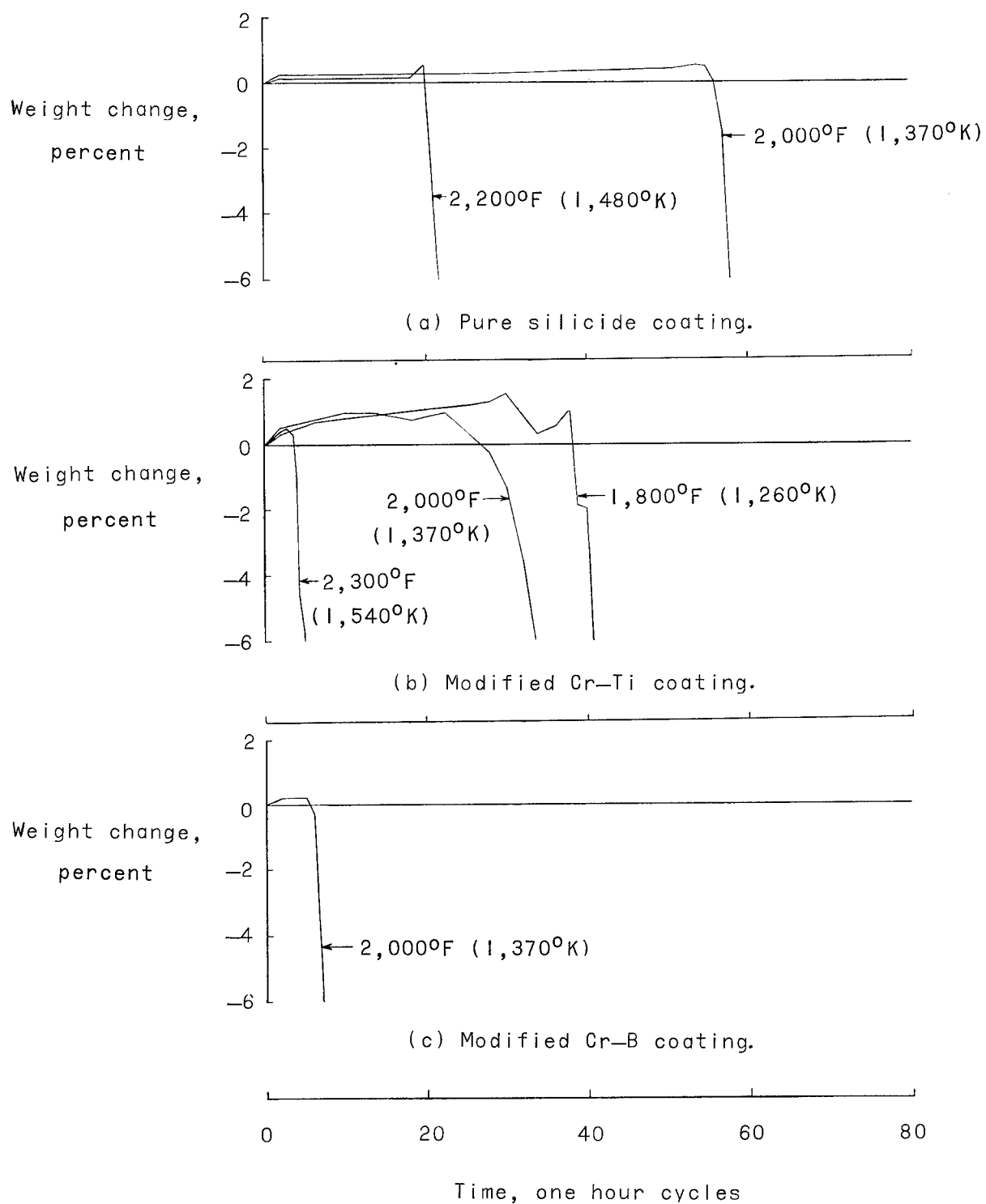
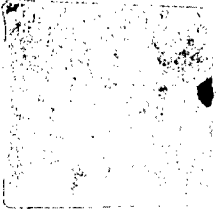


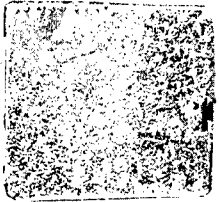
Figure 18.- Cyclic oxidation curves for the V-20Cb alloy showing weight change as a function of time.



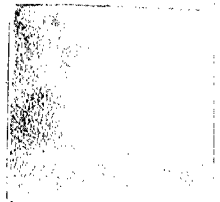
Time, hours: 0



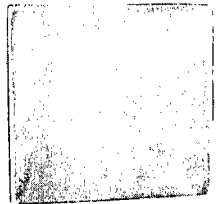
8



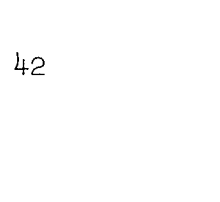
24



28

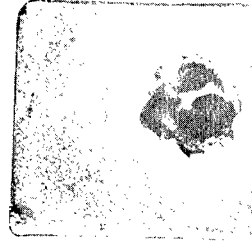


29

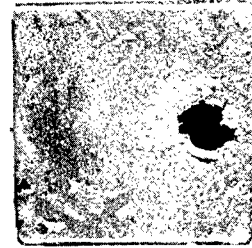


31

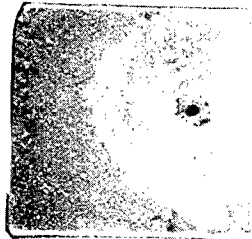
(a) Pure silicide.



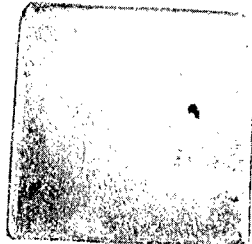
Time, hours: 0



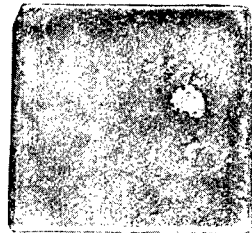
39



46



48



50

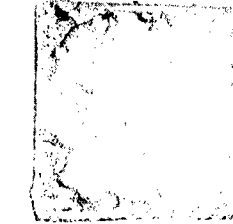


52

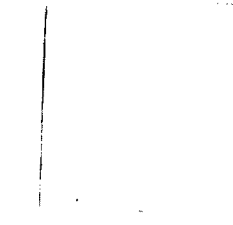
(b) Modified Cr-Ti coating.



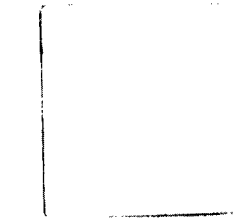
Time, hours: 0



1



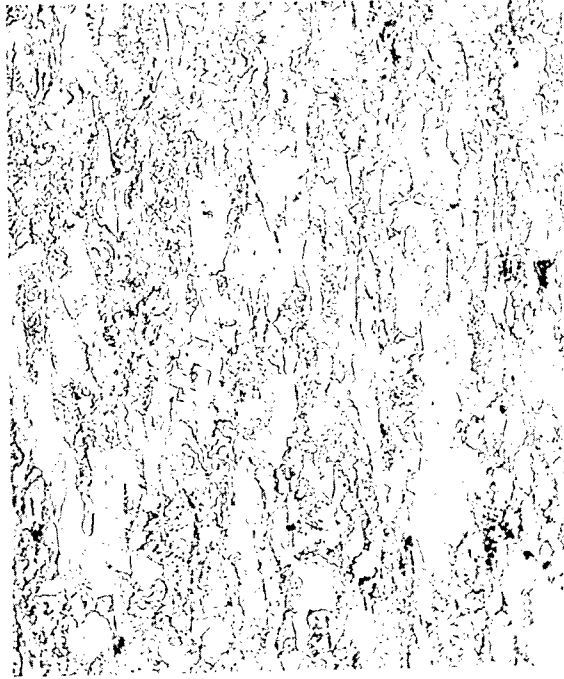
2



3

(c) Modified Cr-B coating.

Figure 19.- Photographs showing the progress of failures of the three silicide coatings studied during cyclic oxidation tests at 2,300° F (1,540° K) exposure.



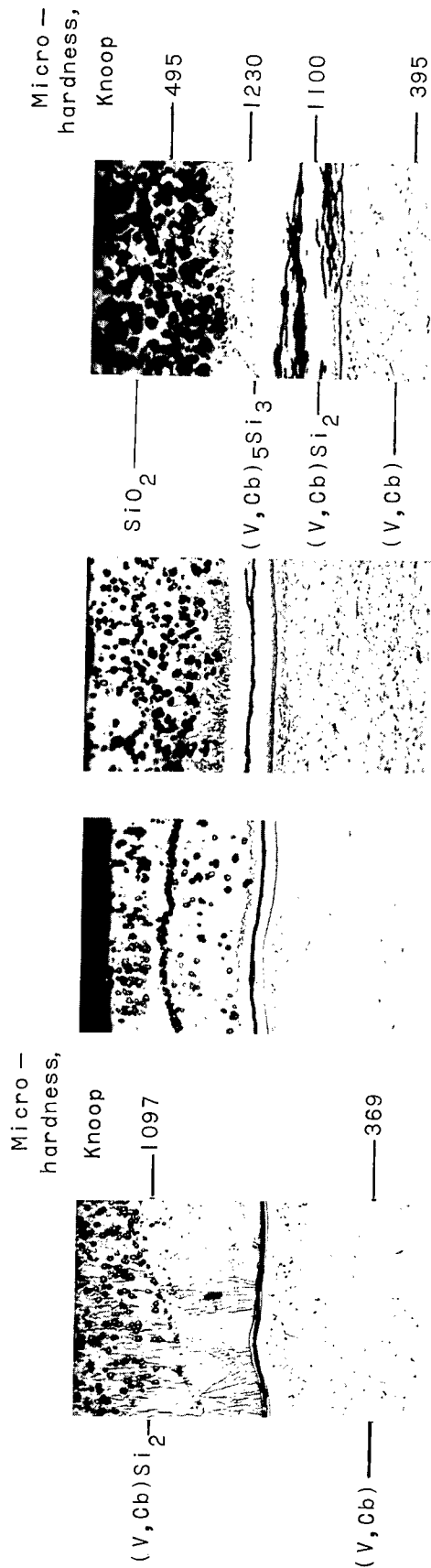
(b) V-20Cb.



(a) V-60Cb.

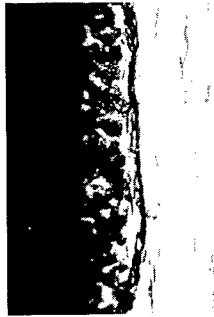
Figure 20.- Microstructure of as-received vanadium base alloys. X100.

I-64-8332



(a) 0 exposure. (b) 7 hours exposure. (c) 72 hours exposure. (d) 192 hours exposure. L-64-8333

Figure 21.- Photomicrographs showing the first group pure silicide coating "as-coated" and the effect of 2,400° F (1,590° K) exposure. X125.

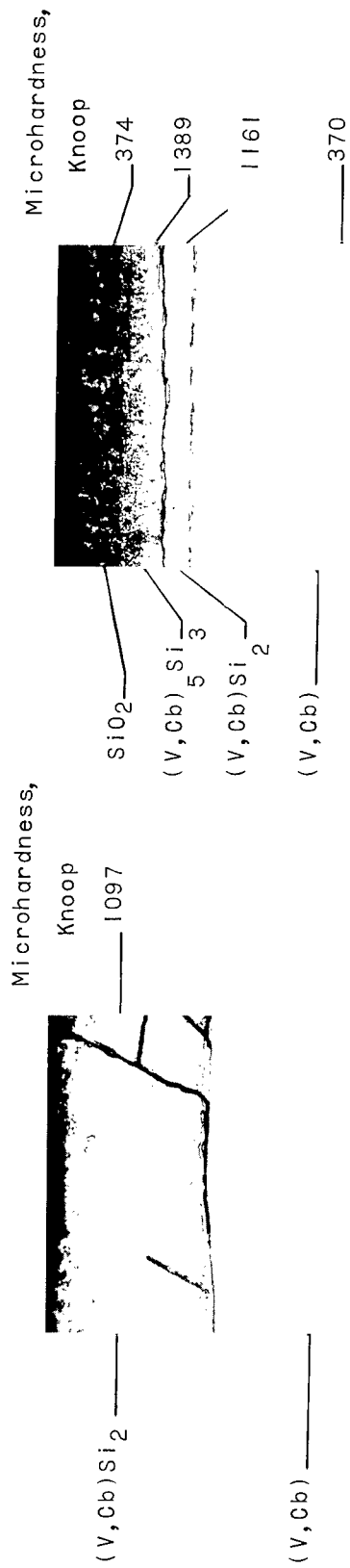


I-64-8334
(a) 7 hours exposure.

(b) 72 hours exposure.

(c) 192 hours exposure.

Figure 22.- Photomicrographs of the second group pure silicide coating showing the effect of 2,400° F (1,590° K) exposure. X125.

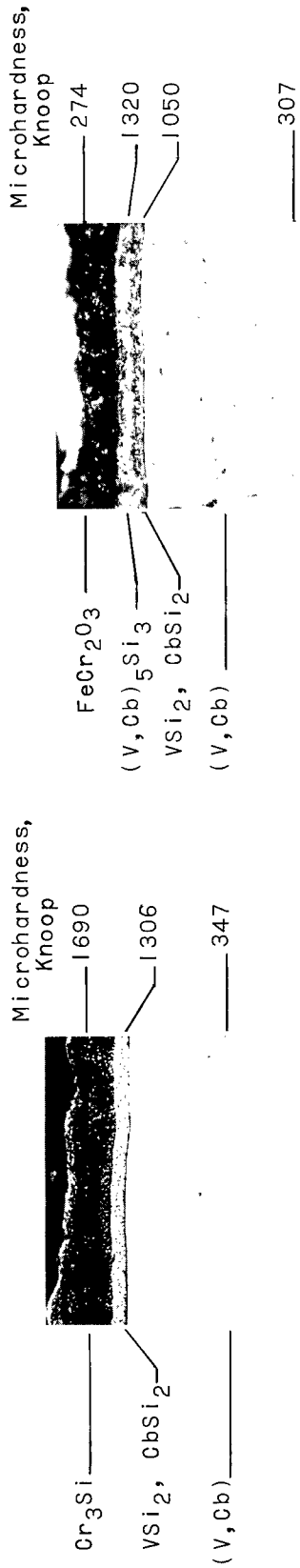


(a) 0 exposure.

(b) 132 hours exposure.

L-64-8335

Figure 25.- Photomicrographs showing the modified Cr-III silicide coating "as-coated" and the effect of 2,400° F (1,590° K) exposure. X125.



(a) 0 exposure.

(b) 1.5 hours exposure.

L-64-8336

Figure 24.- Photomicrographs showing the modified Cr-B silicide coating "as-coated" and the effect of 2,400° F (1,590° K) exposure. X125.

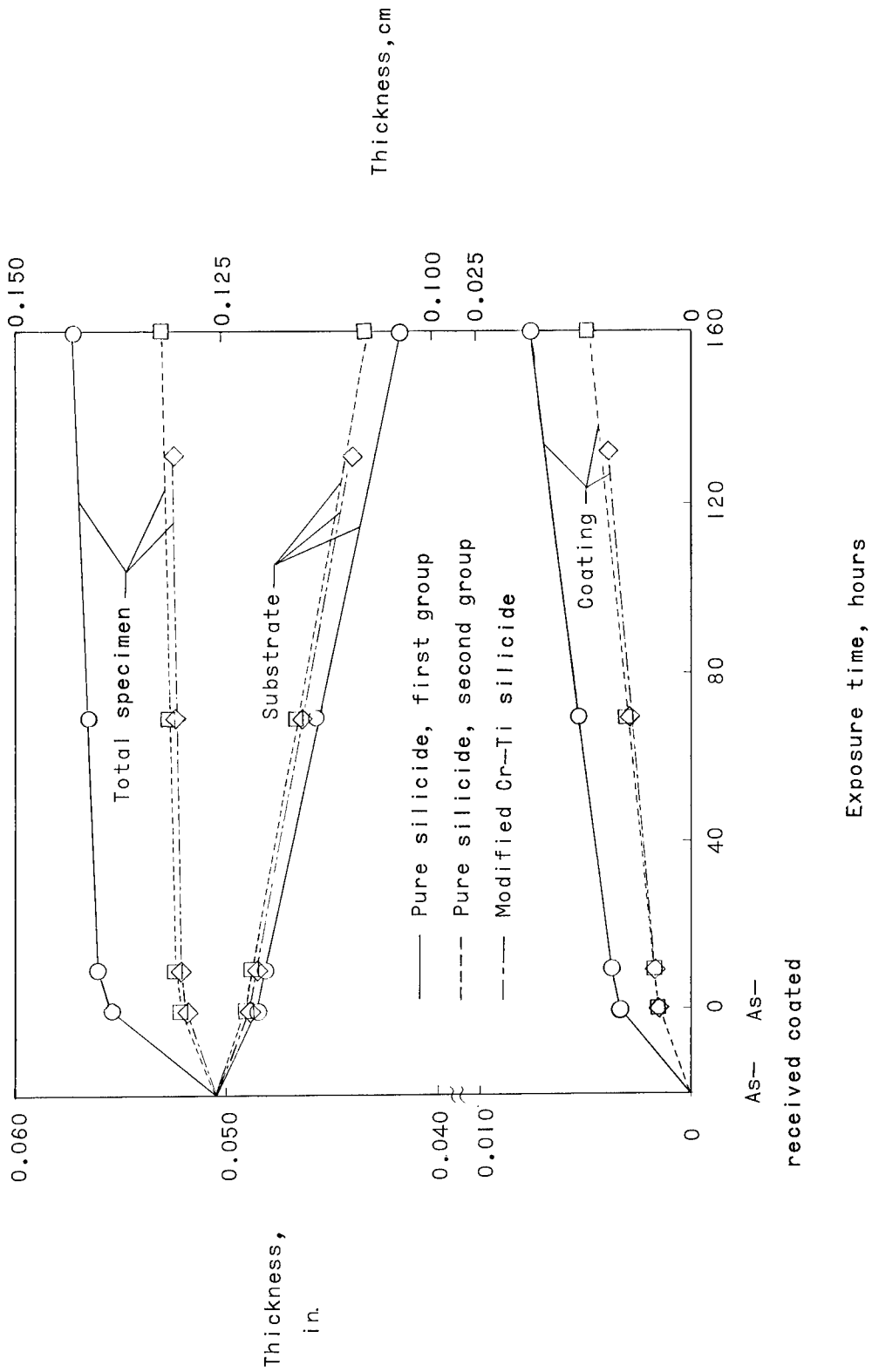


Figure 25.- Effect of coating diffusion on V-60Cb alloy at 2,400° F (1,590° K).

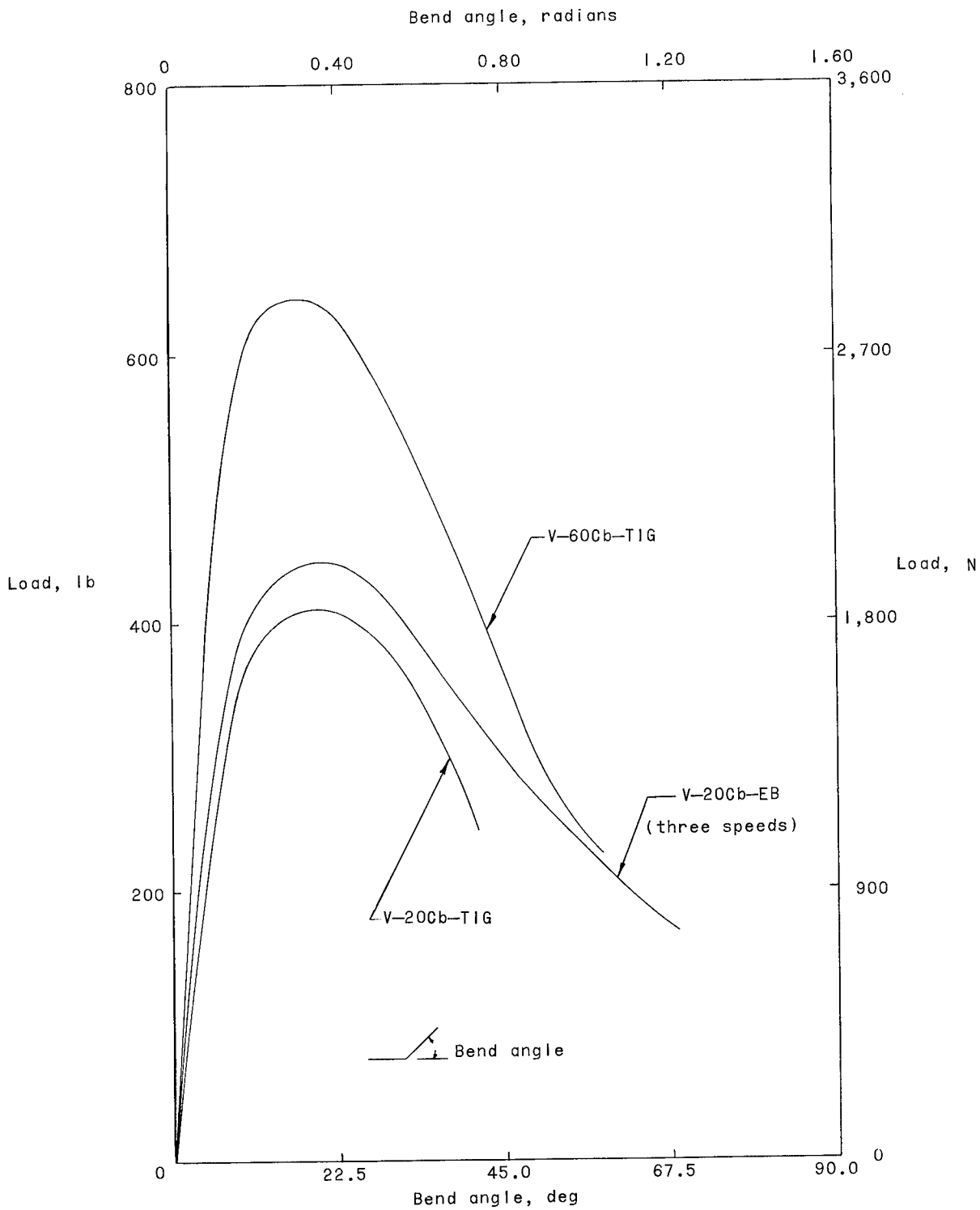
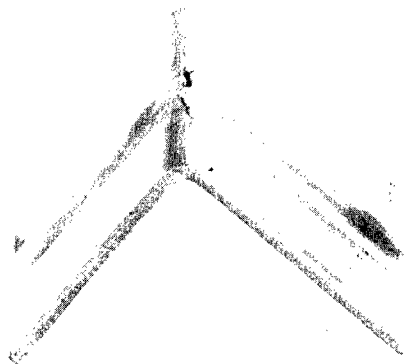


Figure 26.- Room-temperature bend strength as a function of bend angle for EB and TIG welded V-60Cb and V-20Cb alloys.

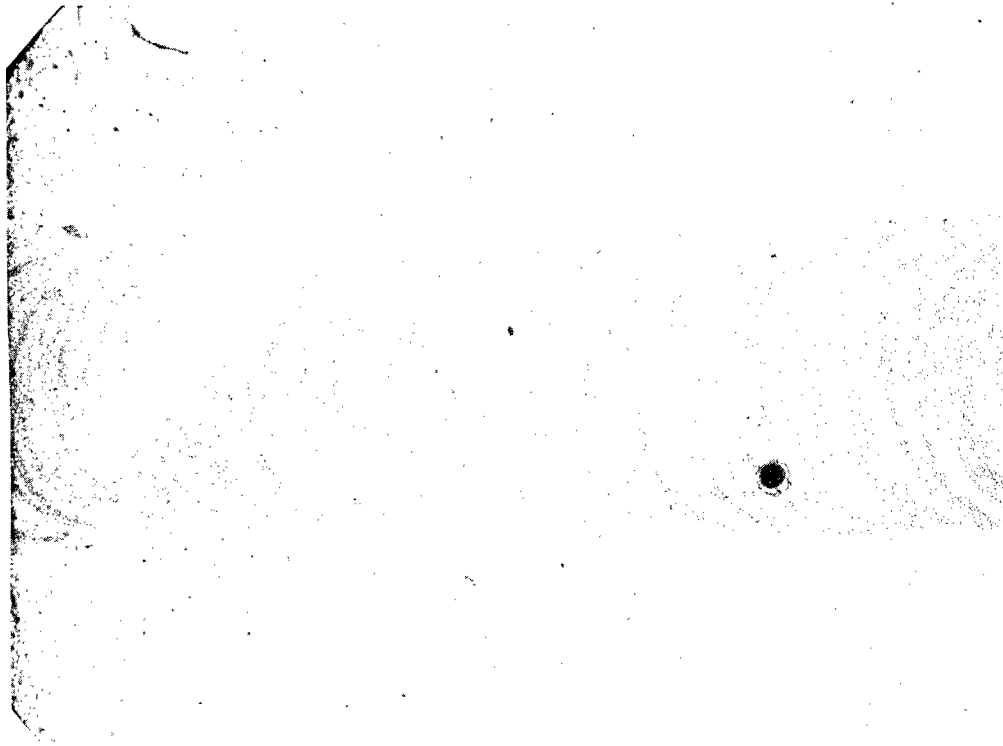


(a) V-60Cb.

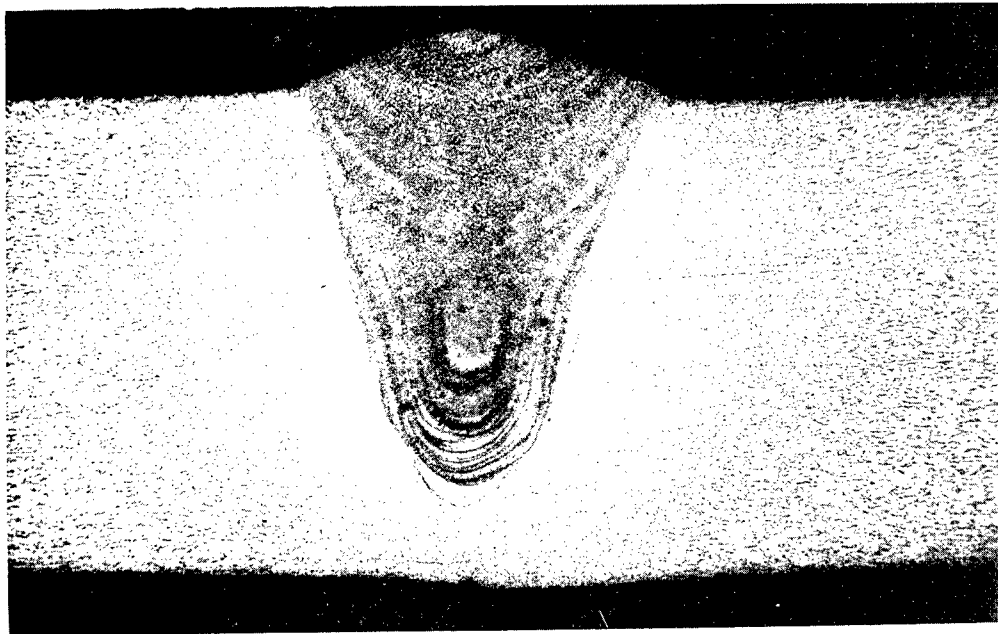
(b) V-20Cb.

Figure 27.- TIG weld specimens after bend.

L-64-2647



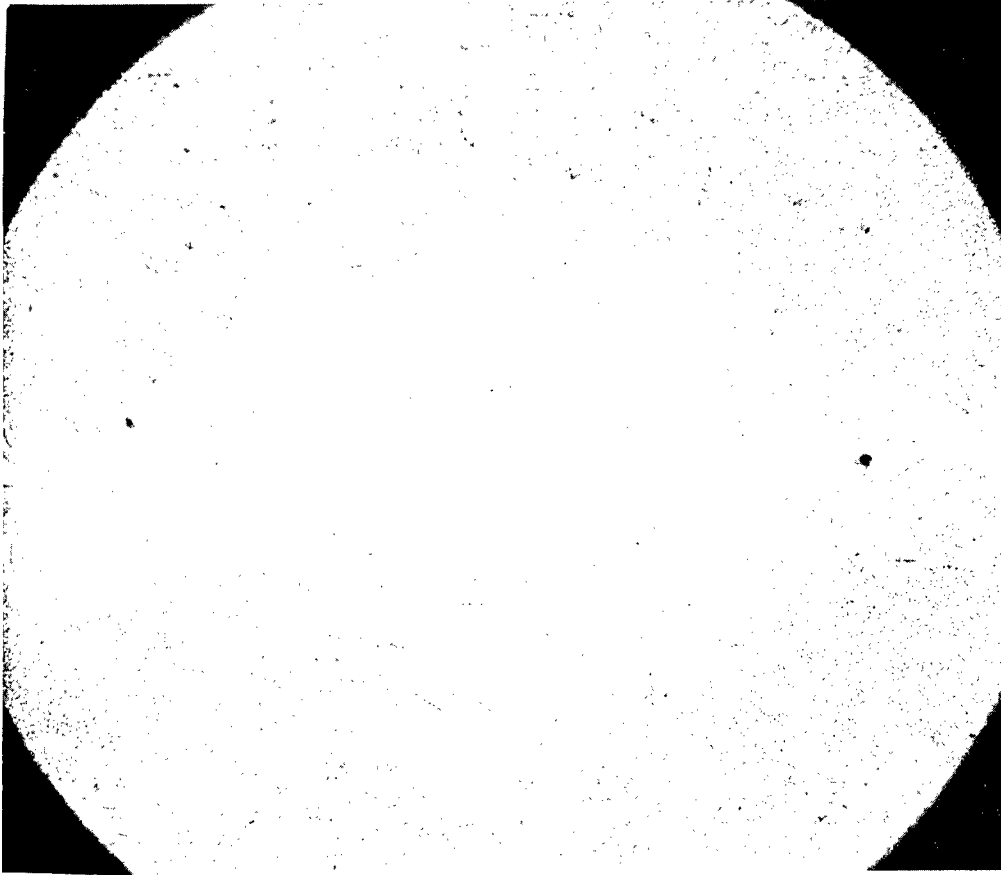
(a) Top view.



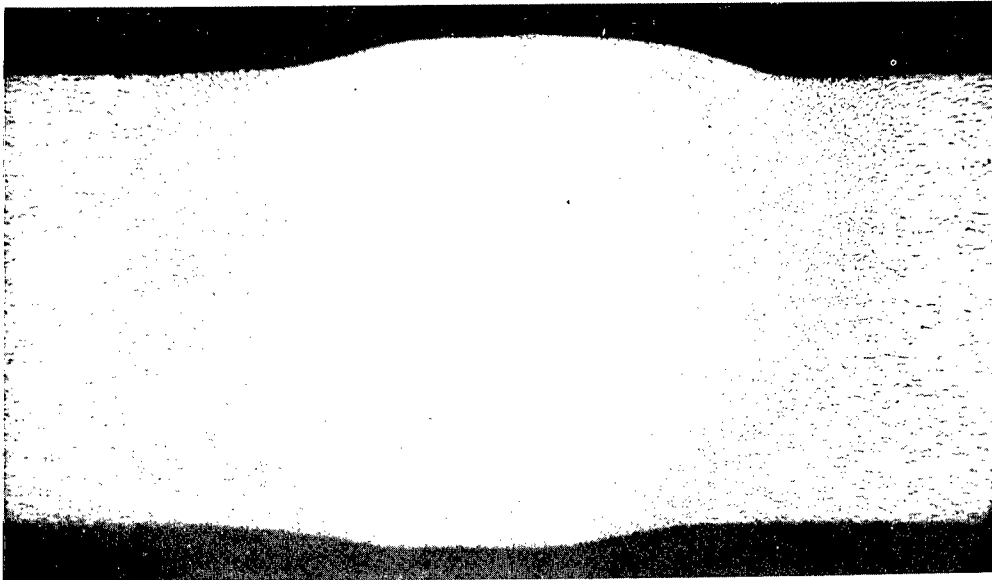
(b) Edge view.

L-64-8337

Figure 28.- Photomicrographs showing the top and edge view of V-60Cb material after electron-beam welding at 30 in./min (1.27 cm/s). x50.



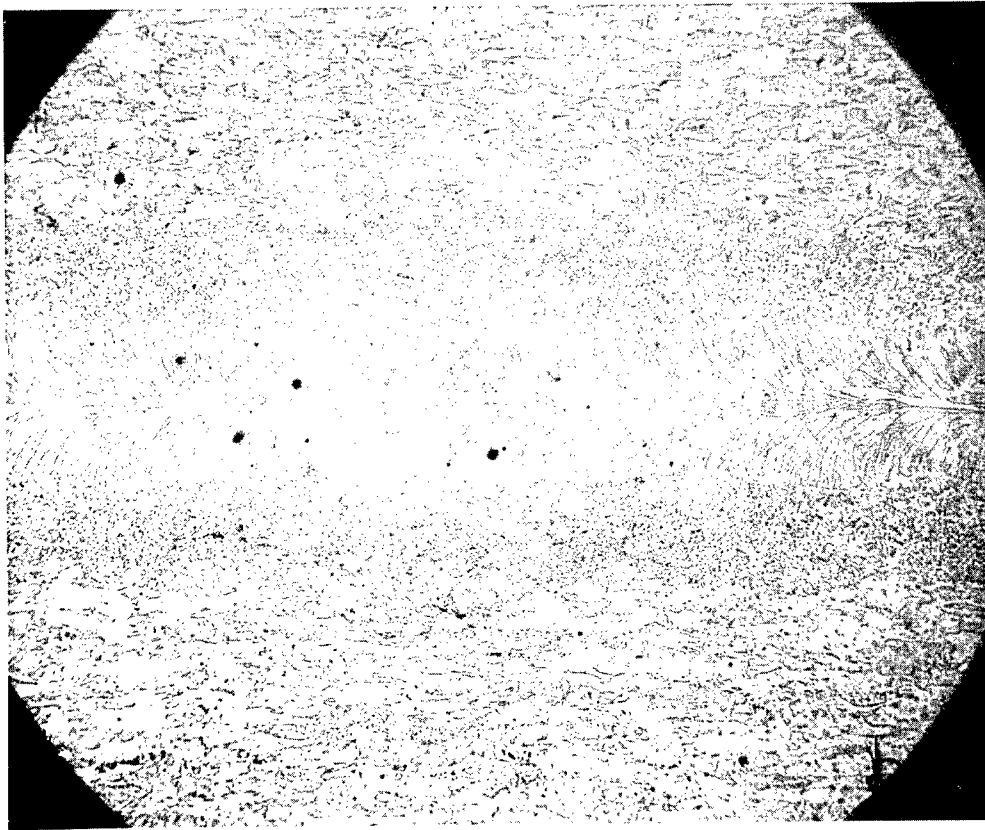
(a) Top view.



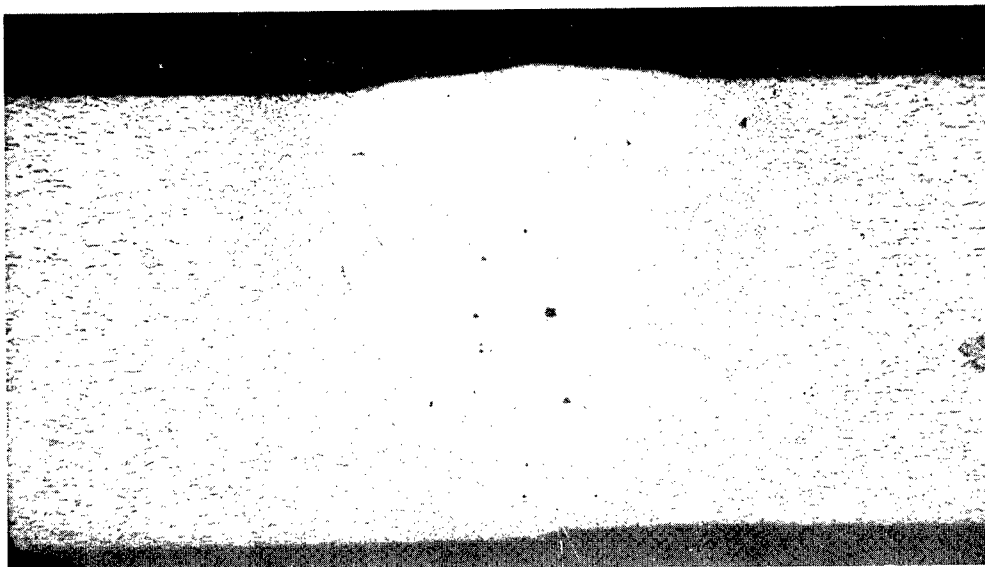
(b) Edge view.

L-64-8338

Figure 29.- Photomicrographs showing the top and edge view of V-20Cb material after electron-beam welding at 20 in./min (0.85 cm/s). $\times 50$.



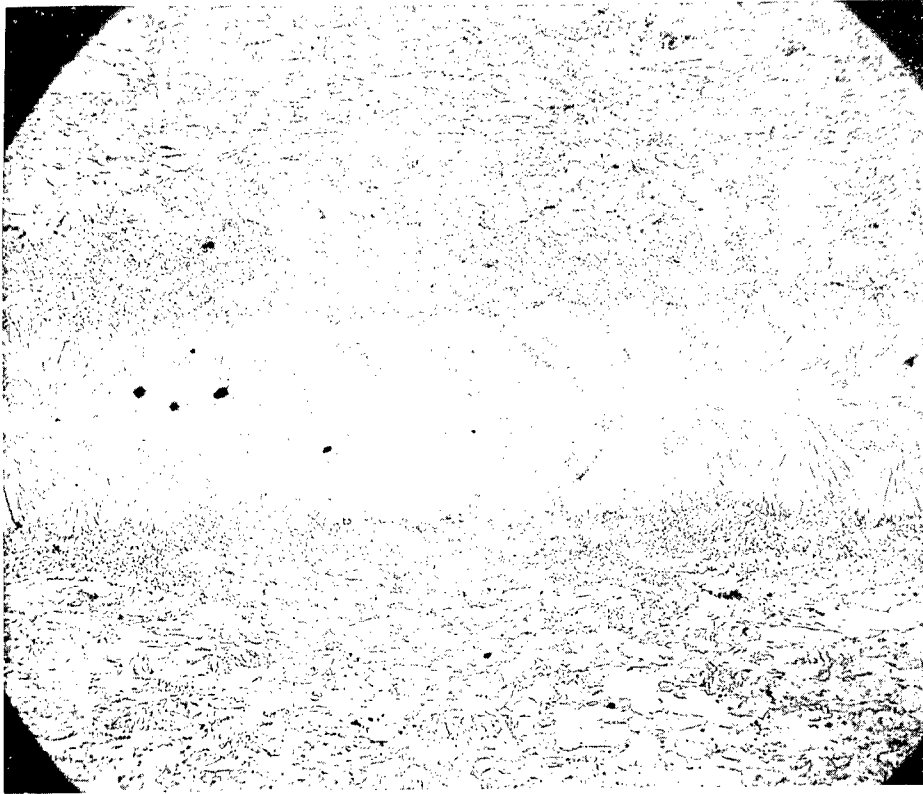
(a) Top view.



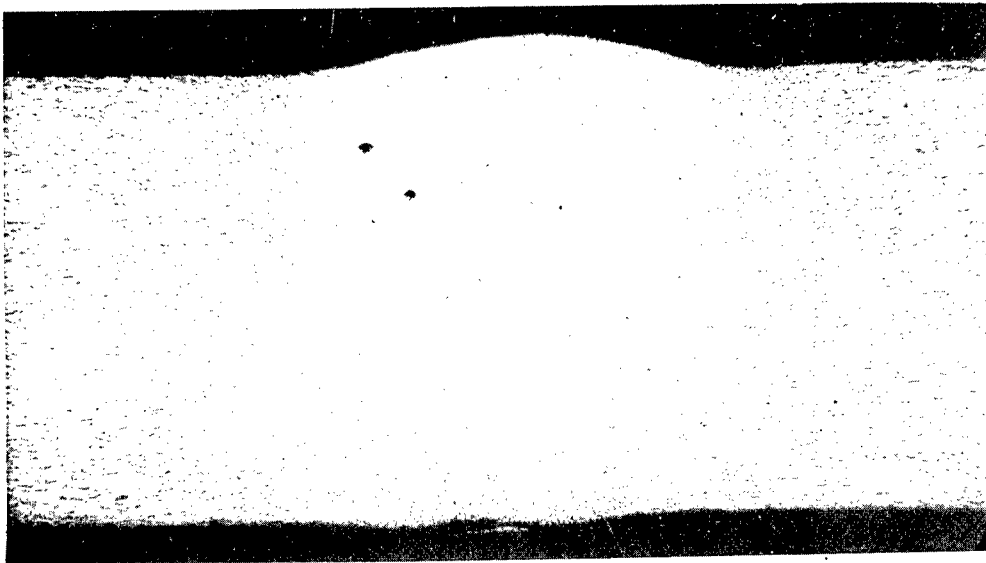
(b) Edge view.

L-64-8339

Figure 30.- Photomicrographs showing the top and edge view of V-200b material after electron-beam welding at 30 in./min (1.27 cm/s). $\times 50$.



(a) Top view.



(b) Edge view.

L-64-8340

Figure 31.- Photomicrographs showing the top and edge view of V-20Cb material after electron-beam welding at 40 in./min (1.69 cm/s). x50.

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—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

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