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VANADIUM
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FINAL REPORT

Issued November, 1969

NUCLEAR
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VANADIUM PURIFICATION
FINAL REPORT

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Contract AT(30-1)-3742
U. S. Atomic Energy Commission

Issued November, 1969

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Final Report

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ABSTRACT

The purpose of this program was the development of a commercially adaptable process to produce high purity vanadium. In addition, a V-15Cr-5Ti alloy was to be melted, fabricated into sheet, and the mechanical properties evaluated.

In this work, several ingots with a total interstitial content of <400 ppm were melted. Starting material, in all cases, was V_2O_5 which was aluminothermically reduced and subsequently electron beam melted to desired purity.

Alloying was performed by vacuum and inert gas arc melting composite electrodes of vanadium, chromium and titanium chips. Alloy sheet, 0.040-inch thick, was produced by upset or side forging followed by hot and warm rolling.

Tensile and stress-rupture tests performed on the sheet showed that the V-15Cr-5Ti alloy exhibits good strength characteristics without the added strengthening of a high interstitial content.

Weld studies show that the alloy is susceptible to embrittlement as a result of TIG welding, due to coring. Electron beam welding showed weld properties similar to parent metal properties when tensile and stress-rupture tested.

INTRODUCTION

The potential of vanadium and its alloys for use as fast reactor cladding and structural materials has been the subject of several reviews.⁽¹⁻³⁾ Nuclear considerations (primarily neutron economy) have provided the major impetus, but properties such as high-temperature strength, compatibility with liquid sodium and fabricability have also made these alloys suitable candidates for use in liquid metal cooled, fast breeder reactor systems.

One problem that is basic to the use of vanadium alloys, however, is control of their interstitial impurity content. This difficulty is common to the body-centered cubic refractory metals, and its ramifications are well known to workers in this field.

Normally, an increase in interstitial content results in embrittlement that is evidenced by higher tensile strength and lower elongation of the metal. For example, the room temperature tensile strength of niobium containing 1200 ppm oxygen has been determined to be twice that of similar material containing only 200 ppm oxygen.⁽⁴⁾ Similarly, the room temperature tensile strength of vanadium increases from 20,000 to 60,000 psi as the metal's nitrogen content is increased from 50 to 1160 ppm.⁽⁵⁾ These results indicate that the mechanical properties of vanadium alloys can probably be optimized by proper control of their interstitial levels. A systematic approach to this control requires that high purity alloys be produced for evaluation against intentionally contaminated materials.

Interstitials also play a role in the corrosion behavior of the refractory metals. DiStefano and Hoffman⁽⁶⁾, who studied the compatibility of the Nb-Li system, demonstrated that the attack on niobium by lithium increases as a function of the oxygen present in the niobium prior to testing. Lithium has a greater affinity for oxygen than niobium and scavenges it from the cladding. Potassium and sodium, on the other hand, lose oxygen to niobium which can become embrittled as a result. Similar behavior can be expected from vanadium alloys and, once again, its study requires a source of high purity vanadium to compare with contaminated metal.

Another aspect that must not be overlooked is the compatibility of cladding alloys with the oxides, carbides and nitrides that are presently proposed as fuels for fast breeder reactors. At the surface temperatures which are currently contemplated, diffusion of interstitials across the fuel-cladding interface could possibly lead to changes in fuel stoichiometry or embrittlement of the cladding. The development of low-interstitial vanadium alloys is necessary in order that this interaction can be adequately investigated.

This program was undertaken with the dual objectives of (1) development of a commercial process for producing high purity vanadium, and (2) determination of the mechanical properties and welding characteristics of an alloy fabricated from high purity vanadium.

The vanadium purification program was divided into three tasks.

Task I, Melting Studies*

This task developed a commercial melting process for producing high purity vanadium.

Task II, Fabrication and Mechanical Testing

After a melting process was developed, a vanadium alloy, V-15 w/o Cr-5 w/o Ti, was melted, fabricated into sheet and tested to determine its mechanical properties.

Task III, Weldability

This task evaluated the welding characteristics of the V-15 w/o Cr-5 w/o Ti sheet processed under Task II.

* All melting and fabrication studies were performed under subcontract at Wah Chang Albany, Albany, Oregon.

I TASK I, MELTING STUDIES

A. Experimental Effort

The objective of this task was to produce, by a commercially adaptable process, a 100-pound ingot of vanadium with the following target impurity levels:

<u>Element</u>	<u>Impurity Level, ppm</u>	
oxygen	< 300	} < 500 ppm total interstitial content
nitrogen	< 100	
carbon	< 100	
hydrogen	-----	
silicon	< 500	
aluminum	< 1000	
iron	< 1000	

The original work under this task was aimed at purifying relatively impure vanadium metal granules. The initial approach was to dope the granules by arc melting them with a gettering agent. Subsequent electron beam melting under high vacuum would remove the oxidized gettering agent and other impurity elements.

Three gettering agents were evaluated: yttrium, aluminum and silicon. Of the three, yttrium was superior under both argon arc melting and vacuum electron beam melting. Yttrium appeared to work as a deoxidant not only by formation of an yttrium oxide, but also through solution of oxygen in the yttrium metal phase which remained insoluble in the vanadium melt. Consequently, both phases were available at the top surface of the pool for removal through volatilization by the electron beam. Tests on button melts indicated none of the gettering agents were effective in the gettering of

nitrogen, as was seen by other investigators.⁽⁷⁾ Accordingly, efforts were aimed at obtaining a starting material as free as possible from nitrogen, with a minimum of oxygen, carbon and hydrogen contamination being a secondary consideration.

Attempts were made to procure starting material containing less than 200 ppm nitrogen, but without success. Therefore, an aluminothermic reduction of V_2O_5 was initiated based on Carlson's work at Ames Laboratory, Iowa State University⁽⁸⁾ and Wah Chang Albany Corporation's experience with columbium reduction.

The vessel used for the reduction is schematically shown in figure 1. The steel vessel is lined with alumina at the bottom where the reduction takes place. The charge is a mixture of V_2O_5 and aluminum granules. Ignition was initiated by electrical means after evacuating and back filling with helium, three to five times.

A series of five ingots was melted, number 5 satisfying all contract requirements. Table I summarizes the melting procedure of each melt. More detailed melting and chemistry data are listed in Appendix A.

Figure 1
Schematic Of The Aluminothemic Reduction
Vessel When Loaded

Table I.
Melting Purification Schedule

Ingot Series No.	Wt. of Reductions lbs	Pertinent Chem of Reductions	Purification Process							
1	wt. 224	O-9,770 ppm N-200 ppm C-115 ppm Si-3,300 ppm	E.B. melted wt. 157#	upper 2/3 high in N ₂ & O ₂ , arc melted with 1 1/4% yttrium wt. 79#	both halves FINAL E.B. melted wt. 109# interstitials <500 ppm; Si-4,270 ppm	Remarks: Silicon contamination from starting materials (Al and V ₂ O ₅) plus steel of reduction vessel.				
2.	wt. 218 using Low Si Aluminum	O-6,000 ppm N-80 ppm C-540 ppm Si-430 ppm Al-0.75%	E.B. melted wt. 167# E.B. furnace leaked causing high hardness in one portion	E.B. melted wt. 82# O-710 ppm	E.B. melted wt. 63# O-570 ppm	arc melted with 1% Y wt. 62#	2nd arc melt with 1/2% Y wt. 58#	E.B. melted wt. 35# O-2,700 ppm N-500 ppm C-210 ppm Si-500 ppm	FINAL E.B. melted wt. 30# O-410 ppm N-280 ppm C-125 ppm Si-415 ppm	Remarks: Low yield due to high oxygen content in reduction resulting from incomplete reaction with coarse aluminum granules.
3.	wt. 342 using excess aluminum	O-5,250 ppm N-96 ppm C-100 ppm Si-360 ppm Al-1.9%	E.B. melted, malfunction resulted in two ingots 139# & 94# O-2,355 ppm	E.B. melted wt. 204# O-440 ppm Si-870 ppm	Arc melted 150 lbs with 0.06% Y. wt. 150#	FINAL E.B. melted malfunction resulted in two ingots wt 117# total O-260 N-190 C-95 H-5 Si-560 Al-<100 Fe-200	REMARKS: The excess aluminum acted as an intrinsic getter. If sufficient aluminum is left in the reductions, the yttrium arc dope could be eliminated.			

(Continued)

Table I (Cont'd)

Ingot Series No.	Wt. of Reductions lbs	Pertinent Chem of Reductions	Purification Process		
4	180 using excess aluminum	O-3,350 ppm N-170 ppm C-79 ppm Si-240 ppm Al-15.5%	E.B. melted into two ingots wt. 52# & 68# O-330 ppm Si-330 ppm Al-0.83%	Final E.B. melted wt. 79# O-200 ppm N-160 ppm C-150 ppm H-5 ppm Si-400 ppm Al-440 ppm Fe-90 ppm	<u>REMARKS:</u> The lower 1/3 showed higher contamination of N & Si than target levels.
5	202 using excess aluminum	O-3,210 N-63 C-52 Si-318 Al-13.6%	E.B. melted into two ingots wt. 148# O-224 ppm N-93 ppm C-55 ppm Si-373 ppm Al-0.17%	E.B. melted into two ingots wt. 124# O-296 ppm N-95 ppm C-68 ppm Si-240 ppm Al-448 ppm	E.B. melted Wt. 102# O-178 ppm N-99 ppm C-50 ppm H-9 ppm Si-318 ppm Al-<20 ppm 336 ppm total interstitial content.

B. Discussion of Results

The aluminothermic reduction under vacuum is an economical way to produce high purity vanadium from V_2O_5 , as compared to electrolytic, iodide cell or calcium reduction. However, precautions should be made to prevent any nitrogen or silicon contamination from starting materials, i.e., V_2O_5 and aluminum, or the Al_2O_3 refractory lining of the reduction vessel.

Also, the aluminum - V_2O_5 charge should contain 10 percent to 12 percent excess aluminum over the theoretical amount required by the following reduction formula:

This will provide intrinsic gettering after the first electron beam melt, reducing the oxygen content without the addition of a gettering agent during subsequent melts.

The electron beam furnace used for this program, shown in figure 2, is of a deflected beam or transverse gun design. The difference between the conventional electron beam furnace and the deflected beam furnace is shown schematically in figures 3 and 4. The path of the electron beam in the conventional furnace is a straight path as shown in figure 3. The path of the deflected beam-type is an arc, controlled by a magnetic field. The deflected beam will not be hindered by the possible plasma buildup around the crucible and, therefore, can tolerate more impurities in the feed stock. This feature eliminated the de-aluminization sintering step that Carlson at Ames Laboratories found necessary.

The effects of vacuum electron beam melting on impurities are discussed by element below.

1. Hydrogen (Interstitial)

Although hydrogen pickup has a detrimental effect on vanadium to a great extent, it is easily eliminated under vacuum melting. All hydrogen contents were less than 10 ppm in the five series of ingots.

Figure 2. Wah Chang Albany Corporation S4 Electron Beam Furnace with a capacity of 450 kw and being able to melt an ingot up to 9 inches diameter by 80 inches long. Diffusion pumps are shown in the background

Figure 3
Conventional Electron Gun Configuration

Figure 4
Sketch Showing The Reflected Electron Beam
For Vanadium Melting

2. Carbon (Interstitial)

In most cases of vacuum melting, carbon will react with oxygen and be eliminated. However, under this program, the stronger reduction from aluminum hinders the carbon-oxygen reaction. As shown in figure 5, the carbon content was essentially constant throughout the melts for each series. The increase on the fourth and fifth electron beam melts for series II is probably due to carbon pickup during the arc melting that preceded the fourth electron beam melt. The high carbon content for the first electron beam melt for series III is attributed to sampling or analysis error.

3. Nitrogen (Interstitial)

The nitrogen content in vanadium tends to increase under the effect of electron beam melting, as shown in figure 6. However, a proper melting rate, better vacuum and lower power applied in order to reduce base metal loss by volatilization will minimize the increase. Series V shows the lowest nitrogen increase, being melted at 3 inches per hour with lower power.

4. Oxygen (Interstitial)

The oxygen content in vanadium is greatly reduced by electron beam melting. This is especially true in the presence of a scavenger such as aluminum or yttrium. As shown in figure 7, it is apparent that the greatest reduction occurred for all the first melts having large amounts of excess aluminum in the vanadium reductions. Also, a high reduction is seen during electron beam melting after arc doping with yttrium. Ten to twelve percent excess aluminum should be included in the reductions, in order that 0.5 to 1.0 percent will remain for gettering during subsequent melting.

5. Silicon

Silicon could not be reduced by employing a gettering agent during vacuum melting. Figure 8 shows the constant level of silicon content from reductions to final ingots. Silicon and vanadium have similar volatility.

Figure 5
Effect Of Vacuum Melting On The Carbon Content In Vanadium

Figure 6
Effect Of Vacuum Melting On The Nitrogen Content In Vanadium

Figure 7
Effect Of Vacuum Melting On The Oxygen Content In Vanadium

Figure 8
Effect Of Vacuum Melting On The Si Content In Vanadium

It is apparent that a selection of starting materials with low silicon content is essential. Also, special precautions should be taken to prevent the molten reductions from coming in contact with impure refractory linings or the steel walls of the reduction vessel.

6. Aluminum

Aluminum is used in this program, not only as a reducing agent, but also as a scavenger in electron beam melting. Aluminum has a vapor pressure 3 orders of magnitude greater than vanadium at 2000°C. Figure 9 shows the elimination of aluminum as a function of melting sequence.

7. Other Impurities

Other impurities included in the reductions, such as iron and copper, will usually be reduced to tolerable levels after the second electron beam melt. Other common or refractory metal elements are not usually contained in the vanadium starting materials.

The percentage yield for each series is plotted in figure 10. Series IV and V have the highest yields due to a proper melting rate (3 in./hr) and a low power setting (< 100 kilowatts). This favors the elimination of aluminum rather than the evaporation of vanadium. The final yield of the second electron beam melt of series IV was 72 percent. This could be raised to 85 percent with optimum aluminum contents and melting power.

Ingot hardness versus total interstitial content for series II through V are plotted in figure 11. Series I is excluded from the curve due to the high metallic impurity contents. Also excluded are the last electron beam melts of series II and III because their interstitial analyses were not representative of the matrix due to yttrium-rich inclusions.

The effects of total interstitial level on the hardness is apparent and, at low levels (around 500 ppm), the effects are drastic. Similar results were reported in the findings of Matheur et al plus others (9, 10, 11, 12 and 13). A summary of hardness and chemical compositions is tabulated in table II.

Figure 9
Effect Of Vacuum Melting On The Al Content In Vanadium

Figure 10
Plot Of Percent Of Yield vs Melts

Figure 11
Effect Of Total Interstitial On The Ingot Hardness Of Vanadium

Table II. Summary of Hardness and Interstitial and Si Contents
of All Series E.B. Ingots

	Total Interstitial ppm	Si%	Hardness, BHN
Series I			
1st melt	1000	0.395	190
2nd melt	570	0.427	188
Series II			
1st	8000	0.032	230
2nd	3464	0.031	214
3rd	2220	0.028	182
4th	3210	0.050	95
5th	810	0.042	96
Series III			
1st	3020	0.051	186
2nd	1410	0.054	176
3rd	550	0.056	131
Series IV			
1st	760	0.033	137
2nd	510	0.040	127
Series V			
1st	377	0.037	96
2nd	464	0.024	74
3rd	336	0.032	72

Cold rolled sheet samples from series V were heat treated to determine the effect of annealing temperature on the hardness of vanadium. The results are plotted in figure 12, showing a rapid decrease in hardness at 760°C. The metal starts to recrystallize at 870°C and is fully recrystallized at 980°C. Complete recrystallization occurs at 1200°C for commercial vanadium.

The microstructure of a vanadium reduction from series I is shown in figure 13. The matrix is the aluminum-vanadium alloy with a dispersed second phase of impurities. The larger black inclusions are mainly oxides. The microstructure of the first E.B. (electron beam) melt of this ingot is shown in figure 14; the yttrium-doped arc melt in figure 15, and the second E.B. melt in figure 16. Notice the finer precipitates in the final E.B. ingot as compared to the first. Also, notice the yttrium-rich nodular second phase in the arc melted ingot and residue yttrium particles in the final E.B. ingot. It is postulated that the yttrium-rich second phase caused the nonhomogeneity of the metal and resulted in inconsistent chemical analyses.

Figure 17 shows the microstructure of the first E.B. ingot of series II. Notice the relatively smaller second phase precipitates as compared to the first series. Microstructures of the remaining melts of series II are shown consecutively in figures 18 through 21.

Typical microstructures from the top-center and center-edge of the final ingot of series V are shown in figure 22. Notice the cleaner matrix of this high purity vanadium as compared to microstructures from the previous series.

Figure 12

Effect Of Annealing Temperature On The Hardness Of Vanadium

500X
Figure 13. Microstructure of Reduction for Series Ingot. Notice the Dispersed Second Phase and the Larger Black Oxide Inclusions

500X
Figure 14. First Electron Beam Ingot, Series I

500X

Figure 15. Arc Melted with Yttrium Doping, Series I.
Note Yttrium-Rich Nodular Second Phase

250X

Figure 16. Second Electron Beam Ingot, Series I.
Note Some Yttrium Residue

500X

Figure 17. First Electron Beam Ingot, Series II

500X

Figure 18. Second Electron Beam Ingot, Series II

500X

Figure 19. First Yttrium Doped Arc Melted Ingot,
Series II

500X

Figure 20. Fourth Electron Beam Melt Ingot, Series II

500X

Figure 21. Fifth Electron Beam Melt Ingot, Series II

(a) Top-center

(b) Center-edge

Figure 22. Microstructures of Third Electron Beam Ingot, Series V

II TASK II, FABRICATION AND MECHANICAL TESTING

A. Fabrication

1. Experimental Effort

The alloy selected for fabrication was V-15 wt/o Cr-5 wt/o Ti. This was developed at Argonne National Laboratory for potential use in liquid metal cooled fast breeder reactors.

The series V vanadium ingot was machined into turnings under a protective blanket of argon in a fixture shown in figure 23. This arrangement minimized contamination by oxygen and nitrogen. High purity chromium flakes and titanium crystal bar choppings were compacted with the vanadium into 2-inch by 30-inch compacts with a composition of 79.7 percent vanadium, 15.2 percent chromium and 5.1 percent titanium. The increase above nominal values was to allow for volatilization during melting. These were welded together and double arc melted into a 6.5-inch alloy ingot weighing 112.9 pounds, shown in figure 24. Melting data are tabulated in table III. A partial pressure of argon was used for the first melt to reduce the tendency for chromium volatilization. The second melt was performed under a dynamic vacuum to minimize porosity in the ingot.

Table III. Melting Data

V-15Cr-5Ti Arc Melt

	<u>1st Arc Melt</u>	<u>2nd Arc Melt</u>
Heat No.	5-950062	56.5-950063
Input	V-15Cr-5Ti Compacts 117 lb	5-950062 V-15Cr-5Ti 113.3 lb
Output	5" Dia., 117 lb	6.5" Dia., 112.7 lb
Yield	100%	99.5%
Power	112 kw	124-192 kw
Atmosphere	25" Argon	Vacuum Max. 30 micron

Figure 23. Inert Gas Chamber for Machining of Vanadium

Figure 24. Double Arc Melted V-15Cr-5Ti-950063 Ingot, 112 Pounds

The final melting sequence from the initial aluminothermic reduction to the last arc melt of the alloy ingot is presented below:

- 1) Aluminothermic reduction of V_2O_5 with excess aluminum;
- 2) Multiple electron beam melts of the thermite reductions;
- 3) Compaction of vanadium, chromium, and titanium chips into electrode for arc melting;
- 4) Double arc melt.

The hardness and chemical analyses are shown in tables IV and V, respectively.

Table IV. Hardness, BHN
3000 kg load
V-15Cr-5Ti Ingot

<u>Heat No.</u>	<u>56.5-950063</u>
(Top)	
1	212
2	217
3	212
4	217
5	217
6	223
7	212
8	212
9	212
10	212
11	217
12	217
13	212
14	212
15	217
(Bottom)	
Ave	215

Table V. Analytical Data, V-15Cr-5Ti

Ingot No. Sample No.	Second Arc Melt 56.5-950063					Ave
	1	2	3	4	5	
Cr	15.2%	15.5%	15.6%	16.0%	15.9%	15.6%
Ti	5.38%	5.45%	5.66%	5.85%	5.64%	5.6%
O	80		140		220	150
H	4.4				4.3	4.4
N	85	90	103	117	91	97
C	110	130	140	160	150	130
Si	300	300	300	350	300	300
Al	100	50	50	50	100	70
B	<1	<1	<1	<1	<1	<1
Cd	<5	<5	<5	<5	<5	<5
Co	<10	<10	<10	<10	<10	<10
Cu	50	50	<40	<40	50	46
Fe	100	150	150	<100	150	130
Mg	20	20	<20	<20	35	23
Mn	<20	<20	<20	<20	<20	<20
Mo	<20	50	50	100	20	48
Ni	<20	<20	<20	<20	<20	<20
Pb	<20	<20	<20	<20	<20	<20
Sn	<20	<20	<20	<20	<20	<20

- Remarks: (1) Units are in ppm unless otherwise indicated.
- (2) For Ingot No. 950063, Samples No. 1 and 5 are solids from the top and bottom of the ingot respectively, others are fine turnings taken from the circumference of the ingot, from top to bottom.

The hardness averaged 215 BHN. The impurity levels of the alloy were comparable to those of the vanadium ingot. Ultrasonic inspection showed the top 5 inches of the ingot contained shrinkage cavities and associated cracks.

After the upper 5 inches were removed, the ingot was cut in half; one half to be used for extrusion into sheet bar, the other into tube blank. However, two unsuccessful preheating attempts by the extrusion vendor resulted in overheating the sheet-bar billet and melting of the 304 stainless steel canning material. The second half of the ingot was therefore fabricated into sheet by upset forging and rolling at Wah Chang Albany, Albany, Oregon according to the following procedure:

Schedule 1

- 1) The second ingot section was cut into two halves, each 6 inch diameter x 2-1/2 inches thick;
- 2) The two halves were vacuum annealed for 2 hours at 1200°C;
- 3) Jacketed in 1/4-inch thick 304 type stainless steel and TIG welded closed in argon atmosphere;
- 4) Heat treated at 1050°C for 1 hour and upset forged at that temperature to thickness of 1.5 inch. Reductions of 0.5 inch were made with reheats after each pass;
- 5) The forged halves (1.5 in. x 8 in. x 12 in.) were rolled lengthwise at 1050°C until a length of 15 inches was obtained.
- 6) Cross rolled to 3/8-inch thick at 1050°C. The reduction per pass was 10 percent with the plates being reheated after every two passes;

- 7) Annealed in the cladding at 900°C for 1 hour;
- 8) The plates were easily decanned by mechanical means.
Plate B, from the bottom part of the section, was discolored at one edge due to cracks and folding over of the cladding during rolling;
- 9) Conditioned by sawing, grinding and pickling;
- 10) Preheated to 300°C and rolled without reheating to approximately 0.28 inch. Reductions of 2 to 3 percent per pass were used;

Initially the intent was to roll to 3/16-inch thickness; however, rolling was stopped because of a wavy appearance on the surface of the plates transverse to the rolling direction. These were probably caused by the original cross-rolling of the canned billet. Also, both plates showed slight laminations at the ends, and plate B showed slight edge cracks at a corner where the plate was of thinner gage. The lamination might have been avoided if a heavier reduction per pass had been made;
- 11) Conditioned to a more uniform thickness and smooth surface, then vacuum annealed at 1100°C for 2 hours;
- 12) Preheated to 300°C and warm rolled to 0.25-inch thick. Five percent reductions were used with no reheating;
- 13) The sheets were conditioned by hand sanding and vacuum annealed at 800°C for 2 hours;
- 14) Preheated to 300°C and rolled without reheating to 0.040-inch thickness. Five percent reductions were made per pass. Warm rolling was performed instead of cold rolling to prevent possible lamination. The resulting sheet was in the 70 percent cold worked condition.

The total weight of the sheet was 22.0 pounds. Based on a billet weight of 29.0 pounds, the yield was 76 percent. With the tapered ends sheared off, the two sheets measured approximately 0.040 in. x 15 in. x 53 in. as shown in figure 25.

The V-15Cr-5Ti extrusion billet, which was returned from the extrusion vendor, together with scrap and turnings, was compacted and remelted. The shrink pipe section was cut off and the ingot side forged and rolled into sheet according to the procedure presented below, in schedule 2:

Schedule 2

- 1) The ingot section 3.56 inches diameter by 13.5 inches long, weighing 33 pounds, was vacuum annealed 2 hours at 1200°C;
- 2) Jacketed in 1/4-inch thick 304 stainless steel;
- 3) Forged at 1050°C to 1.6 inches by 4.5 inches by 18 inches. Due to uneven reduction between the alloy and the sheath, one end of the sheath was cracked;
- 4) Decanned. One end of the forging was seriously cracked and was cut off;
- 5) Annealed 2 hours at 1200°C and rejacketed in 304 stainless steel;
- 6) Preheated to 1050°C for 90 minutes and rolled to 0.225 by 15 inches;

- 7) Decanned and conditioned until free of surface cracks.
Vacuum annealed at 1200°C for 1 hour;
- 8) Preheated to 300°C and rolled to 0.125 inch thick;
- 9) Vacuum annealed at 800°C for 2 hours and ultrasonically inspected;
- 10) Preheated to 300°C and rolled to size (0.040 inch thick).

Rolling schedules are outlined in table VI. The weight of sheet produced from this heat was 13.6 pounds. The total weight of sheet produced was 35.6 pounds. A 23-pound ingot produced from the extrusion billet and other scrap was melted and sent to Argonne National Laboratory for possible fabrication into tubing or rod.

2. Discussion Of Results

To minimize contamination when compacting electrodes for arc melting, all crushing, cutting or machining should be done under an inert atmosphere. It has been found that machining vanadium in air will increase interstitial contamination in the melt by about 100 ppm.

For the first arc melt, it is recommended that an argon atmosphere be used to prevent chromium and titanium volatilization. However, under an argon atmosphere, porosity tends to form along the side wall and at the top of the ingot. Therefore, a second melt under vacuum is required to reduce porosity.

According to Carson⁽¹⁴⁾, extrusion was the general practice for the breakdown fabrication of high interstitial alloys. However, 1050°C breakdown forging was successful for the high purity V-15Cr-5Ti alloy.

Special attention should be paid to the geometrical uniformity during rolling to reduce tendencies to crack. A 10 percent reduction per pass should be employed for hot rolling while a comparatively high, 5 percent, reduction is required during cold rolling to prevent laminations. Warm rolling at 300°C is preferred for optimum fabricability.

68-52-A

Figure 25. V-15Cr-5Ti Sheets A and B Approximately 0.040 in. x 15 in. x 53 in.

Table VI. Fabrication of V-15Cr-5Ti

Schedule 1

Schedule 2

B. Mechanical Testing

1. Experimental Efforts

A study was performed to determine the recrystallization temperature of the high purity alloy sheet material in the 70 percent cold-worked condition. Based on metallographical results shown in figures 26 and 27, the recrystallization temperature for a 1-hour anneal is 1050°C. Grain size of the recrystallized material is ASTM No. 7 to 8. Hardness readings on the heat treated samples also show that 1050°C produces the minimum hardness, indicative of complete recrystallization, as shown in figure 28. The increase in hardness resulting from the higher temperature heat treatments is due to solution hardening.

Tensile tests results are shown in table VII. The 1050° and 1250°C annealed material demonstrated a serrated-type stress-strain curve and reduced ductility when tested at 600°C. This type of serrated stress-strain curve has also been seen with the V-10Ti and V-20Ti alloys and is indicative of strain hardening. The low elongation for as-received material (approximately 5 percent) is to be expected for a 70 percent cold-worked structure. Tensile test results from the 1050°C annealed material (recrystallized) are plotted in figure 29.

Stress rupture tests were conducted on 1050°C annealed (recrystallized) sheet samples. All testing was performed in static argon, purified by passing the gas through a molecular sieve and titanium sponge at 1600°F. The creep furnace was evacuated to 10^{-5} torr and back filled three times prior to heating up for testing. In addition, the 0.040-inch thick, sheet-type, samples, were further protected from contamination by being loosely wrapped in tantalum foil. Strain was measured by a dial gage attached to the lever arm of the creep machine.

Stress-rupture tests were conducted to provide 10, 100 and 500-hour rupture data at 650° and 800°C. The data are presented in table VIII.

Minimum creep rate and time-to-rupture data as a function of stress are plotted in figures 30 and 31. No anomalies in creep data were observed. Strain versus time curves are shown in figures 32 and 33.

7099

100X

Figure 26. Small Equiaxed Grains After 1050°C/1 hr
Indicating Complete Recrystallization

7111

500X

Figure 27. Same Structure as in Figure 26 Photographed
at 500X

-37-

Figure 28. V-15Cr-5Ti Heat Treat Study

Table VII. Tensile Results on V-15Cr-5Ti Alloy*
(0.040" thick sheet)

Condition and Test Temperature	Ultimate Tensile Strength (psi)	0.2% Yield Strength (psi)	Upper Yield Strength (psi)	Lower Yield Strength (psi)	Reduction of Area (%)	Elongation (%)
As-Received	131,700	129,300	--	--	18.9	4.5
Room Temperature	140,000	133,100	--	--	18.0	5.5
(70% cold worked)	<u>132,000</u>	<u>128,900</u>	--	--	—	—
Average:	134,600	130,400			18.5	5.0
As-Received	111,400	101,600	--	--	6.7	5.0
600°C Test Temp.	<u>115,400</u>	<u>95,600</u>	--	--	<u>7.2</u>	<u>4.0</u>
Average:	113,400	98,600			6.9	4.5
1050°C/1 hour	86,900	--	73,600	73,200	45.6	25.0
Room Temperature	88,400	--	76,900	75,900	46.5	25.0
	<u>87,100</u>	--	<u>77,600</u>	<u>73,500</u>	<u>47.2</u>	<u>28.0</u>
Average:	87,500		76,000	74,200	46.4	26.0
1050°C/1 hour	74,400	--	48,000	46,500	34.6	17.0
500°C Test Temp.	<u>71,700</u>	--	<u>47,100</u>	<u>44,900</u>	<u>34.7</u>	<u>16.0</u>
Average:	73,100		47,600	45,700	34.7	17.0
1050°C/1 hour	74,200	--	46,400	45,200	39.7	15.0
600°C Test Temp.	75,600	--	47,400	44,500	32.8	14.0
	<u>76,300</u>	--	<u>47,700</u>	<u>45,100</u>	<u>36.9</u>	<u>14.0</u>
Average:	75,300		47,200	44,900	36.5	14.0
1050°C/1 hour	74,800	--	50,400	47,400	43.8	20.0
700°C Test Temp.	<u>74,700</u>	--	<u>46,000</u>	<u>45,300</u>	<u>38.0</u>	<u>18.5</u>
Average:	74,800		48,200	46,900	40.9	19.2

*Interstitial Content (ppm) C N O H
 130 90 100 4

Table VII. (Cont.)

Condition and Test Temperature	Ultimate Tensile Strength (psi)	0.2% Yield Strength (psi)	Upper Yield Strength (psi)	Lower Yield Strength (psi)	Reduction of Area (%)	Elongation (%)
1050°C/1 hour	58,800	--	38,500	38,100	43.9	25.0
800°C Test Temp.	59,200	--	40,000	39,600	43.8	23.0
	<u>59,500</u>	--	<u>40,000</u>	<u>40,000</u>	<u>50.1</u>	<u>24.0</u>
Average:	59,200		39,500	39,200	45.9	24.0
1250°C/1 hour	90,200	75,500	--	--	39.9	24.0
Room Temp.	91,900	74,700	--	--	37.8	24.0
Test Temp.	<u>92,700</u>	<u>76,200</u>	--	--	<u>38.8</u>	<u>24.0</u>
Average:	91,600	75,500			38.8	24.0
1250°C/1 hour	75,900	42,400	--	--	33.4	15.0
600°C/1 hour	75,300	40,100	--	--	27.7	15.0
Test Temp.	<u>76,800</u>	<u>42,300</u>	--	--	<u>39.4</u>	<u>16.0</u>
Average:	76,000	41,600			33.5	15.0
1250°C/1 hour	66,000	45,300	--	--	42.7	17.0
800°C Test	64,800	42,700	--	--	43.5	15.0
Temp.	<u>67,000</u>	<u>40,800</u>	--	--	<u>45.5</u>	<u>14.0</u>
Average:	65,900	42,900			43.9	15.0

Figure 29

Mechanical Test Results 1050⁰C/ 1 hr Annealed (Recrystallized) V-15Cr-5Ti

Table VIII. V-15Cr-5Ti Stress-Rupture Data

Temp (°C)	Stress (ksi)	Time to Rupture (hr)	Time to 3rd Stage (hr)	Min Creep Rate (in./in./hr)	Initial ϵ (%)	ϵ_0 Intercept (%)	ϵ at 3rd Stage (%)	Final ϵ (%)	Test No.
650	74.0	3.0	1.3	4.6×10^{-2}	11.4	11.4	4.6	25.0	5
650	72.5	5.6	2.7	2.2×10^{-2}	11.2	11.5	14.0	22.0	6
650	65.0	58.8	33.0	1.0×10^{-3}	5.1	5.6	7.1	18.5	7
650	63.0	45.8	44.0	3.1×10^{-4}	4.4	4.9	6.3	6.8	11
650	64.0	49.3	39.3	4.1×10^{-4}	4.6	5.0	7.0	7.0	12
650	64.0	63.0	63.0	3.9×10^{-4}	4.8	5.6	8.0	8.0	13
800	50.0	1.4	0.5	6.1×10^{-2}	1.2	2.4	4.6	34.0	3
800	40.0	28.1	12.0	4.0×10^{-3}	0.2	0.8	3.2	21.0	4
800	37.5	28.2	8.5	1.4×10^{-2}	0.1	0.4	2.0	18.0	8
800	35.0	118.3	28.0	5.8×10^{-4}	0.1	0.6	2.0	16.5	9
800	35.0	72.0	27.0	1.0×10^{-3}	0.1	0.6	4.0	23.5	10
800	32.0	425	-stopped-	2.0×10^{-5}	0.1	0.4	---	---	14
800	32.0	580	-stopped-	2.6×10^{-5}	0.1	0.2	---	---	16
800	32.0	333	-stopped-	2.4×10^{-5}	0.1	0.2	---	---	17

Figure 30

Minimum Creep Rate vs Stress-V-15Cr-5Ti Alloy Tested in Recrystallized Condition ($1050^{\circ}\text{C}/1$ Hour)

Figure 31
Time To Rupture Results
V-15Cr-5Ti Alloy Sheet
Annealed 1 Hour at 1050°C
All Tests Performed In Argon Atmosphere

2. Discussion Of Results

Argonne National Laboratory, in their evaluation of a lower purity V-15Cr-5Ti alloy (~1200 ppm total interstitial), found that recrystallization occurred after annealing at 900°C for 1 hour with a resulting grain size of ASTM-9 to 10. Heat treating at higher temperatures produced little if any change in grain size up to 1400°C. Above this temperature very rapid grain growth occurred. It is assumed that the abrupt increase in grain size was due to solution of grain boundary precipitates above 1400°C. Argonne National Laboratory chose to anneal their material at 1250°C for 1 hour for optimum stress-rupture properties.

Heat treatment studies on the higher purity materials for this program showed complete recrystallization after 1 hour at 1050°C. The grain size after this treatment was ASTM-7 to 8. Annealing above 1050°C caused initiation of grain growth. This differed from the Argonne National Laboratory results, and is assumed to be related to insufficient grain boundary impurities in the higher purity material to retard grain growth. Heat treating at 1250°C for 1 hour resulted in a grain size of ASTM-4 to 5, as compared to approximately ASTM-8 for the Argonne National Laboratory material.

Based on the above heat treatment studies, it was decided to test the high purity material in the 1050°C and 1250°C annealed conditions. The majority of the tests were performed on recrystallized (1050°C) specimens since this would more nearly duplicate the structure and grain size of the Argonne National Laboratory tests facilitating a comparison of results. A limited number of tests was performed at 1250°C to evaluate the effects of grain size and second phase on strength properties. As shown by tensile test results, the larger grained material was consistently stronger but was slightly less ductile than the 1050°C annealed material.

Stress-rupture tests conducted at 650°C required loadings in excess of the 0.2 percent yield strength to cause failure within 100 hours. Table VIII shows that considerable strain occurred on initial loading of these specimens, 5 to 11 percent. The material at this temperature is obviously yield strength dependent. In all cases, failure was transgranular with failure ductility in the range of 7 to 25 percent, the lower ductilities being caused by strain hardening mechanisms.

The 800°C stress-rupture tests produced a 100-hour rupture strength of approximately 35,000 psi, 87.5 percent of the yield strength. Again, failure was transgranular. Two of the test specimens (CR-4 and CR-9) showed evidence of contamination on the surface after test. Micro hardness measurements showed a case less than 5 mils in depth on both samples. The presence of the surface contamination did not appear to affect the creep or rupture data shown in figures 30 and 31.

The 500-hour tests, run at 800°C, are not plotted on the time-to-rupture curves. Contamination during the 800°C tests required that the tests be terminated prior to rupture. Figure 34 shows the creep characteristics of three tests tested at 800°C. Interstitial contamination caused strengthening of the alloy and a reduction in the creep rate. Test No. 17 was run using a hot tantalum gettering coil; however, contamination still occurred.

Other laboratories testing vanadium alloys have also had contamination problems during long time stress-rupture testing. However, our problem is further aggravated by the high purity of the starting material (~400 ppm total interstitial content). Results of interstitial analysis show that contamination to the extent of 1100 ppm is sufficient to alter the creep rate. Most investigators start with material containing 1200 to 1500 ppm interstitials. Comparing the linear secondary creep rates of the contaminated test to those of shorter times in figure 30, indicates the initial minimum creep rates are representative of uncontaminated material.

One stress-rupture test on the lower purity Argonne National Laboratory (ANL) material is presently available for comparison. The ANL test was conducted at 700°C and 42,000 psi. The test did not run to failure but was terminated after 300 hours. The minimum creep rate was approximately 1.6×10^{-7} in./in./hour, which falls between the 650°C and 800°C results shown in figure 35. There are insufficient data to comment on the relative strength or creep properties of the high and low interstitial materials at this time. It does appear, however, that the reduction of interstitial content does not significantly reduce the alloy's strength.

Figure 34

% Strain Creep Curves For V-15Cr-5Ti Alloy Tested In Recrystallized Condition (1050°C/1Hour)

The Data Plotted Are For Tests Number 14, 16 and 17
Showing Length Of Linear Creep Rate. Parameters
Of Tests Were 800°C at 32,000 psi In Purified Argon
Atmosphere.

Figure 35
Comparison Of ANL and CENPD Creep Data

III TASK III, WELDABILITY

A. Experimental Effort

The last task of this program was an evaluation of TIG and electron beam welding characteristics of the high purity V-15 wt/o Cr-5 wt/o Ti alloy sheet. The 0.040-inch thick sheet was welded by both methods using the following schedules:

<u>Electron Beam</u>	<u>Tungsten Inert Gas</u>
80 kilovolts	1/8" dia. thoriated tungsten electrode
2.5 milliamps	90 amperes dc
30" per minute travel	10" per minute travel

All sheets were welded from one side using a "bead-on-plate" technique necessitating no filler metal. The weld beads were transverse to the rolling direction of the sheet. All material was welded in the recrystallized condition (1050°C/1 hr). After welding, the sheets were bowed due to weld metal shrinkage. Subsequent to welding, another 1050°C/1 hour heat treatment was performed to facilitate straightening and machining. Figures 36 and 37 show the bead-on-plate welds.

Tensile results of the TIG and electron beam welds are shown in table IX. The data for the TIG welded samples show low elongation and reduction in area values indicative of brittle welds.

7627

8X

Figure 36. TIG Weld Bead on V-15Cr-5Ti Sheet

7628

8X

Figure 37. Electron Beam Weld Bead on V-15Cr-5Ti Sheet

Table IX. Tensile Tests on Welded Sheet
V-15Cr-5Ti
Preweld and Postweld Anneals of 1050°C/1 hr

Weld	Test Temp	UTS ksi	Y.S. ksi	R.A. %	Elong %	Comments	No.
TIG	R.T.	80.40	73.6	~ 1%	2.5	Failed in weld	TIG-2
TIG	R.T.	68.5	62.3	16.0	3.0	Failed in weld	TIG-3
EB	R.T.	90.1	74.1	47.0	18.0	Failed in Parent metal	EB-2
EB	R.T.	91.8	75.4	45.6	21.5	Failed in Parent metal	EB-3
none	R.T.	87.5	~ 75	46.4	26.0	(Control)	-----
TIG	800°C	23.5	17.0	-----	~ 3	Failed in weld	TIG-1
EB	800°C	63.0	41.0	45.6	21.5	Failed in Parent metal	EB-1
EB	800°C	60.0	38.6	15.0	17.0	Failed in weld	EB-4
none	800°C	59.2	39.3	45.9	24.0	(Control)	-----

Two 650°C stress rupture tests were performed on the electron beam welded material. Results are tabulated in table X. The creep rates are compared to parent metal tests (figure 30).

Table X. Electron Beam Welded V-15Cr-5Ti
650°C Stress - Rupture Data

Stress ksi	Time to Rupture Hours	Min. Creep Rate (in./in./ hour)	Initial ϵ (%)	ϵ_0 Intercept (%)	Final ϵ (%)	Test No.
63.0	146 --stopped--	6.00×10^{-5}	3.1	3.2	3.5	18
68.0	98 --stopped--	2.86×10^{-4}	5.8	5.9	6.5	19

Both tests showed contamination after the sample was removed from the furnace; however, the initial creep rate was unaffected.

B. Discussion Of Results

All TIG welds were extremely brittle as noted in table IX. Similar results were observed by Westinghouse Atomic Power Laboratory in their preliminary survey of vanadium alloys (ref: WCAP-3487-16). Cross sections of as-welded and welded-and-annealed samples are shown in figures 38 and 39. The figures show that the 1050°C anneal caused precipitate agglomeration. The agglomeration in the TIG welds is analogous to "coring" in ingots. The second phase is concentrated due to differences in solubility on cooling. In an effort to more uniformly distribute the agglomerated second phase, fractured samples of annealed welds were heat treated at 1500°C for 4 hours, then cooled at 700°C per hour. Figure 40 shows the results. As seen, the precipitate went into solution and precipitated in a more uniform distribution. Of course, this does not offer a solution to the problem since the grains are grossly oversize as a result of the 1500°C heat treatment. It would appear from these preliminary tests that welding procedures which do not lead to agglomeration should be evaluated. One method appears to be electron beam welding. Due to the rapid cooling after welding, agglomeration cannot occur, as can be seen in figure 41. It should be noted, however, that one centrally located grain shows agglomeration. This grain would be one of the last to solidify. Ductility of the electron beam welds is good, as shown in table IX. Figure 42, a cross section of an electron beam welded specimen tensile test at 800°C, shows a ductile, transgranular fracture even though the failure occurred in the weld.

Stress-rupture tests also demonstrate that electron beam welded samples behave similar to the parent metal samples.

8621

35X

Figure 38. As-Welded TIG Weld. Preweld Heat Treatment of $1050^{\circ}\text{C}/1\text{ hr}$

7648, 7649

Figure 39. Fractured TIG Weld with a Postweld Heat Treatment of 1050°C/1 hr.
Note Severe Agglomeration of Precipitate

7822

-62-

35X

Figure 40. Fractured TIG Weld After Heat Treating at 1050°C/1 hr and 1500°C/4 hr.
The Second Phase is Redistributed

100X

Figure 41. Cross Section of Electron Beam-Welded V-15Cr-5Ti Sheet. Note Lack of Agglomerated Precipitate. The Weld had Preweld and Postweld Heat Treatments of $1050^{\circ}\text{C}/1\text{ hr}$

69-133

50X

Figure 42. Cross Section of V-15Cr-5Ti Fractured Tensile Sample Tested at 800°C . Sample Failed in Weld with a Ductile Time Fracture

CONCLUSIONS

It has been shown that high purity vanadium can be commercially produced by aluminothermic reduction of V_2O_5 followed by subsequent electron beam melting. Contamination by nitrogen and silicon should be avoided during the reduction process.

Alloying by compaction of alloying elements followed by double arc melting, created no problems. Machining of vanadium chips, however, should be done under an inert gas cover to prevent contamination. Two arc melts should be performed; the first in a partial pressure of argon to prevent volatilization of alloying elements and the second under a dynamic vacuum to reduce porosity.

Fabrication of sheet showed some tendency of the V-15Cr-5Ti alloy to laminate and produce edge cracks. This can be avoided if relatively large reductions in thickness are employed.

Mechanical tests show the high purity V-15Cr-5Ti alloy to be as strong as lower purity vanadium alloys of V-Ti and VANSTAR (Westinghouse) compositions. Creep strength for the V-15Cr-5Ti alloy are also comparable to these alloys; however, the high purity V-15Cr-5Ti alloy is susceptible to interstitial contamination during testing.

Welding studies showed the V-15Cr-5Ti alloy was susceptible to coring during TIG welding which resulted in brittle welds. This can be prevented, however, by the use of electron beam welding which reduces coring due to the rapid cooling rate inherent with this type of process. Tensile and stress-rupture tests of electron beam welded samples showed mechanical properties similar to the parent material.

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APPENDIX A
COMPLETE CHEMICAL ANALYSIS
OF ALL INGOT MELTS

TABLE 1
ANALYTICAL DATA OF REDUCTIONS, SERIES I

Lot Number	2-18-67	2-13-67	3-4-67	2-24-67
Melt. Sequence	1	2	3	4
C	0.032	<0.004	0.009	0.004
N	0.008	0.004	0.005	0.021
O	0.950	0.330	1.570	1.060
Al	1.900	2.740	2.010	2.820
Fe	0.710	0.640	-	0.870
Cu	0.350	0.250	-	0.180
Si	0.330	0.330	0.330	0.310
Mo	0.030	0.020	0.025	0.030
Hardness after button melting				
(10 kg DPH)	258	250	-	275
Weight (Lbs)	38	60.1	57.2	62

Table 2
Melting Data, Series I

	<u>1st E. B. Melt</u>	<u>Yttrium Doped Arc Melt</u>	<u>2nd E. B. Melt</u>
Heat No.	820006-S4A	5.5-950018	940009-S2B
Power (KW)	180	202.0	90.0
Vacuum (Micron)	0.2	5 - 10	0.02
<u>Material Balance</u>			
Input (lb.)	224.3	80.5	131.0
Output (lb.)	157.2	79.4	109.0
Yield (%)	70.0	98.5	83.4

Table 3

Chemical Analyses, First E. B. Ingot, Series I

Heat No. 820006

(Weight Percent)

[illegible]

TABLE 4
CHEMICAL ANALYSIS, ARC-MELTING INGOT WITH YTTRIUM DOPING

Series I

Heat No. 5.5-950018

Sample No.	1	2	3	4	5
C	0.014	0.014	0.012	0.012	0.011
N	0.027	0.022	0.019	0.014	0.013
O	0.026		0.032		0.029
Al	0.226	0.269	0.232	0.243	0.276
Si	0.390	0.380	0.380	0.350	0.380
Fe	0.195	0.178	0.173	0.157	0.155
Y	1.590	0.910	0.890	1.240	0.650
Cu	0.004	0.008	0.010	0.010	0.010
Cr	0.007	0.007	0.005	0.002	0.005
Mo	0.035	0.030	0.035	0.035	0.065
Sn	<0.010	<0.010	<0.010	<0.010	<0.010
B	0.0007	0.0005	0.0007	0.0005	0.0010
Cd	All less than 0.0005				
Co	All less than 0.0010				
Mg	0.004	0.004	0.004	0.004	0.0075
Mn	All less than 0.002				
Ni	0.010	0.010	0.010	0.010	0.010
Pb	All less than 0.002				
Ti	All less than 0.005				

Table 5
Chemical Analysis, 2nd E. B. Ingot

Series I

Heat No. 940009-S2B

Upper Portion						Lower Portion				
1st E. B. -Arc- 2nd E. B.						1st E. B. - 2nd E. B.				
Sample No.	1	2	3	4	5	1	2	3	4	5
C	0.016	0.014	0.013	0.013	0.015	0.010	0.008	0.009	0.011	0.016
N	0.028	0.032	0.023	0.018	0.016	0.007	0.006	0.008	0.010	0.011
O	0.036		0.030	0.028		0.027			0.019	
Al	0.042	0.202	0.074	0.135	0.074	0.054	0.039	0.024	0.022	0.039
Fe	0.101	0.108	0.088	0.122	0.091	0.101	0.076	0.056	0.072	0.142
Y	0.006	0.025	0.016	0.024	0.007					
B	0.0007	0.0006	0.0005	0.0006	0.0005	0.0005	0.0005	0.0005	0.0006	0.0005
Cd				All less than 0.0005						
Co				All less than 0.001						
Cr				All less than 0.002						
Cu				All less than 0.0004						
Mg				All less than 0.002						
Mn				All less than 0.002						
Mo	0.035	0.035	0.035	0.040	0.035	0.040	0.040	0.040	0.050	0.035
Si	0.440	0.440	0.420	0.380	0.410	0.420	0.430	0.440	0.570	0.320
Ni				All less than 0.002						
Pb				All less than 0.002						
Sn	0.020	0.030	0.250	0.250	0.250	0.030	0.035	0.030	0.035	0.050
Ti				All less than 0.005						

Table 6
HARDNESS Data, Series I

Section	Reduction	820006 1st E B Ingot	5.5-950018 Arc - Melt Ingot	940009 2nd E B Ingot
	(Button Melted)	BHN (3000 kg)	BHN (500 kg)	BHN (3000 kg) DPH (10 kg)
1		201	82.6	183
2	275	197	82.6	201
3		179	76.8	192
4		192	79.6	156
5		197	89.0	170
6		183	79.6	197
Average		(190)	(82)	(183)
7		197		183
8	250	187		207
9		179		163
10	258	197		197
Average		(190)		(180)

Table 7
ANALYSES OF THE ALUMINUM Granules
Weight Percent

N	-	0.0005
B	-	<0.0005
Cd	-	<0.0005
Co	-	<0.0010
Cr	-	<0.0020
Cu	-	<0.0040
Fe	-	<0.0100
Mg	-	<0.0020
Mn	-	<0.0020
Mo	-	<0.0020
Ni	-	0.0050
Pb	-	<0.0020
Si	-	<0.0100
Sn	-	<0.0020
Ti	-	<0.0050
V	-	<0.0020

TABLE 8
Chemical Analyses, Reduction, Weight Percent
Series II

Lot No.	7-19-67	7-23-67	7-22-67	7-28-67	7-26-67	Ave.
C	0.006	0.145	<0.004	<0.040	0.075	.054
O	0.20	0.900	0.20	0.21	1.500	.602
N	0.0072	0.0118	0.0100	0.0085	0.0026	.008
Al	0.850	0.540	0.500	0.500	1.370	.752
Si	0.045	0.033	0.0330	0.047	0.056	.043
Fe	>0.100	>0.100	>0.100	>0.100	>0.100	>.100
B	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<.0001
Cd	<0.0005	<0.0005	<0.0005	0.0005	<0.0005	<.0005
Co	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<.0020
Cr	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<.0020
Cu	>0.100	>0.100	>0.100	>0.100	>0.100	>.100
Mg	0.003	0.0035	0.0035	0.002	0.002	.003
Mn	<0.002	<0.002	<0.002	<0.002	<0.0020	<.002
Mo	<0.002	<0.002	<0.002	<0.002	<0.002	<.002
Ni	<0.002	<0.002	<0.002	<0.002	<0.002	<.002
Pb	<0.002	<0.002	<0.002	<0.002	<0.002	<.002
Sn	0.015	0.025	0.015	0.020	0.020	.020
Ti	<0.005	<0.005	<0.005	<0.005	<0.005	<.005

TABLE 9
Hardness Data, BHN
Series II, Ingots

	1st E. B.*	2nd E. B.*	3rd E. B.*	1st Dope**	2nd Dope**	4th E. B.**	5th E. B.**
Heat No. (Top)	940010	940012	940013	950044	950046	950015	950018
1	157	81	262	126	53.4	114.0	89.0
2	102	105	277	121	56.8	89.0	100.0
3	223	123	293	140	55.1	92.6	100.0
4	103	121	229	143	53.4	100.0	92.6
5	81	321	179	159	51.8	74.1	100.0
6	363	352	176	143	57.5	80.8	
7	363	131	229	143	56.8	111.0	
8	477	217	207	143	60.5		
9	444	321	118	163	69.1		
10	187	363	118	179	74.1		
11	116		130				
12	143		101				
13			105				
14			116				
(Bottom)							
Average	229.5	213.5	181.5	146	58.5	94.5	96.3

* 3000 kg Load

** 500 kg Load

TABLE 10
Chemical Analyses, Weight Percent
First Electron Beam Ingot
Heat No. 940010-V-XIA
Series II

Sample No.	1	2	3	4	4.5	5
C	0.006	<0.004	<0.004	0.004	0.011	0.088
O	0.60			1.420		0.300
N	0.0060	0.0070	0.010	0.0125	0.0150	0.0170
Al	0.1020	0.1460	0.1860	0.0515	0.0150	0.0710
Si	0.0710	0.0430	0.0090	0.0140	0.0140	0.0430
Fe	0.0078	0.0066	0.0078	0.0083	0.0316	0.0079
B	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
Cd	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005
Co	<0.0010	<0.0010	<0.0010	<0.0010	<0.0010	<0.0010
Cr	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Cu	<0.0040	0.0040	0.0040	0.020	0.0075	<0.0040
Mg	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Mn	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Mo	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Ni	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Pb	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Sn	0.0050	0.0050	0.0050	0.0050	0.0075	0.0050
Ti	0.0050	0.0075	0.0075	0.0100	0.0050	0.0050

TABLE 11
Chemical Analyses, Weight Percent
Second Electron Beam Ingot
Heat No. 940012-V-XIB
Series II

Sample No.	1	2	3	4	5
C	<0.0040	<0.0040	<0.0040	<0.0040	<0.0050
O	0.0860				0.570
N	0.0065	0.0120	0.0125	0.0170	0.0240
Al	0.0310	0.0430	0.0480	0.0360	0.0085
Si	0.070	0.040	0.0070	0.0240	0.0120
Fe	0.0225	0.0260	0.0200	0.0248	0.0210
B	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
Cd	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005
Co	<0.0010	<0.0010	<0.0010	<0.0010	<0.0010
Cr	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Cu	0.0050	<0.0040	<0.0040	<0.0040	<0.0040
Mg	0.0020	0.0020	0.0020	0.0020	<0.0020
Mn	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Mo	<0.0020	<0.0020	<0.0020	<0.0020	<0.0100
Ni	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Pb	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Sn	0.0050	0.0075	0.0050	0.0050	0.0050
Ti	0.0075	0.0100	0.0200	0.0050	0.0050

TABLE 12
Chemical Analyses, Weight Percent
Third Electron Beam Ingot
Heat No. 940013-V-S2C
Series II

	1	2	3	4	5
C	0.0060	<0.0040	<0.0040	0.0040	0.0080
O	0.470	0.540	0.320	0.100	0.054
N	0.0205	0.0175	0.0165	0.0165	0.0145
Al	<0.00025	<0.00025	<0.00025	<0.00025	<0.00025
Si	0.0047	0.023	0.012	<0.049	0.051
Fe	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100
B	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
Cd	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005
Co	<0.0010	<0.0010	<0.0010	<0.0010	<0.0010
Cr	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Cu	<0.0040	<0.0040	<0.0040	<0.0040	<0.0040
Mn	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Mo	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Ni	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Ti	0.0005	0.0005	0.0005	0.0005	0.0005
H					<0.0007

TABLE 13
Chemical Analyses, Weight Percent
First Doped Arc Ingot
Heat No. 6-950044-VY
Series II

	1	2	3	4	5
C	0.041	0.011	0.009	0.004	0.004
O	0.450		0.190		0.110
N	0.0215	0.0222	0.0198	0.0198	0.0219
Al	<0.0025	<0.0025	<0.0025	<0.0025	<0.0025
Si	--	--	--	--	--
Fe	<0.010	<0.020	<0.010	<0.010	<0.010
B	0.0001	0.0001	0.0001	0.0001	0.0001
Cd	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005
Co	<0.0010	<0.0010	<0.0010	<0.0010	<0.0010
Cr	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Cu	0.0050	0.0040	<0.0040	<0.0040	0.0050
Mg	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Mn	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Mo	<0.0020	<0.0020	<0.0020	0.0030	0.0050
Ni	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Pb	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Sn	<0.0020	0.0025	<0.0020	<0.0020	<0.0020
Ti	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
Y	0.084	1.080	1.170	0.360	0.620

TABLE 14
Chemical Analyses, Weight Percent
Second Doped Arc Ingot
Heat No. 66-950046-VY

	Series II				
	1	2	3	4	5
C	0.0050	0.0040	0.0050	0.0040	0.0040
O	0.570		0.290		0.220
N	0.0205	0.0180	0.0200	0.0205	0.0220
Al	<0.0025	<0.0025	<0.0025	<0.0025	<0.0025
Si	0.0250	0.0230	0.0200	0.0200	0.0130
Fe	<0.0100	0.0100	<0.0100	<0.0100	<0.0100
B	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
Cd	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005
Co	<0.0010	<0.0010	<0.0010	<0.0010	<0.0010
Cu	<0.0040	0.0040	<0.0040	<0.0040	<0.0040
Mg	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Mn	<0.0040	<0.0040	<0.0040	<0.0040	<0.0040
Mo	0.0050	0.0050	0.0050	0.0020	0.0020
Ni	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Pb	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Sn	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020
Ti	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
Y	0.0409	1.230	1.400	1.950	1.950
W	.0024		.0013		.0012
Cr	< .002	< .002	< .002	< .002	< .002

TABLE 15
Chemical Analyses, Weight Percent
Fourth Electron Beam Ingot
Heat No. 940015-V-XIA
Series II

Sample No.	1	2	3
C	0.0240	0.0190	0.0200
O	0.290		0.259
N	0.0245	0.0275	0.0225
Al	<0.0025	<0.0025	<0.0025
Si	0.0500	0.0500	0.0500
Fe	<0.010	<0.010	<0.010
B	<0.0001	<0.0001	<0.0001
Cd	<0.0005	<0.0005	<0.0005
Co	<0.0010	<0.0010	<0.0010
Cr	<0.0020	<0.0020	<0.0020
Cu	0.040	<0.004	<0.004
Mg	<0.002	<0.002	<0.002
Mn	<0.002	<0.002	<0.002
Mo	<0.002	<0.002	<0.002
Ni	<0.002	<0.002	<0.002
Pb	<0.002	<0.002	<0.002
Sn	<0.002	<0.002	<0.002
Ti	<0.005	<0.005	<0.005
Y	0.097	0.3420	.0494

TABLE 16
Chemical Analyses
Fifth Electron Beam Ingot
Heat No. 940018-V-S3B
Series II

Sample No.	1	2	3
C	0.0120	0.0130	0.0120
O	0.0300		0.0520
N	0.0270	0.0320	0.0255
Al	<0.0025	<0.0025	
Si	0.0390	0.0440	
Fe	<0.010	<0.010	
B	<0.0001	<0.0001	
Cd	<0.0005	<0.0005	
Co	<0.001	<0.001	
Cr	<0.002	<0.002	
Cu	<0.004	<0.0075	
Mg	<0.002	<0.002	
Mn	<0.002	<0.002	
Mo	<0.010	<0.010	
Ni	<0.002	<0.002	
Pb	<0.002	<0.002	
Sn	<0.002	0.002	
Ti	<0.002	<0.002	
Y	0.0380	0.0340	0.3440
H	0.00049		0.00049

TABLE 17
Melting Data
Series II

Heat No.	Power KW/KVA	Vacuum Micron	Input lbs.	Output lbs.	Yield
940010-V-XIA					
1st E. B.	150	.1	213.7	126.7	59.5%
940012-V-XIB					
2nd E. B.	150	.1	103.0	82.0	79.5%
940013-V-S2C					
3rd E. B.	210	.05	77.0	62.9	81.5%
6-950044-VY					
1st Dope	180	20-50	59.3	62.0	101.4%
66-950046-VY					
2nd Dope	240	1.5-2.5	59.0	58.2	98.5%
940015-V-XIA					
4th E. B.	150	.1	57.0	35.2	61.5%
940018-V-S3B					
5th E. B.	210	.05	34.6	30.4	87.9%

TABLE 18
Chemical Analyses, Weight Percent
Series III, Vanadium Reductions

	8-7-67	8-8-67	8-9-67	8-10-67	8-11-67	8-24-67	8-25-67	Ave.
O	.13	.14	.39	1.01	1.44	.1400	.4360	0.525
N	.0070	.0045	.0115	.0144	.0230	.0020	.0040	.0096
C	.0080	.0050	.0140	.0090	.0120	.0150	.0100	.0104
H	.0050			.0047		.0034	.0031	.0040
Si	.0600	.04	.04	.03	.03	.027	.026	.0361
Al	1.40	1.48	1.56	1.89	1.31	3.17	2.69	1.93
Fe	.0370	.0440	.0410	.0390	.0360	.025	.043	.0378
B	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Cd	<.0005	<.0005	<.0005	<.0005	<.0005	<.0005	<.0005	<.0005
Co	<.0010	<.0010	<.0010	<.0010	<.0010	<.0010	<.0010	<.0010
Cr	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020
Cu	.0500	.0500	.0040	>.10	.10	.0100	<.0040	.0454
Mg	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020
Mn	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020
Mo	<.0020	.0025	.0020	.0020	.0020	<.0020	<.0020	.0020
Ni	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020
Pb	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020
Sn	.0100	.0075	.0075	.0100	.0200	<.0020	<.0020	.0064
Ti	.0100	.0075	.0050	<.0050	.0100	<.0050	<.0050	.0068

Table 19
Hardness Reading, BHN, 300 kg Load
Series III

First E. B.	2nd E. B.	Arc-Doped	3rd E. B.
<u>Heat No. 940017</u>	<u>Heat No. 940020</u>	<u>Heat No. 6-95048</u>	<u>Heat No. 940021</u>
375	96	69.1	116
241	143	66.8	137
277	143	74.1	143
311	170	69.1	121
156	293	76.8	143
179	229	77.9	121
174	285	75.2	
116	321	71.5	<u>Heat No. 940022</u>
96	96	72.5	137
<u>Heat No. 940014</u>	105	85.8	143
163	121		116
207	156		126
167	137		131
179	179		
192	183		
152	183		
152	167		
174	174		
96			
107			
Average 186	176	74	131

TABLE 20
Chemical Analyses, Weight Percent
Series III, First E. B. Ingot
(Combination of 940014 and 940017)

Sample No.	Ingot No. 940014-V-XIA			940017-V-XIA		Ave.
	1	2	3	1	2	
O	.1600		.0320	.65	.10	.2355
N	.0225	.0095	.0125	.0260	.0125	.0166
C	.0550	.0500	.0500	.0460	.0470	.0496
H	.0011		.0008	.00026	.0003	.00011
Si	.0690	.0610	.0350	.0440	.0440	.0506
Al	.20	.40	.15	.3600	.1010	.2422
Fe	.0450	.0590	.0410	.0470	.0400	.0464
B	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Cd	<.0005	<.0005	<.0005	<.0005	<.0005	<.0005
Co	<.0010	<.0010	<.0010	<.0010	<.0010	<.0010
Cr	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020
Cu	.0150	<.0040	.0040	.0040	.0040	.0062
Mg	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020
Mn	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020
Mo	.0075	.0025	.0075	.0075	<.0020	.0054
Ni	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020
Pb	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020
Sn	<.0010	<.0010	<.0010	<.0020	<.0020	<.0014
Ti	<.0050	<.0050	<.0050	<.0050	<.0050	<.0050

TABLE 21
Chemical Analyses, Weight Percent
Series III, Second E. B. Ingot
Heat No. 940020-V-S3B

Sample No.	1	2	3	4	5
O	.0640		.0360		.0440
N	.0135	.0305	.0350	.0120	.0250
C	.0060	.0060	.0050	.0050	.0100
H	.0009		.0009		.0006
Si	.0420	.0350	.0460	.0610	.0870
Al	.0330	.0100	.0105	.0070	.0115
Fe	<.0100	<.0100	<.0100	<.0100	<.0100
B	<.0001	<.0001	<.0001	<.0001	<.0001
Cd	<.0005	<.0005	<.0005	<.0005	<.0005
Co	<.0010	<.0010	<.0010	<.0010	<.0010
Cu	<.0040	<.0040	<.0040	<.0040	<.0040
Mg	<.0020	<.0020	.0020	.0020	.0020
Mn	<.0020	<.0020	<.0020	<.0020	<.0020
Mo	.0300	.0100	.0075	.0250	.0250
Ni	<.0020	<.0020	<.0020	<.0020	<.0020
Pb	.0025	<.0020	<.0020	<.0020	<.0020
Si	.0420	.0350	.0460	.0610	.0870
Sn	<.0020	<.0020	<.0020	<.0020	<.0020
Ti	<.0050	<.0050	<.0050	<.0050	<.0050

Table 22
Chemical Analyses, Weight Percent
Arc-Doped Ingot, Ht. No. 6-950048-VY
Series III

Sample	1	2	3	4	5	Average
O	.061		.106		.073	.080
N	.0175	.0185	.0185	.0160	.0175	.0176
C	.0060	.0050	.0060	.0050	.0050	.0054
Si	.0440	.0570	.0700	.0560	.0360	.0526
Al	.0350	.0320	.0310	.0310	.0300	.0318
Fe	.0360	.0470	.0550	.0510	.0415	.0461
B	<.0001	<.0001	<.0001	<.0001	<.0001	<.0001
Cd	<.0005	<.0005	<.0005	<.0005	<.0005	<.0005
Co	<.0010	<.0010	<.0010	<.0010	<.0010	<.0010
Cr	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020
Cu	<.0040	.0040	<.0040	<.0040	<.0040	<.0040
Mg	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020
Mn	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020
Mo	.0250	.0250	.0250	.0250	.0200	.0240
Ni	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020
Pb	<.0020	<.0020	<.0020	<.0020	<.0020	<.0020
Sn	.0035	.0035	<.0020	<.0020	<.0020	<.0026
Ti	<.0050	<.0050	<.0050	<.0050	<.0050	<.0050

Third Electron Beam Melt, Series III

[illegible]

TABLE 24
Melting Data
Series III Ingots

Heat No.	Power KW	Vacuum Micron	Melting Rate in/hr.	Input Lbs.	Output Lbs.	Yield
940014-XIA 1st E. B.	150	<.1	3	342.0	139	68%
940017-XIA 1st E. B.	150	<.1	3		94	
940020-S3B 2nd E. B.	150	<.05		231.9	204.1	88%
6-950048-VY Arc-doping	240	3-12		149.2	150.3	>100%
940021-S2A } 940022-S2A } 3rd E. B. }	150	.05	3	147	117	79.5%

Table 25
Reduction Data
Series IV

INPUT				OUTPUT		
V_2O_5	lbs. Al	lbs. Al, % Theoretical		Derby lbs.	Yield of V % Theo.	Machined Weight
11-13-67	100	60.5	122	63	112	49.6
11-17-67	100	58.5	118	60	107	47.8
11-27-67	100	58.8	118	57	102	51.9
Total				180		149.3

Note: Theoretical ratio $V_2O_5:Al:V=100:49.5:56$

TABLL 23
CHEMICAL ANALYSES
ppm

Series IV Reductions

No.	11-13-67			11-17-67			11-27-67			Average 90%
	1	2	3	1	2	3	1	2	3	
O	1890		6900	2600		2600	4400		4730	3350
C	210		230	130		100	130		210	170
N	35		56	83		48	115		135	79
Si	<100	180	350	90	130	90	510	280	410	240
Al	10.7%		16.6%	15.4%		16.0%	16.0%		17.4%	15.5%
B	<1		5	<1		<1	<1		<1	<1
Cd	<5		<5	<5		<5	<5		<5	<5
Co	<10		<10	<10		<10	<10		<10	<10
Cr	<20		<20	<20		<20	<20		<20	<20
Cu	200		200	350		250	250		250	250
Fe	1800		120	95		110	650		600	560
Mg	<20		<20	<20		<20	<20		<20	<20
Mn	20		<20	<20		<20	<20		<20	<20
Mo	<20		<20	<20		<20	<20		100	<30
Ni	<20		<20	<20		<20	<20		<20	<20
Pd	<20		<20	<20		<20	<20		<20	<20
Sn	<20		<20	<20		<20	<20		<20	<20
Ti	<50		<50	<50		<50	<50		<50	<50

TABLE 27
HARDNESS DATA, BHN, 3000 KG LOAD
SERIES IV

<u>FIRST E. B.</u>	<u>SECOND E. B.</u>
Ingot No. 940026-V-S4A	Ingot No. 940027-V-S4B
86	99
87	107
96	96
90	96
109	96
116	187
116	179
116	156
Average 102	Average 127
Ingot No. 940023-V-S4A	
217	
170	
179	
181	
Average 183	
Weighed Average 137	

TABLE 28
CHEMICAL DATA WEIGHT PERCENT
First E.B. Melt
Series IV

Heat No.	940023-V-S4A			940026-V-S4A				Weighed
Sample No.	1	2	Ave.	1	2	3	Ave.	Average
N	.018	.017	.018	.011	.01	.009	.010	.013
O	.042	.029	.035	.044		.020	.032	.033
C	.037	.029	.033	.027	.023	.031	.027	.029
H	.0003	.0009	.0006	.0004		.0005	.0005	.0005
Si	.038	.027	.033	.039	.031	.030	.033	.033
Al	.56	1.5	1.03	.90	.56	.62	.69	.83
B	.0004	.0004	.0004	.0008	.0002	.0008	.0006	.0005
Cd	<.0005	<.0005	<.0005	<.0005	<.0005	<.0005	<.0005	<.0005
Co	<.001	<.001	<.001	<.001	<.001	<.001	<.001	<.001
Cr	<.002	<.002	<.002	<.002	<.002	<.002	<.002	<.002
Cu	.005	.005	.005	<.004	<.004	<.004	<.004	.004
Fe	.026	.023	.025	.023	.013	.0155	.017	.020
Mg	<.002	<.002	<.002	<.002	<.002	<.002	<.002	<.002
Mn	<.002	<.002	<.002	<.002	<.002	<.002	<.002	<.002
Mo	<.03	.035	.033	.005	<.002	.002	.003	.016
Ni	<.002	<.02	<.02	<.002	<.002	<.002	<.002	<.007
Pb	<.002	<.002	<.002	<.002	<.002	<.002	<.002	<.002
Sn	<.002	<.002	<.002	<.002	.25	<.002	.085	.049
Ti	.09	>1.0	.56	.005	.01	.03	.015	.25

TABLE 29
 CHEMICAL DATA, WEIGHT PERCENT
 SERIES IV
 SECOND E.B., HEAT NO. 940027-V-S4B

<u>Sample No.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>Ave.</u>
N	.009	.010	.021	.022	.016
O	.016	.017	.013	.032	.020
C	.020	.014	.013	.013	.015
H	.0003			.0008	.0005
Si	.030	.039	.037	.052	.040
Al	.031	.021	.042	.080	.044
B	.0002	.0002	.0001	.0002	.0002
Cd	<.0005	<.0005	<.0005	<.0005	<.0005
Co	<.001	<.001	<.001	<.001	<.001
Cr	<.002	<.002	<.002	<.002	<.002
Cu	<.004	<.004	<.004	<.004	<.004
Fe	.0053	.005	.0075	.0175	.009
Mg	<.002	<.002	<.002	<.002	<.002
Mn	<.002	<.002	<.002	<.002	<.002
Mo	.02	.015	.03	.05	.03
Ni	<.002	<.002	<.002	<.002	<.002
Pb	<.002	<.002	<.002	<.002	<.002
Sn	<.002	<.002	<.002	<.002	<.002
Ti	.015	.055	.15	.35	.142
W	.001	.002	.008	.008	.005

TABLE 30
MELTING DATA
SERIES IV

	FIRST E.B. MELT		SECOND E.B. MELT
Heat Number	<u>940023</u>	<u>940026</u>	<u>940027</u>
Input	Reduction No. (11-13-67) & (11-27-67) (11-17-67)		Ingots
	67.2 lbs.	97.4 lbs.	940023 & 940026
Output	52.0 lbs.	67.5 lbs.	93.9 lbs.
Yield	86%*		84%
Power	90 KW	100 KW	100 KW
Vacuum	.02 Micron	.02 micron	.02 micron
Melting Rate	2 in/hr	3 in/hr	3 in/hr

*The percentages of yield for first E.B. melts were based on the input weight of vanadium in the Reductions. Considering the aluminium content in Reductions averaging 15.5%, the true weight of vanadium input was 139 pounds.

TABLE 31
Analytical Data of V_2O_5 , Vancoram

<u>Sample No.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>Ave.</u>
C	<40	<40	<40	<40	<40
N	17	14	27	25	21
Al	420	410	450	530	452
B	<1	<1	<1	<1	<1
Cd	<5	<5	<5	<5	<5
Co	<10	<10	<10	<10	<10
Cr	<20	<20	<20	<20	<20
Cu	<40	<40	<40	<40	<40
Fe	<50	<50	<50	<50	<50
Mg	<20	<20	<20	<20	<20
Mn	<20	<20	<20	<20	<20
Mo	<20	<20	<20	<20	<20
Ni	<20	<20	<20	<20	<20
Pb	<20	<20	<20	<20	<20
Si	350	305	300	350	326
Sn	<20	<20	<20	<20	<20
Ti	<50	<50	<50	<50	<50

- Note: (1) Data are expressed in ppm on metallic V basis.
 (2) Samples are taken by using a sampling thief, one from each 100-lb. barrel.

TABLE 32

Analytical Data of Al Powder, Alcan MD 101
(ppm)

O	300
N	5
C	120
B	< 5
Cd	< 5
Co	<10
Cr	<20
Cu	<40
Fe	<100
Mg	<20
Mn	<20
Mo	<20
Ni	<20
Pb	<20
Si	<100
Sn	<20
Ti	<50
V	50

TABLE 33
Reduction Data of Vanadium Reductions
Series V

Lot No.	Input			Output			
	V ₂ O ₅ lbs.	Al, lbs.	Al of Theoretical	Reduction, lbs.	V Content	V in Reduction	Yield
V-1-11-68	100	59	119%	60	81.5%	49	87.5%
V-1-13-68	100	59	119%	56	87.1%	48.8	87.0%
V-1-27-68	100	59	119%	56	89.9%	50.1	89.8%
V-1-29-68	102	59.5	118%	57	87.8%	49.5	89.0%

Table 34

Analytical Data, Series V Reductions

Lot No.	V-1-11-68				V-1-13-68				V-1-27-68				V-1-29-68				Grand Ave
Sample No.	1	2	3	Ave	1	2	3	Ave	1	2	3	Ave	1	2	3	Ave	
O	1960	1450	1390	1600	1920	2510	2170	2200	5500	4100	5100	4900	5300		3000	4150	3210
N	60	65	60	62	60	60	65	62	50	55	55	57	70		67	69	63
C	60	60	40	53	80	80	60	60	73	<40	<40	<40	<40		<40	<40	52
Si	210	270	170	217	260	200	120	193	230	270	280	260	740	405	660	601	318
Al	17.5%	18.5%	19.5%	18.5%	13.1%	13.0%	12.7%	12.9%	10.3%	10.0%	10.3%	10.2%	11.42%		12.91%	12.2%	13.6%
B	2	2	2	2	7	2	4	4	1	1	1	1	<1	<1	<1	<1	2
Cd	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
Co	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
Cr	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Cu	250	250	250	250	250	250	250	250	750	750	1000	833	250	250	500	333	417
Fe	800	880	720	800	520	580	470	523	500	480	480	487	500	490	610	533	586
Mg	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Mn	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Mo	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Ni	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Pb	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Sn	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Ti	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50

Note: (1) Units are ppm unless otherwise indicated.

(2) O samples are solids taken from the circumference at equal distance along the length of the ingot from top to bottom.

(3) Other samples are milled fines taken from the circumference along the length of the ingot, from top to bottom.

Table 35
Melting Data
First E.B. Melt, Series V

Ingot No. <u>940029-S4A</u>		<u>940030-S4A</u>	
Input Reductions 1-29-68 and turning		Reductions 1-11-68, 1-13-68, 1-27-68	
Gross Wt.	66.8 lbs.		135.0 lbs.
Minus Al 12.2%	<u>8.2</u>	Minus Al 13.9%	<u>18.8</u>
Net V Wt	58.6		116.2
Output (6"dia. x 8")	47.8 lbs.	(6" dia. x 17.5")	100.3 lbs.
Yield	82.0%		89.0%
Power	90-100 kw		90-100 kw
Vacuum	.02 Micron		.02 Micron
Melting Rate	3 in/hr		3 in/hr

(continued)

Table 35 (cont.)

Melting Data, Third E.B. Melt, Series V

Ingot No. 940034-XIC

Input	Ingot No. 940031 and 940032 114.0 lbs.
Output	6" dia x 19" 101.5 lbs.
Yield	89.0%
Power	80 kw
Vacuum	.07 Micron
Melting Rate	3 in/hr

(continued)

TABLE 35 (cont.)

Melting Data

Second E.B. Melt, Series V

<u>Ingot No.</u>	<u>940031-S4B</u>	<u>940032-S4B</u>
Input	Ingot No. 940029-S4A 44.5 lbs.	Ingot No. 940032-S4A 96.0 lbs.
Output	(6" dia x 7") 38.5 lbs.	(6" dia x 14.5") 85.1 lbs.
Yield	86.5%	89.0%
Power	90 kw	Start 60 kw for 2" then 90 kw
Vacuum	.02 Micron	.02 Micron
Melting Rate	3 in/hr	3 in/hr

TABLE 36
Hardness Data, BHN
First E.B. Melt, Series V

<u>INGOT No.</u> (Top)	<u>940029-S4A</u> (3,000 kg. load)	<u>940030-S4A</u> (500 Kg. load)
1	96	92.6
2	92	119.0
3	96	92.6
4	96	100.0
5	86	100.0
6	87	98.5
7	86	92.6
8	83	104.0
9	92	158.0
10	92	96.3
11		100.0
12		100.0
13		104.0
14		109.0
15		50.3
(Bottom)		
Ave.	91	100.5

(continued)

TABLE 36 (cont.)

Hardness Data

BHN, 500 kg load

Second E.B. Melt, Series V

<u>INGOT NO.</u>	<u>940031-S4B</u>	<u>940032-S4B</u>
(Top)	(500 kg load)	
1	100	69.1
2	79.6	66.8
3	74.1	69.1
4	76.8	71.5
5	74.1	66.8
6	74.1	62.5
7		71.5
8		69.1
9		66.8
10		64.6
11		64.6
12		66.8
13		71.5
14		71.5
(Bottom)		
Ave	80	68

(continued)

Table 36 (cont.)

Hardness Data

BHN, 500 kg load

Third E.B. Melt, Ingot No. 940034-XIC, Series V

(500 kg load)

(Top)

1	71.5
2	69.1
3	82.5
4	89.0
5	74.1
6	64.6
7	64.6
8	69.1
9	69.1
10	69.1

(Bottom)

Ave	72
-----	----

TABLE 37
Analytical Data
First Melt Ingots, Series V

Ingot No.	940029				940030						Weighted Ave.
Sample No.	1	2	3	Ave	1	2	3	4	5	Ave	
O	340	320	260	307	110	200	160	210	230	182	224
H	3.6	5	4.8	5	5	6	5	5	6	5	5
N	85	88	80	84	91	100	95	100	97	97	93
C	40	40	90	57	<40	<40	<40	<40	<40	<40	55
Si	430	380	520	443	260	350	370	325	370	338	373
Fe	.54%	.42%	.44%	.47%	225	210	420	400	290	369	.17%
Al	.9%	.8%	.3%	.67%	.78%	.65%	1%	1%	.9%	.86%	.80%
B	<1	<1	<1	<1	1	1	1	<1	1	1	<1
Cd	<5	<5	<5	<5	<1	<1	<1	<1	<1	<1	<5
Co					<10	<10	<10	<10	<10	<10	<10
Cr	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Cu	<40	<40	<40	<40	<40	<40	<40	<40	<40	<40	<40
Mg	25	<20	<20	22	<20	<20	<20	<20	<20	<20	<20
Mn	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Mo	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Ni	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Pb	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Sn	<20	<20	<20	<20	<10	<10	<10	<10	<10	<10	<20
Ti	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50
W	20	10	90	40	20	20	30	20	30	24	32

NOTE: (1) Units are ppm unless otherwise indicated.

(2) O and H samples are solids taken from the circumference at equal distance along the length of the ingot, from top to bottom.

(3) Other samples are milled fines taken from the circumference along the length of the ingot from top to bottom.

TABLE 38
Second E.B. Melt

Ingot No. Sample No.	940031-S4B				940032-S4B						Weighted Ave
	1	2	3	Ave	1	2	3	4	5	Ave	
O	210	360	370	313	280	210	410	210	330	288	296
H	6	6	5	6	4.3	4.6	4.4	4.4	4.8	4.5	5
N	110	85	107	101	80	94	87	95	105	92	95
C	70	60	60	63	90	70	70	70	50	70	68
Si	240	420	440	367	130	140	230	190	190	176	240
Fe	50	90	80	73	<50	<50	200	80	96	95	88
Al	200	580	270	350	195	130	.1%	500	660	497	448
B	<1	1	1	1	1	1	1	<1	<1	<1	<1
Cd	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
Co	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
Cr	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Cu	<40	<40	<40	<40	<40	<40	<40	<40	<40	<40	<40
Mg	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Mn	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Mo	<20	<20	25	22	<20	<20	<20	<20	<20	<20	<20
Ni	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Pb	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Sn	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Ti	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50

- NOTES: (1) Units are ppm unless otherwise indicated.
- (2) O and H samples are solids taken from the circumference at equal distances along the length of the ingot, from top to bottom.
- (3) Other samples are milled fines taken from the circumference along the length of the ingot, from top to bottom.

TABLE 39

Analytical Data

Third E.B. Ingot No. 940034-XIC, Series V

Sample No.	1	2	3	4	5	Ave.
O	140	200	140	210	200	178
H	7	12	11	10	7	9
N	125	100	80	90	100	99
C	50	50	40	60	40	50
Si	280	360	160	420	370	318
Al	<20	<20	<20	<20	<20	<20
B	1	1	1	1	1	1
Cd	<5	<5	<5	<5	<5	<5
Co	<10	<10	<10	<10	<10	<10
Cr	<20	<20	<20	<20	<20	<20
Cu	<40	<40	<40	<40	<40	<40
Fe	<100	<100	<100	<100	105	<100
Mg	<20	<20	<20	<20	20	<20
Mn	<20	<20	<20	<20	<20	<20
Mo	<20	<20	<20	<20	<20	<20
Ni	<20	<20	<20	<20	<20	<20
Pb	<20	<20	<20	<20	<20	<20
Sn	<20	<20	<20	<20	<20	<20
Ti	<50	<50	<50	<50	<50	<50
W	60	140	30	40	20	59

Notes: (1) Units are in ppm

(2) O and H samples were solids taken from the circumference equally spaced along the length of the ingot from top to bottom.

(3) Other samples were milled fines taken from the circumference along the length of the ingot from top to bottom one after another.

TABLE 40
CHEMICAL DATA
Alloying Material for V-15Cr-5Ti
Weight Percent

Elchrome H.P.		English Ti Sponge		
Lot No. 37645		G 466 Ti		
	Vendor's Analysis	Wah Chang Analysis	Vendor's Analysis	Wah Chang Analysis
H	<.001	.057		.0013
O	<.002		.079	
C	<.01	.009		.006
N	.008	.01	.011	.02
Si	<.008			.02
S	.008			
Fe	<.01			
Cr	99.95			
Cu				.01
Sn				.02

TABLE 41
CHEMICAL ANALYSES

(ppm)

V-15Cr-5Ti, Heat No. 4.5-950059

Sample No.	1	2
C	120	120
N	225	210
H	9	---
O	940	950
Al	<50	<50
B	<1	<1
Cd	<5	<5
Co	<10	<10
Cu	75	75
Fe	240	260
Mg	<20	<20
Mn	<20	<20
Mo	<20	<20
Ni	<20	<20
Pb	<20	<20
Si	330	340
Sn	<20	<20
Cr	15.05%	15.19%
Ti	6.1	6.1
W	390	400

Table 42
Analysis of Alloy Constituents

Titanium Crystal Bar Supplied by General Electric Co., Evendale, O. Wah Chang Albany Analyses Filament No.					Chromium, Electrolytic Flake Electrochrome H.P. Lot # 37667		
Element	601-4 Tip	Tip	604-7 Center	Bottom	Vendor's Analysis	Wah Chang Coarse	Wah Chang Fine
Ti	-	-	-	-	-	<20	<20
Cr	<20	<20	<20	<20	99.947%	99.93%*	99.8%*
Al	<20	<20	<20	<20	-	25	100
B	1.3	1.0	1.5	1.5	-	<1	<1
Cd	<5	<5	<5	<5	-	<5	<5
Co	<10	<10	<10	<10	-	<10	<10
Cu	40	<40	40	40	-	<40	200
Fe	262	175	175	172	100	<100	<100
Mg	20	20	20	20	-	40	500
Mn	75	50	75	75	-	<20	<20
Mo	<20	<20	<20	<20	-	<20	<20
Ni	<20	<20	<20	25	-	<20	<20
Pb	<20	<20	<20	<20	-	<20	<20
Si	<100	<100	<100	<100	80	250	500
Sn	<10	<10	<10	<10	-	<20	<20
V	<20	<20	<20	<20	-	<20	<20
W	250	200	200	250	-	-	-
Zr	50	<50	<50	50	-	-	-
C	100	80	60	70	100	<40	<40
N	35	33	29	29	100	20	20
O	300	290	140	280	40	127 170	327 280
O**	430	260	160	-	-	-	-
H	-	-	-	-	10**	-	-
S	-	-	-	-	100	-	-

* Analysis by difference

** Oregon Metallurgical Corporation Analysis

Note: All units are ppm except as noted.

TABLE 43

Melting Data, Arc Melt

	<u>3rd Melt</u>	<u>4th Melt</u>
Ingot No.	4-950065	4-950067
Input	Scrap from 950063 49.2 lbs.	Scrap from ingot No. 4-950063 and 4-950065, 36.9 lbs.
Output	48.2 lbs.	4" diameter X 15" 36.2 lbs.
Yield	98%	98%
Power	124 kw	105 kw
Atmosphere	Vacuum max. 50 micron	Vacuum max. 30 micron

TABLE 44

Chemical Analyses, Arc Melt V-15Cr-5Ti, (ppm).

Heat No.	4-950065				4-950067		
Sample No.	1	2	3	Ave.	Top	Bottom	Ave.
C	170	140	180	163	220	100	160
N	98	94	100	97	102	98	100
H	3.0		2.5	3	4	4	4
O	220		230	225	410	280	345
Al	25	<25	<25	<25	<50	<50	<50
B	2	2	2	2	<1.5	<1.5	<1.5
Cd	<5			<5	<5	<5	<5
Co	<10			<10	<10	<10	<10
Cu	<40	<40	<40	<40	100	250	175
Fe	150	150	150	150	50	<50	<50
Mg	<20	<20	<20	<20	20	20	20
Mn	<20	<20	<20	<20	<20	<20	<20
Mo	<20	<20	<20	<20	<20	20	<20
Ni	<20	<20	<20	<20	20	20	20
Pb	<20	<20	<20	<20	<20	<20	<20
Si	500	250	300	350	500	500	500
Sn	20	<20	20	<20	50	20	35
Ti	5.04%	5.15%	5.21%	5.13%	5.95%	5.75%	5.85%
Cr	15.05%	15.0%	14.8%	15.0%	14.8%	15.1%	14.95%

TABLE 45

SOME THERMODYNAMIC VALUES OF V, Y, Al, Si, C AND THEIR OXIDES, NITRIDES AND CARBIDES

Material	Free Energy of Formation		Vapor Pressure		Melting Point	Boiling Point
	At 200°C ΔF : Cal		At 2000°C, mm		°C	°C
	Per Mole of Gas Atom		Ref. 2	Ref. 3	Ref. 1	Ref. 1
V			7×10^{-2}	3.06×10^{-2}	--	3450
Y			8	1.74×10^{-1}	1059	2927
Al			70	20.8	660	2450
Si			5	--	1410	2680
C			--	--	Sublimes	4350
VO	-54,100	Ref. 1	0.3		--	3127
YO	N.A.		N.A.		N.A.	N.A.
Y_2O_3	-95,700	Ref. 1	N.A.		2270	4300
AlO	-28,100	Ref. 1	N.A.		N.A.	N.A.
Al_2O_3	-74,700	Ref. 1	N.A.		N.A.	N.A.
Al_2O_3	-76,700	Ref. 1	0.4		2040	2500
SiO_2	-53,500	Ref. 1	8×10^{-3} (glass)		1953	N.A.
CO	-68,000	Ref. 1	--		--	--
VN	+69,000	Ref. 2	N.A.		2047	N.A.
YN	-61,000 at 25°C	Ref. 1	N.A.		N.A.	N.A.
AlN	+ 1,200	Ref. 1	N.A.		2227	2490
	+ 9,800	Ref. 2	--		--	--
VC	-18,000	Ref. 1	N.A.		2827	N.A.
SiC	- 8,650	Ref. 1	N.A.		2700	N.A.
TiC	-38,700	Ref. 1	N.A.		3177	N.A.

Ref. 1: Bulletin 605 -- U. S. Bureau of Mines

Ref. 2: O. Kubaschenski: Metallurgical Thermochemistry

Ref. 3: R. Holtgren: Selected values of thermodynamic properties of metals and alloys.

N.A. -- Not available

TABLE 46

Microhardness of Ingot No. 940027 Heat Treated Samples
(D.P.H., 10 Kg. load)

	Top	Bottom
As Cast	113	190
1 hr.-1100°C	113	164
1 hr.-1500°C	119	179
1 hr.-1100°C and 4 hrs. at 704°C	108	189
1 hr.-1500°C and 4 hrs. at 704°C	116	195

TABLE 47

As Cast Hardness of V-15Cr-5Ti Ingots

Ingot No.	950059	950062	950063	950065	950067
Total					
Interstitial	1300	288	371	488	609
Ingot Hardness					
BHN, 3000 Kg	227		215	203	210
Microhardness					
DPH, 10 Kg	225		213	200	212

TABLE 48

Hardness Data of Heat Treated Samples of
Different Heats of V-15Cr-5Ti

	<u>950059</u>	<u>950063</u>			<u>As Cast</u>	<u>950065</u>	
	2" plate	.040" sheet .125" sheet				.125	.225
		(A)	(B)	(C)			
As worked	279	280	215		213	262	239
2 hr. anneal, °C							
700		261	257	258			
800		244	243	244		241	
900		227	213	235			
950		200					
1000		208	192	208	203		
1100		206	194	203	210	206	
1200	204	196			206	213	200
1300		211			222	207	
1400	226	218					

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