CEND-3742-336

VANADIUM PURIFICATION

SIXTH QUARTERLY PROGRESS REPORT

Issued April, 1968

NUCLEAR POWER DEPARTMENT
COMBUSTION ENGINEERING, INC.

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CEND-3742-336
UC-25, Metals, Ceramics
and Materials
TID-4500, 51st Edition

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SIXTH QUARTERLY

PROGRESS REPORT

For Period

January 1 to March 31, 1968

Prepared by:

C. T. Wang, Wah Chang, Albany, Oregon
E. F. Barock, Wah Chang, Albany, Oregon
J. C. La Vake, CENPD
S. S. Christopher, CENPD

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W. P. Chernock, Manager
Nuclear Laboratories

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COMBUSTION ENGINEERING, INC.
WINDSOR, CONNECTICUT

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VANADIUM PURIFICATION

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ABSTRACT

During this report period, a 100-pound high purity V-15Cr-5Ti alloy ingot was produced with a total interstitial content of less than 400 ppm.

The process developed for the production of the alloy ingot was as follows:

1. Aluminothermic reduction of V_2O_5
2. Consolidation by electron beam melting of the reduced vanadium derbies
3. Double electron beam melting
4. Compaction of vanadium, titanium and chromium flakes and chips
5. Double arc melt, first in argon, then in vacuum.

I INTRODUCTION

This program was initiated to produce a 100-pound, high purity vanadium ingot by a process suitable for commercial applications. This material will be used as feedstock for producing a high purity, V-15Cr-5Ti, alloy ingot. Mechanical properties of the alloy will be determined to evaluate the effect of purity on the tensile, stress rupture and weld characteristics of the alloy.

As reported in the fifth quarterly report (CEND-3742-329), it was proposed that portions of ingots 3 and 4 be blended to provide feedstock for the alloy ingot. However, it was felt that higher purity material was producible if sufficiently pure V_2O_5 could be obtained. Accordingly, high purity V_2O_5 was obtained from Vancoram and a fifth ingot produced. The resulting 101-lb ingot was of high purity containing <350 ppm interstitials. Since the entire fifth ingot was used to produce the alloy ingot, selective blending was not necessary.

II EXPERIMENTAL EFFORT

During this quarter, a 100-pound high purity V-15Cr-5Ti alloy ingot was produced using commercial melting techniques.

The alloy ingot was produced using the fifth ingot, melted during this report period as feedstock. Four batches of high purity V_2O_5 from Vancoram were aluminothermically reduced using high purity Alcan MD101 aluminum powder to provide the high purity vanadium ingot. The chemical analyses of the V_2O_5 and the aluminum powder are shown in Tables I and II, respectively.

TABLE I

CHEMICAL ANALYSES OF VANCORAM V_2O_5

Used for the Fifth Ingot (ppm)

Batch No.	1	2	3	4	Avg
C	<40	<40	<40	<40	<40
N	17	14	27	25	21
Al	420	410	450	530	452
Si	350	305	300	350	326

TABLE II

CHEMICAL ANALYSES OF ALCAN MD101

Aluminum Powder

Element	Level (ppm)
O	300
N	5
C	120
Si	<100
Fe	<100

The reduction yield of the V_2O_5 was quite good, approximately 88% of theoretical. The chemical analyses of the four derbies (reductions) are listed in Table III.

Derby No. V-1-29-68 was analyzed to be high in silicon, therefore it was electron beam melted separately into Ingot No. 940029-S4A. The other derbies were melted into Ingot No. 940030-S4A. The analytical data and hardness values for the two ingots after consolidation are shown in Table IV.

Both ingots were remelted using the same techniques as in the first melt. The analytical data and hardness values are shown in Table V. The analytical data show the total interstitial contents of Ingots Nos. 940032 and 940031 to be 455 and 483 ppm and the silicon contents 176 and 367, respectively. Both were well below the target values of the program (i.e., 500 ppm interstitial and 500 ppm silicon). However, the oxygen contents were comparatively high (296 ppm average) as were the aluminum (448 ppm average). With the expectation that the oxygen level would be reduced by combining with aluminum, we decided to electron beam melt a third time.

The two second electron beam melted ingots were then melted into one ingot, with the higher interstitial content ingot (No. 940031) forming the top portion of the ingot. The analytical and hardness values are shown in Table VI. As shown, the ingot is quite homogeneous with an interstitial level of 336 ppm and a silicon level of 318 ppm. The microstructure from top, center and bottom locations of the ingot are shown in figure 1. The cleanliness of the pure metal, compared with the microstructures from Ingots Nos. 2 and 3, is shown in figure 2.

TABLE III

ANALYTICAL DATA OF DERBIES FOR INGOT NO. 5

Element	Derby No. V-1-11-68	V-1-13-68	V-1-27-68	V-1-29-68	Avg.
O	1600	2200	4900	4150	3210
N	62	62	57	69	63
C	53	60	<40	<40	52
Si	217	193	260	601	318
Al	18.5%	12.9%	10.2%	12.2%	13.6%
Fe	800	523	487	333	417

TABLE IV

ANALYTICAL AND HARDNESS DATA OF THE CONSOLIDATION MELTS FOR INGOT NO. 5

Element (ppm)	Ingot No. 940029-S4A (58.6 lb)	Ingot No. 940030-S4A (116.2 lb)	Weighted Avg.
O	307	182	224
H	5	5	5
N	84	97	93
C	57	<40	55
Si	443	338	373
Fe	4700	369	1700
Al	6700	8600	8000
Hardness BHN	91	100	

TABLE V

ANALYTICAL AND HARDNESS DATA OF THE SECOND ELECTRON BEAM MELT FOR INGOT NO. 5

Element	Ingot No. 940031-S4B (38.5 lb)	Ingot No. 940032-S4B (85.1 lb)	Weighted Avg.
O	313	288	296
H	6	4.5	5
N	101	92	95
C	63	70	68
Si	367	176	240
Fe	73	95	88
Al	350	497	448
Hardness			
BHN	80	68	

TABLE VI

ANALYTICAL AND HARDNESS VALUES OF THE THIRD ELECTRON BEAM MELTED FIFTH INGOT (101.5 lb)

Element	Sample No.					Avg.
	Top 1	2	3	4	Bottom 5	
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
Fe	<100	<100	<100	<100	105	<100
Hardness						
BHN	71.5	82.5	74.1	69.1	69.1	72

Permission was obtained from the Atomic Energy Commission to use the fifth ingot for the alloy ingot instead of the originally proposed procedure of blending the better parts of ingots Nos. 3 and 4.

As requested by the AEC Reactor Development and Technical Division, 9.87 pounds of research grade titanium crystal bar were obtained from General Electric Co., Evendale, Ohio, to be used in the production of the V-15Cr-5Ti alloy.

The chemical analyses of the titanium bars are shown in Table VII.

TABLE VII

ANALYTICAL DATA OF Ti CRYSTAL BAR,
GENERAL ELECTRIC CO., EVENDALE, OHIO

Element	Level, ppm		
	Top	Center	Bottom
O	290	140	280
N	33	29	29
C	80	60	70
Si	<100	<100	<100
W	200	200	250

High purity chromium flakes (Elchrome H.P.) were purchased from Union Carbide Company, Cleveland, Ohio. The analytical data showed the coarser particles to be the more pure. As a result, the material was screened and the coarser particles used for alloying. The analytical results are shown in Table VIII.

TABLE VIII

ANALYTICAL DATA OF ELCHROME FLAKES, UNION CARBIDE CO., CLEVELAND, OHIO

Element	Level, ppm
O	145
N	20
C	<40
Si	250

The following procedure was used for producing the alloy ingot:

1. The vanadium ingot was carefully machined into coarse chips. Contamination during machining was minimized by constructing a hood around the lathe and doing the actual mocking in an argon atmosphere. In addition, a stream of argon was directed at the tool tip to cool the work area, as shown in figure 3.

2. The constituents were compacted into five 2 x 2 x 20 inch compacts with a composition of 79.7% vanadium, 15.2% chromium and 5.1% titanium. The increase above nominal for chromium and titanium was to allow for some volatilization during melting. Figure 4 shows two such compacts formed into an arc melt electrode by T.I.G. welding in an inert gas chamber.

3. The electrodes were arc melted under an argon atmosphere into a 5-inch diameter ingot. A partial pressure of argon was used for the first melt to reduce chromium volatilization. The second melt was performed under a dynamic vacuum since volatilization is diminished by alloy partial pressure effects. Also, high vacuum was desired to minimize the hydrogen content.

The double arc melted ingot is shown in figure 5. The melting parameters for both melts are listed in Table IX.

The chemical and hardness values for the single and double arc melted alloy ingots are shown in Table X. Metallographical samples taken from the top slice are shown in figure 6. Macrographic sections from

TABLE IX

MELTING DATA
V-15Cr-5Ti ARC MELT

	1st Arc Melt	2nd Arc Melt
Heat No.	5-950062	56.5-950063
Input	V-15Cr-5Ti compacts - 117 lb	5-950062 113.3 lb
Output	5 in. dia., 117 lb	6.5 in dia., 112.7 lb
Yield	~100%	99.5%
Power	112 kw	124 - 192 kw
Atmosphere	25 in. argon	Vacuum, max 30 micron

the top and bottom are shown in figure 7. A cracking phenomenon, apparently associated with the shrinkage cavity, was noted in the ingot. It is thought that the cooling rate may be instrumental in its formation since hot topping operations were not very uniform.

Ultrasonic inspection of the ingot showed it to be free of defects below 5 inches from the top, above which there were shrinkage cavities and associated cracks.

In preparation for the fabrication of this ingot into sheet, a series of heat treatments was performed on material from the top of the ingot. The metallographical results are shown in figure 8. Note that the second phase appears to be going into solution after 6 hours at 1200°C and 2 hours at 1300°C. The nature of the very fine porosity occurring in conjunction with the second phase is similar to pores occurring in columbium-based alloys containing group IV A metals. The fineness and rarity of these

TABLE X

ANALYTICAL DATA, V-15Cr-5Ti

Elements	First Arc Melt Ingot No. 5-950062						Second Arc Melt Ingot No. 56.5-950063					
	Sample No.						Sample No.					
	1	2	3	4	5	Avg.	1	2	3	4	5	Avg.
Cr	14.9%	14.9%	14.8%	14.7%	14.8%	14.8%	15.2%	15.5%	15.6%	16.0%	15.8%	15.6%
Ti	4.7%	4.2%	3.3%	3.1%	3.4%	3.7%	5.38%	5.45%	5.66%	5.85%	5.64%	5.6%
O	70		<50		170	97	80		140		220	150
H	12		12		7	10	4.4				4.3	4.4
N	87	87	87	87	87	87	85	90	103	117	91	97
C	130	90	90	70	90	94	110	130	140	160	150	130
Si	250	300	250	200	250	250	300	300	300	350	300	310
Al	50	50	<50	<50	50	<50	100	50	50	50	100	70
B	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
Cd	<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
Cu	<40	<40	<40	<40	<40	<40	50	50	<40	<40	50	46
Fe	100	100	<100	100	100	<100	100	150	150	<100	150	130
Mg	<20	<20	<20	<20	<20	<20	20	20	<20	<20	35	23
Mn	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Mo	35	25	25	50	25	31	<20	50	50	100	20	48
Ni	<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
Sn	50	<20	<20	<20	<20	26	<20	<20	<20	<20	<20	<20

Remarks: (1) Units are in ppm unless otherwise indicated
(2) For Ingot No. 950062, O and H are analyzed from solid samples, others are fine turnings taken from the circumference of the ingot from top to bottom.
(3) 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.

pores suggest that no problems will result. It is possible, since the samples are from the top of the ingot, that the pores are related to the shrinkage. This would be similar to a cup of porosity which is always present under the main shrinkage cavity on zirconium and titanium-based ingots.

In an effort to compare analytical techniques between laboratories, vanadium samples were sent to Vancoram Corporation, Ames Laboratory and others for analyses. The results, shown in Tables XI and XII, indicate general agreement among the labs, considering that different techniques of analysis were used. The discrepancy of silicon content is likely attributed to the nonhomogeneous nature of the particular sample; however, a "round-robin" series of tests would be in order.

TABLE XI

COMPARISON OF ANALYSES OF SILICON IN VANADIUM (ppm)

Sample No.	A	B	C	D	E
Wah Chang (AC arc Spectrography)	4270	830	680	1400	420 ± 45
Bureau of Mines Boulder City, Nevada	4200				
Vancoram Cambridge, Ohio (Chemical)		870			
Kaman Nuclear Colorado Springs, Colo. (Neutron Activation)			360	1500	
Bureau of Mines Albany, Oregon (Chemical)					100 ± 30

TABLE XII

COMPARISON OF HARDNESS AND ANALYSES OF IMPURITIES IN VANADIUM (ppm)

Element	Wah Chang	Fines Vancoram	Ames Lab	Solids Ames Lab
Si	830 520	870 -	- 540	- 457
C	220	-	268	235
Al	280	-	450 & 100	240
Mo	700	-	700	-
Hardness	BHN 164	-	-	BHN 168

III DISCUSSION OF RESULTS

The melting history for the fifth vanadium ingot indicates a substantial reduction in the complexity of the purifying process. Specifically, when the proper amount of excess aluminum is added to the aluminothermic reduction, the yttrium doping is no longer required to lower the oxygen content to acceptable values (as reported in the last quarterly report). This is shown in figure 9. The effect of vacuum melting on the oxygen content has been plotted for Ingots No. 2 through No. 5. The aluminothermic reduction of Ingots No. 4 and No. 5 were run in a modified furnace as described in the fifth quarterly report. The modification eliminates the need for a reaction moderator (Al_2O_3) and reduces contamination. Thus, for Ingots No. 4 and No. 5, a more complete and controlled reaction lowered the initial oxygen content as compared to Ingots No. 2 and No. 3. Subsequent reductions of the oxygen level were experienced during electron beam melting as a result of the reaction of oxygen with aluminum.

The carbon content was essentially of the lowest detectable value from the starting derbies through the third electron beam melt, with a final average value of 50 ppm. The content from melt to melt, as shown in figure 10, was very consistent, probably due to the homogeneity of the material in each case. The variations in Ingots Nos. 2, 3 and 4 would indicate a less homogeneous material.

The nitrogen content started at an average of 52 ppm in the derbies and remained below 100 ppm from the first through the third electron beam melts as shown in figure 11. There was a distinctive difference between Ingot No. 5 and any previous series, in which nitrogen tended to increase

after each melt. Possible reasons for this and the very constant silicon content shown in figure 12 are:

a) Extra high vacuum (0.02 micron) and very low leak rate (~0.05 micron/minute) were used during electron beam melting;

b) Low power was used throughout all three electron beam melts (70-100 kw), which resulted in less loss due to vaporization;

c) A melting rate was determined to provide a compromise between the elimination of impurities and maximum yield. The melting rate (3 in./hr) closely approximated that used at Ames Laboratory.

It is apparent from figure 13 that the hardness values of the fifth ingot were much lower than all the other ingot series in this program. Ingot No. 5 started at an average hardness of BHN 94 for the first melt and dropped down to 72 in both the second and third melts. This compares very favorably with the DPH 69 value determined at Ames Laboratory for laboratory scale electron beam melted vanadium.

While hardness is a general indication of purity, it is not possible to use it at this stage as an absolute control method. The effect of yttrium doping is to drastically reduce hardness without a proportional reduction in contaminants. Surface preparation prior to hardness testing apparently also interferes with the hardness test. The vanadium-oxygen system also suggests that heat treating affects the hardness even at low oxygen contents. Noting this, samples were taken from Ingot No. 4 for heat treatment studies. From the results shown in Table XIII, it is apparent that the as-cast hardness is not the dead soft value of the metal.

The percentage yield of Ingot No. 5, together with those of the previous ingots, are shown in figure 14. The values are based on the theoretical amount of vanadium in the derbies.

TABLE XIII

MICROHARDNESS OF INGOT NO. 4 HEAT TREAT 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. @ 704°C	108	189
1 hr. @ 1500°C and 4 hrs. @ 704°C	116	195

It can be seen that Ingot No. 5 had the highest yield among all the ingots. The yield per each melt ranged from 84.5 percent to 89.0 percent. Comparing the average yield per melt for the laboratory operation at Ames Laboratory (private communication), 90 percent, the scale-up yield for Ingot No. 5 is very satisfactory. The reasons for the high yield are the same as those listed for the low nitrogen content. The third electron beam melt may be unnecessary in the future if the metal reaches its high purity level after the second melt.

IV CONCLUSIONS

No technical obstacles remain in the large-scale production of vanadium meeting the target purities using the process based on the work by Carlson at Ames Laboratory. Use of transverse electron beam guns has eliminated the need for the aluminum deoxidation sinter.

Scale-up of the aluminothermic reduction to individual runs of 300 pounds of metal will parallel current aluminothermic reduction practice for columbium. No additional melting is planned under Task I.

V FUTURE WORK

The alloy ingot will be machined, checked metallographically and chemically, ultrasonically inspected and cut into two sections. One section will be used for producing 0.040-inch thick sheet, the other will be single extruded to a 2-inch diameter tube bar for a possible further reduction into tubing at Argonne National Laboratory.

Both sections will be homogenized at 1200°C and canned. The processing procedures for the two billets are as follows:

Procedure for Sheet Production

1. The Stainless Steel or Inconel-canned, 6-inch diameter billet with a 120-degree angle nose will be extruded at 1100°C with an extrusion ratio of 9:1 to a sheet bar of 1.5 inches x 3 inches x length.
2. After de-canning, the sheet bar will be annealed at 1100°C for 2 hours.
3. Sheet bar will be cut into 15-inch lengths and re-canned.
4. Cross-rolled at 1100°C to approximately 0.60-inch thickness.
5. De-canned and annealed at 900°C for 1 hour.
6. Rolled at 300°C to 0.24 inch.
7. Annealed at 900°C for 1 hour.
8. Rolled at room temperature to 0.10 inch.
9. Annealed at 900°C for 1 hour.
10. Rolled to final size (0.040 inch) at room temperature.

Procedure for Tube Bar Production

The second Stainless Steel or Inconel-canned, 6-inch diameter billet will be extruded at 1100°C with an extrusion ratio of 9:1 to a 2-inch diameter.

This diameter was selected in cooperation with Mr. Neil Carson of Argonne National Laboratory to facilitate their handling of a possible tube drawing program.

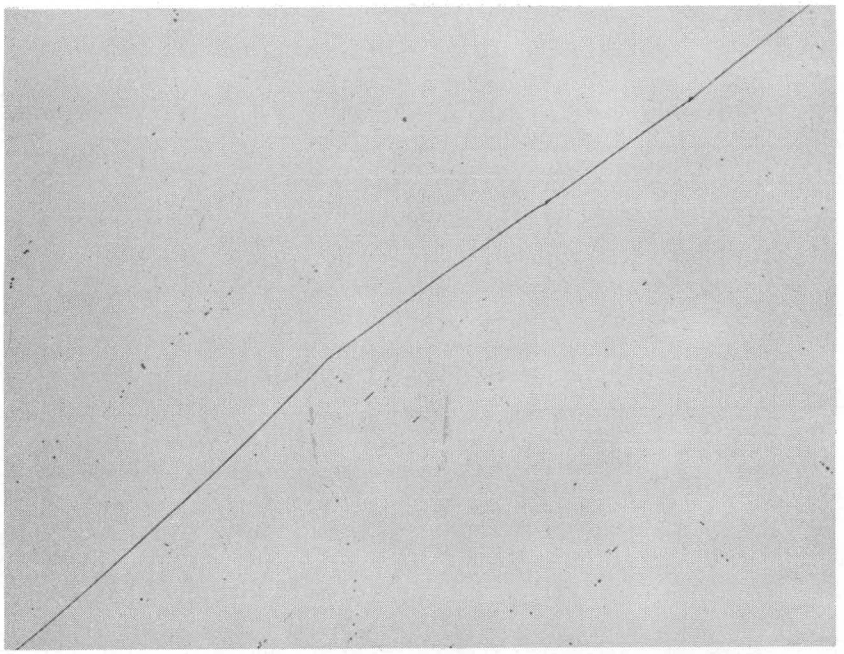

Figure 1A: Microstructure Of Ingot No. 5
Top-Center Mag. 150X

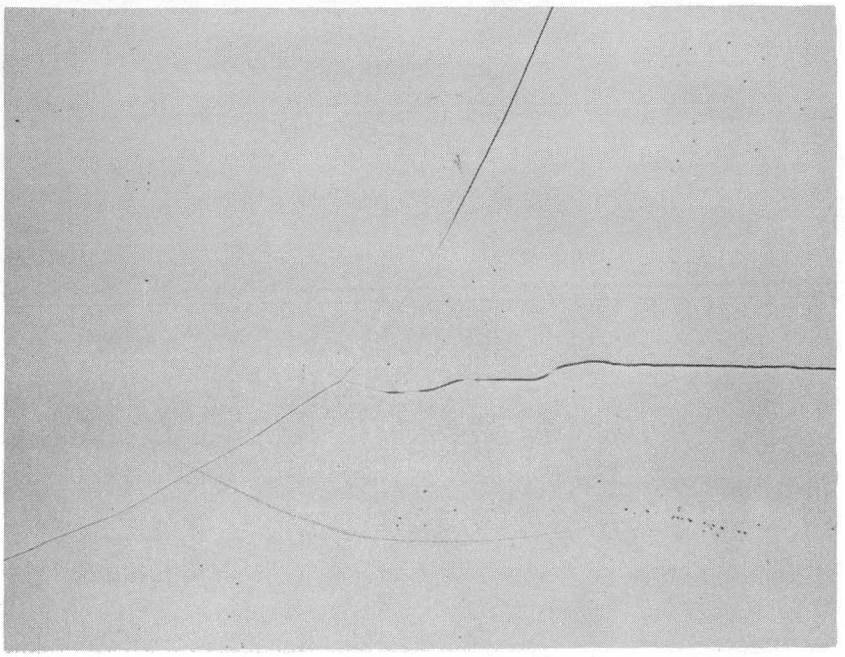

Figure 1B: Microstructure Of Ingot No. 5
Center-Edge Mag. 150X

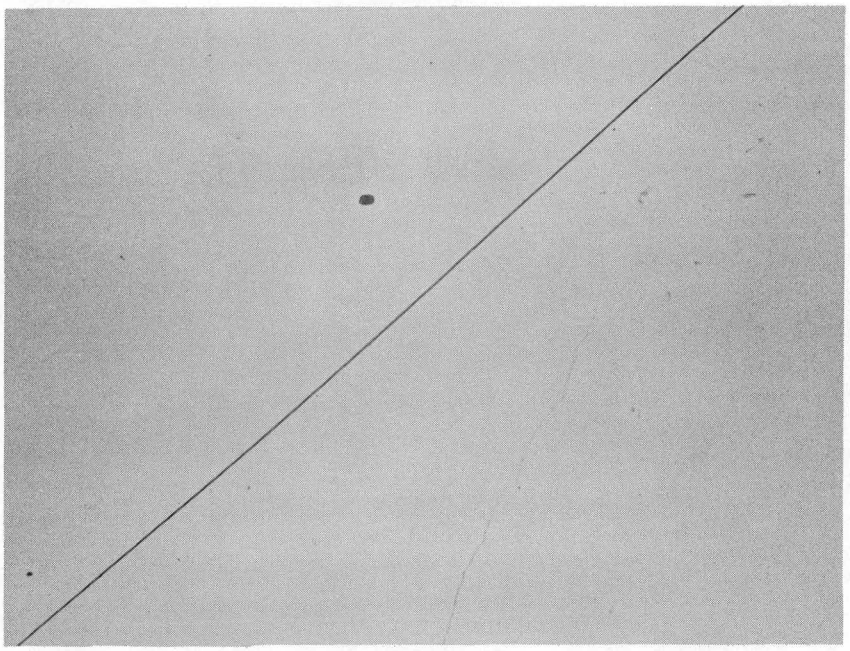

Figure 1C: Microstructure Of Ingot No. 5
Bottom-Edge Mag. 150X

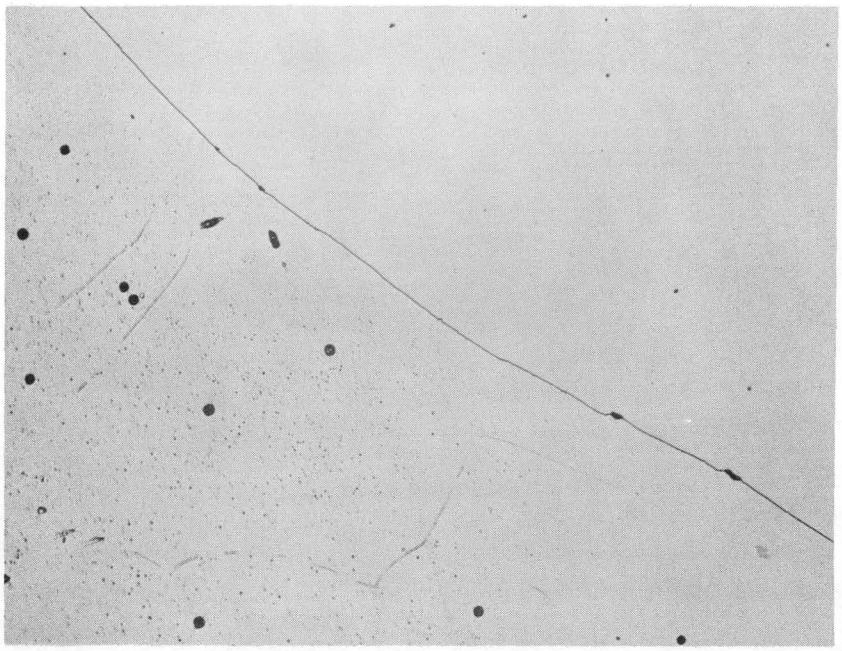

Figure 2A: Microstructure Of Ingot No. 3 Second E. B. Melt
Top Mag. 150X

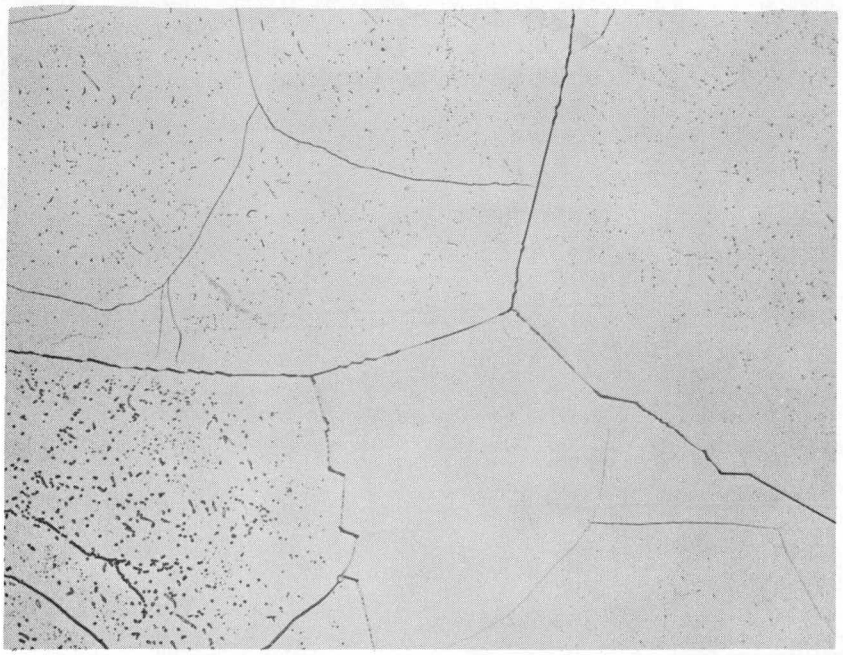

Figure 2B: Microstructure Of Ingot No. 2 Fifth E. B. Melt
Top Mag. 150X

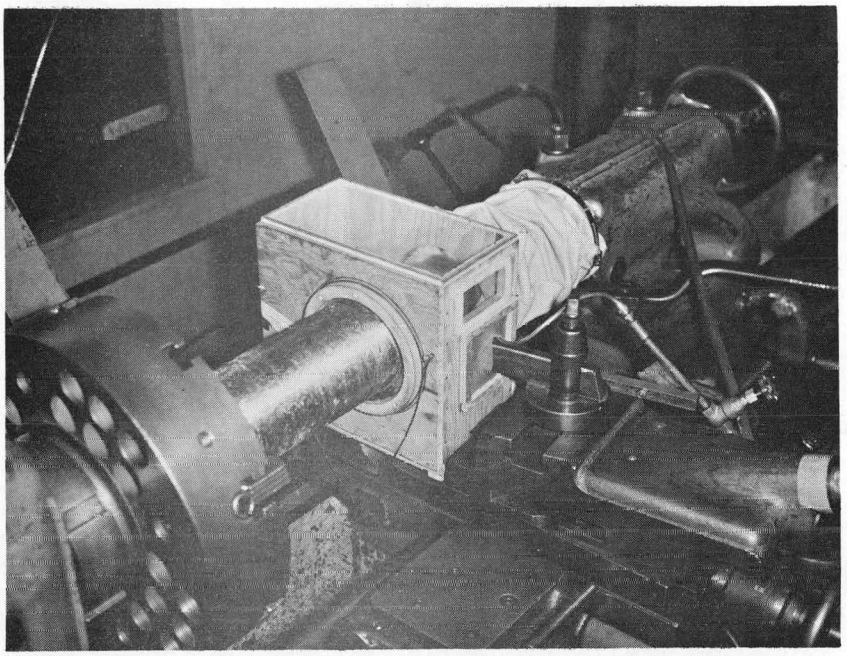

Figure 3: Inert Gas Hood For Machining Ingot No. 5 To Minimize Oxygen And Nitrogen Contamination

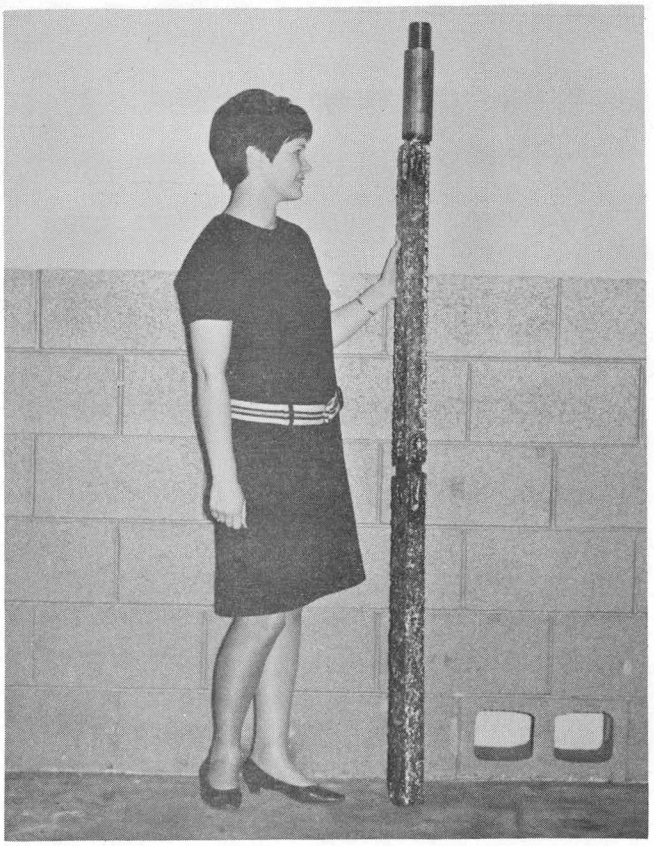

Figure 4: First Melt Electrode Consisting Of Two Compacts

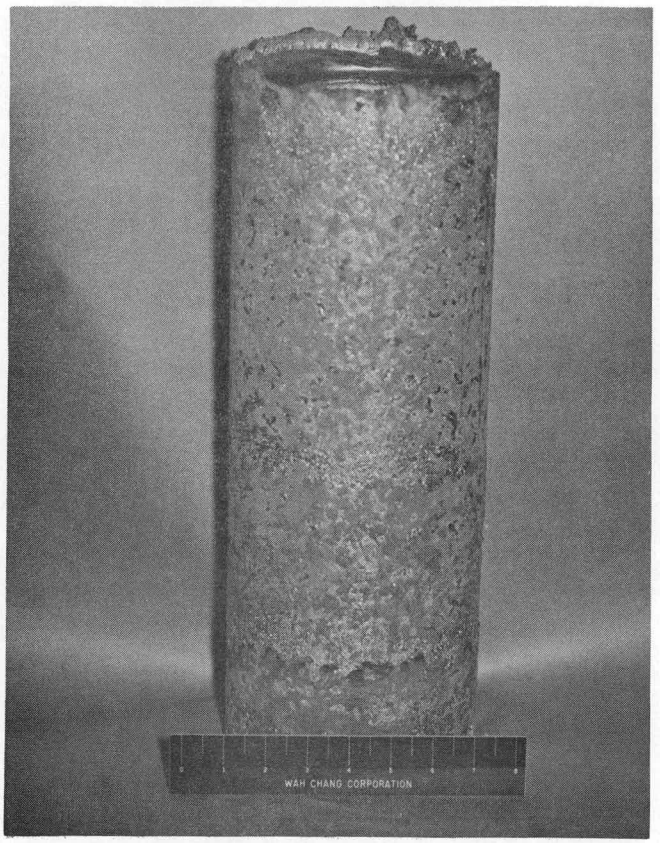

Figure 5: Double Arc Melt V-15Cr-5Ti Ingot As-Cast Weight 112.0 lb

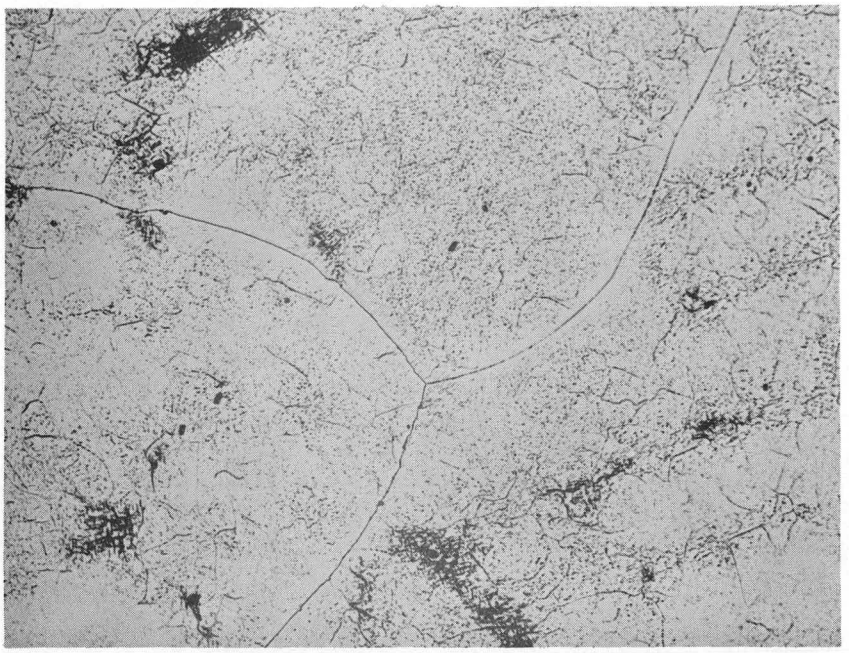

Figure 6A: As-Cast Microstructure Of Alloy Ingot
Center Mag. 150X

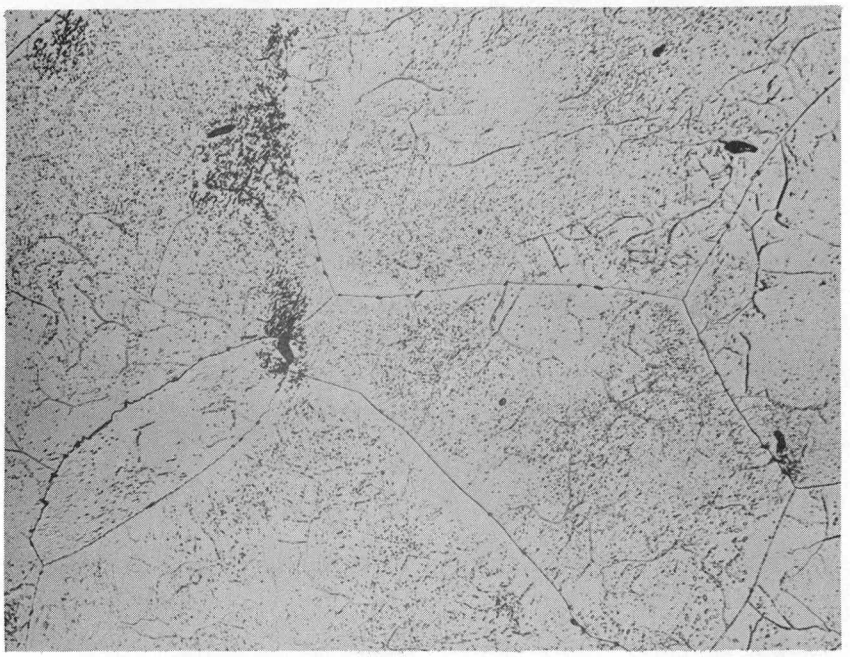

Figure 6B: As-Cast Microstructure Of Alloy Ingot
Mid-Radius Mag. 150X

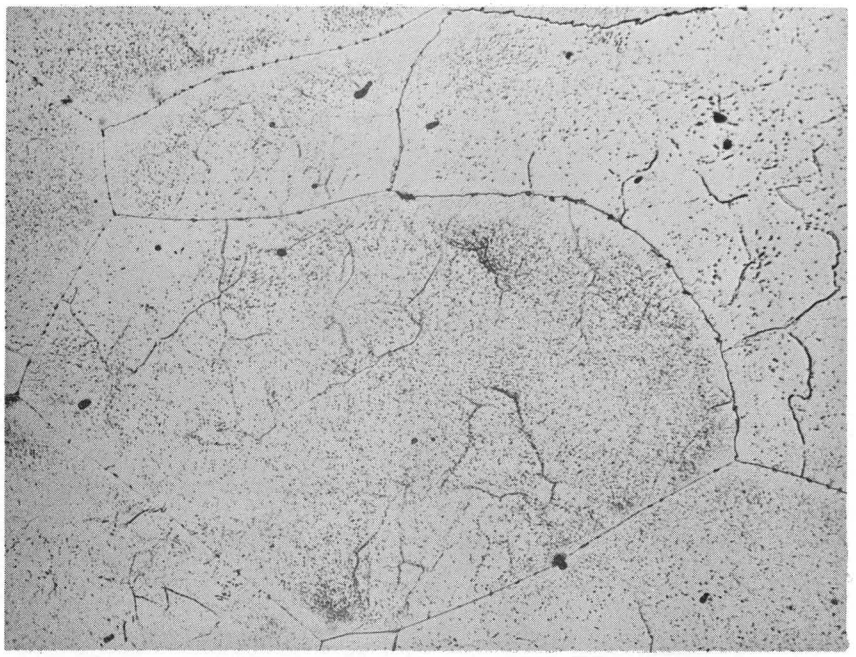

Figure 6C: As-Cast Microstructure Of Alloy Ingot Edge Mag. 150X

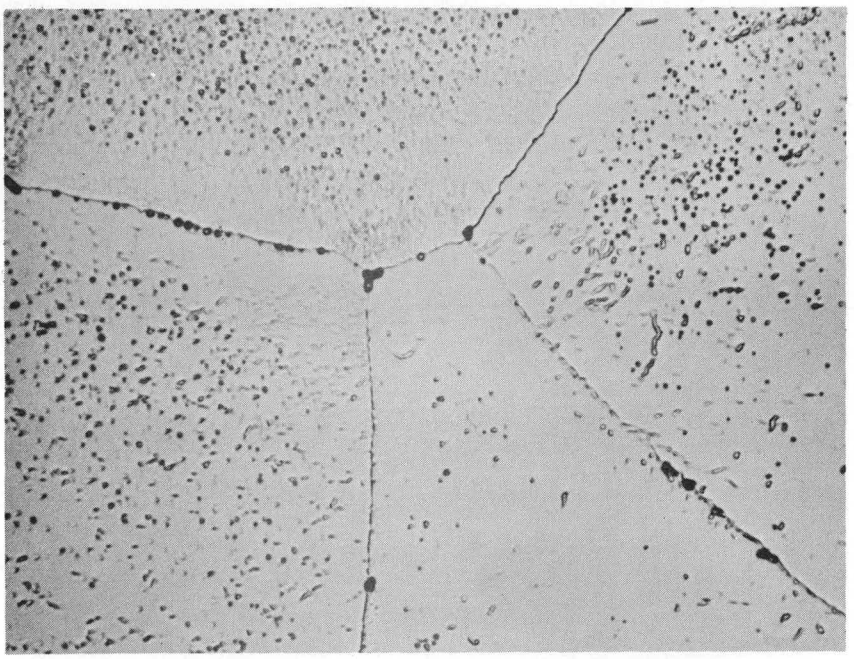

Figure 6D: As-Cast Microstructure Of Alloy Ingot Edge Mag. 750X

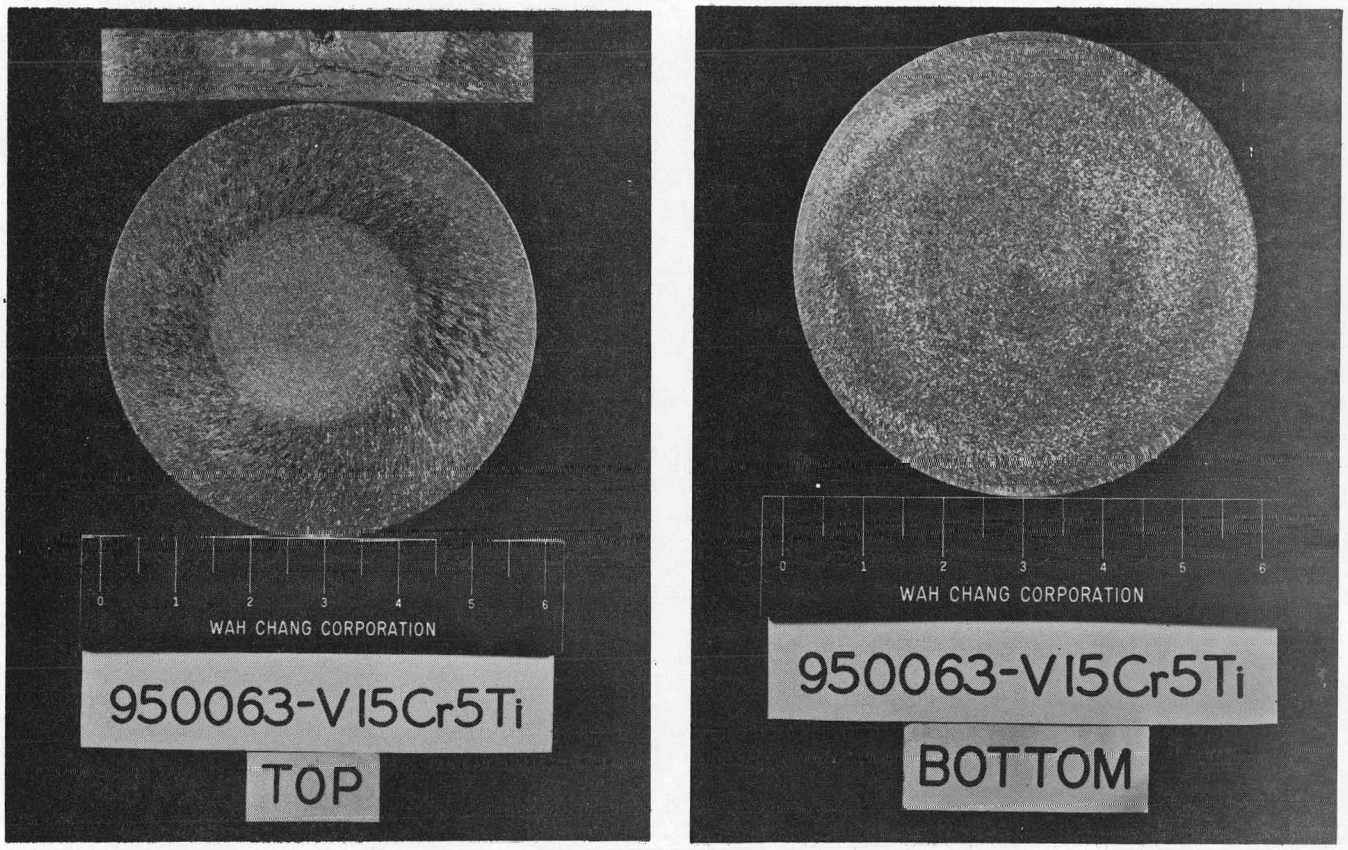

Figure 7: As-Cast Microstructure

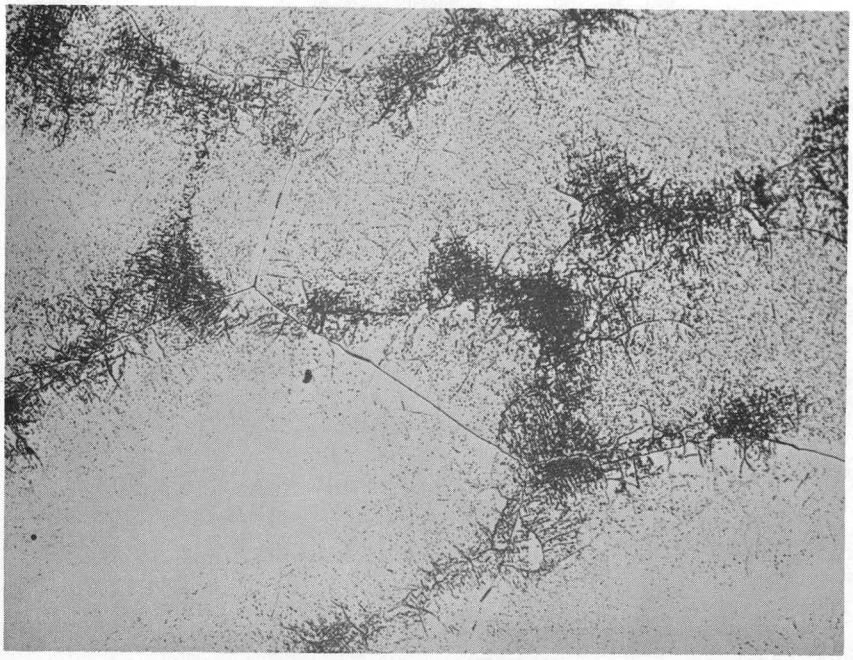

Figure 8A: Microstructure Of V-15Cr-5Ti After 2 Hours At 1100^{0}C
Mag. 150X

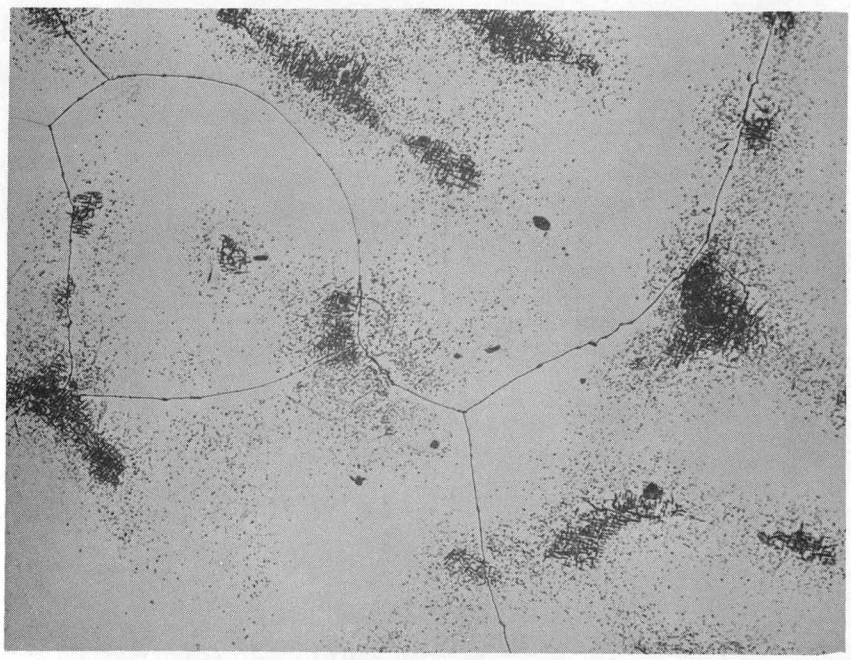

Figure 8B: Microstructure Of V-15Cr-5Ti After 2 Hours At 1200^{0}C
Mag. 150X

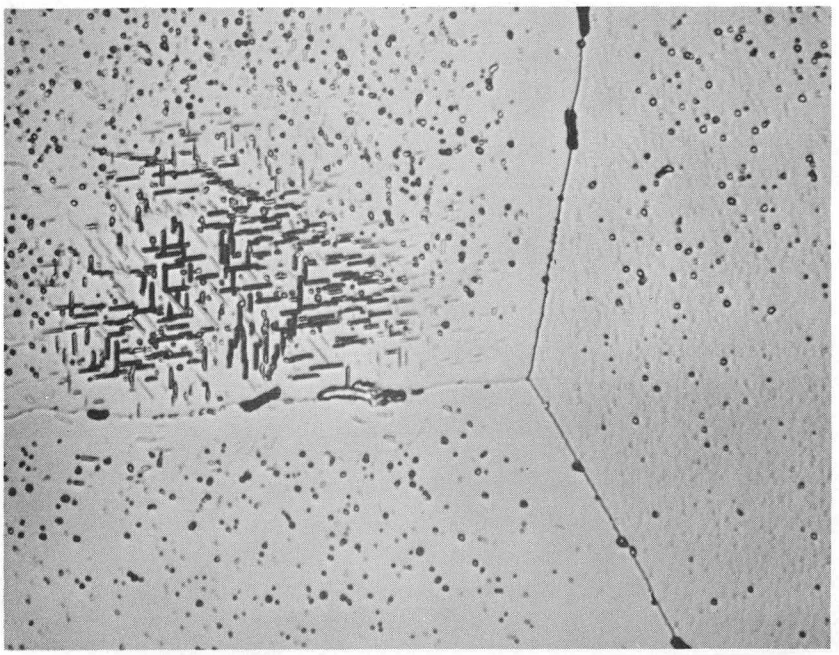

Figure 8C: Microstructure Of V-15Cr-5Ti After 2 Hours At 1200°C
Mag. 750X

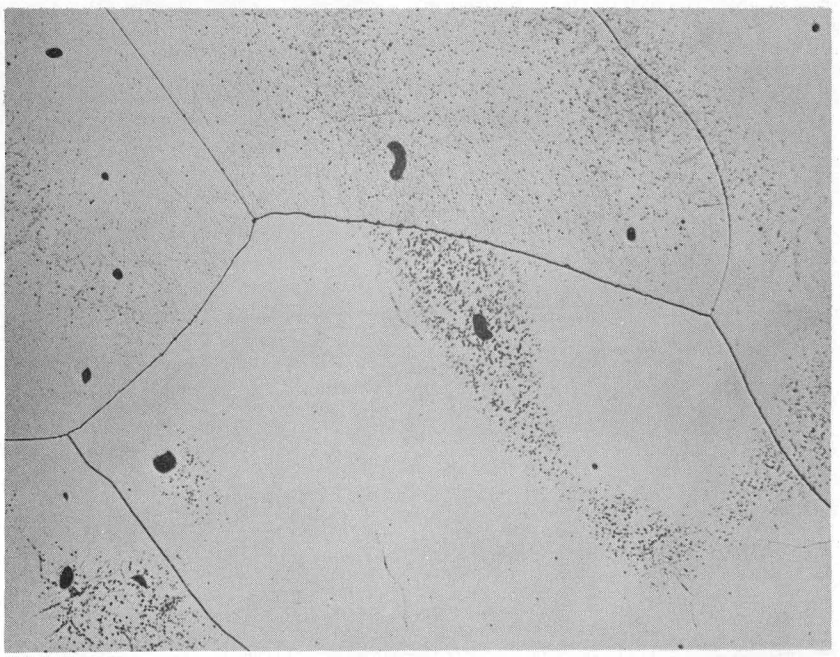

Figure 8D: Microstructure Of V-15Cr-5Ti After 6 Hours At 1200°C
Mag. 150X

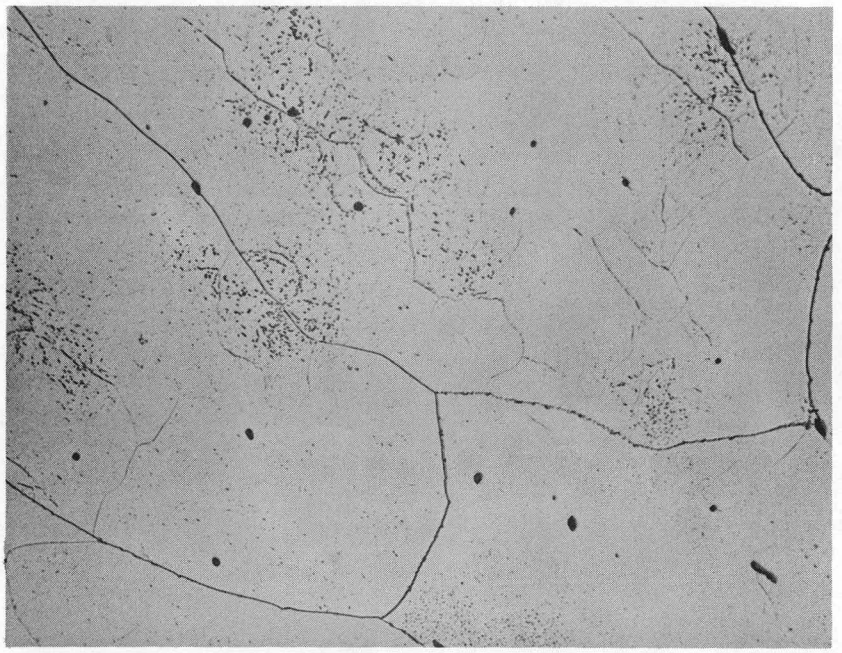

Figure 8E: Microstructure Of V-15Cr-5Ti After 2 Hours At 1300^{0}C
Mag. 150X

Figure 9: Effect Of Vacuum Melting On The Oxygen Content In Vanadium

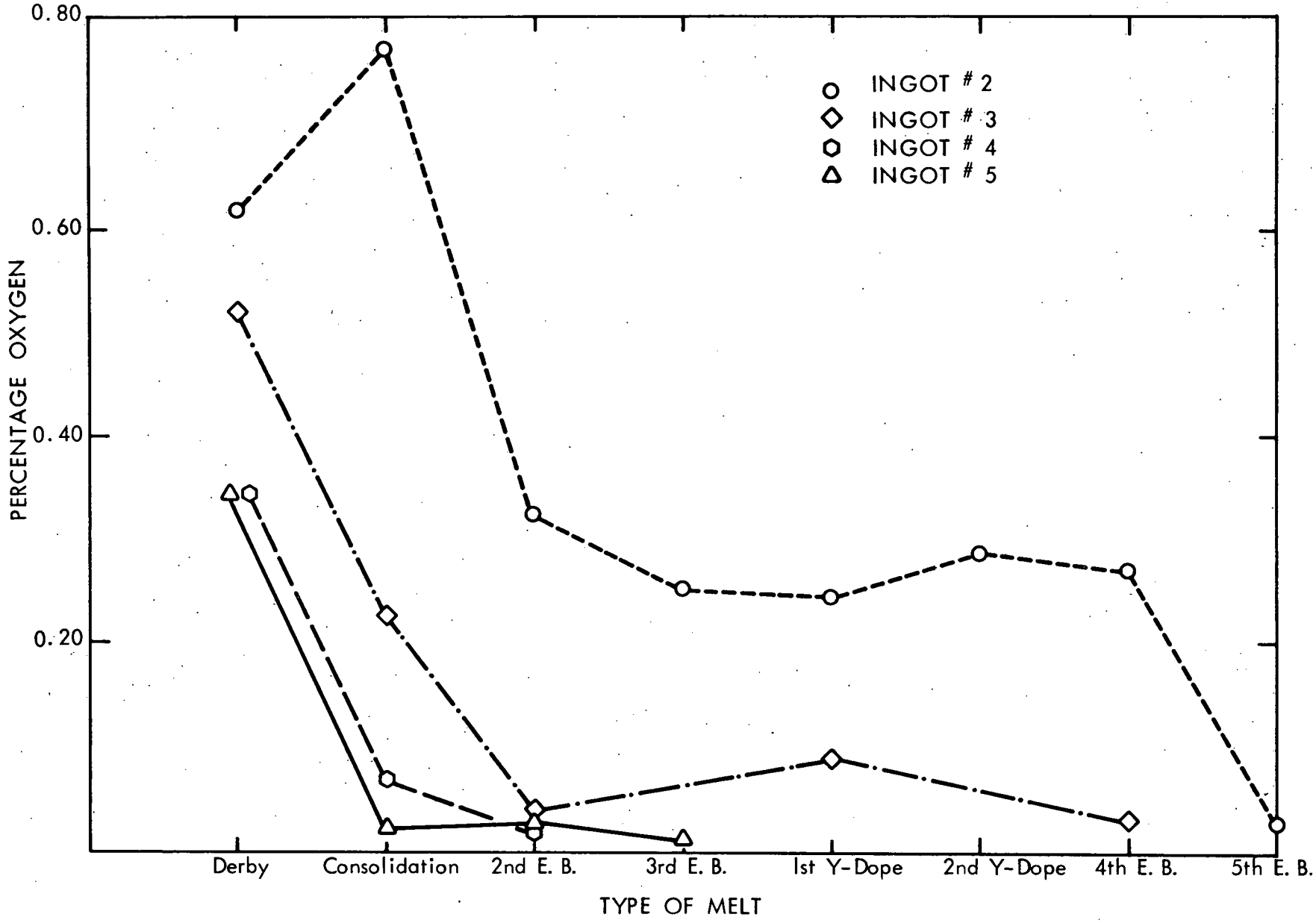

Figure 10: Effect Of Vacuum Melting On The Carbon Content In Vanadium

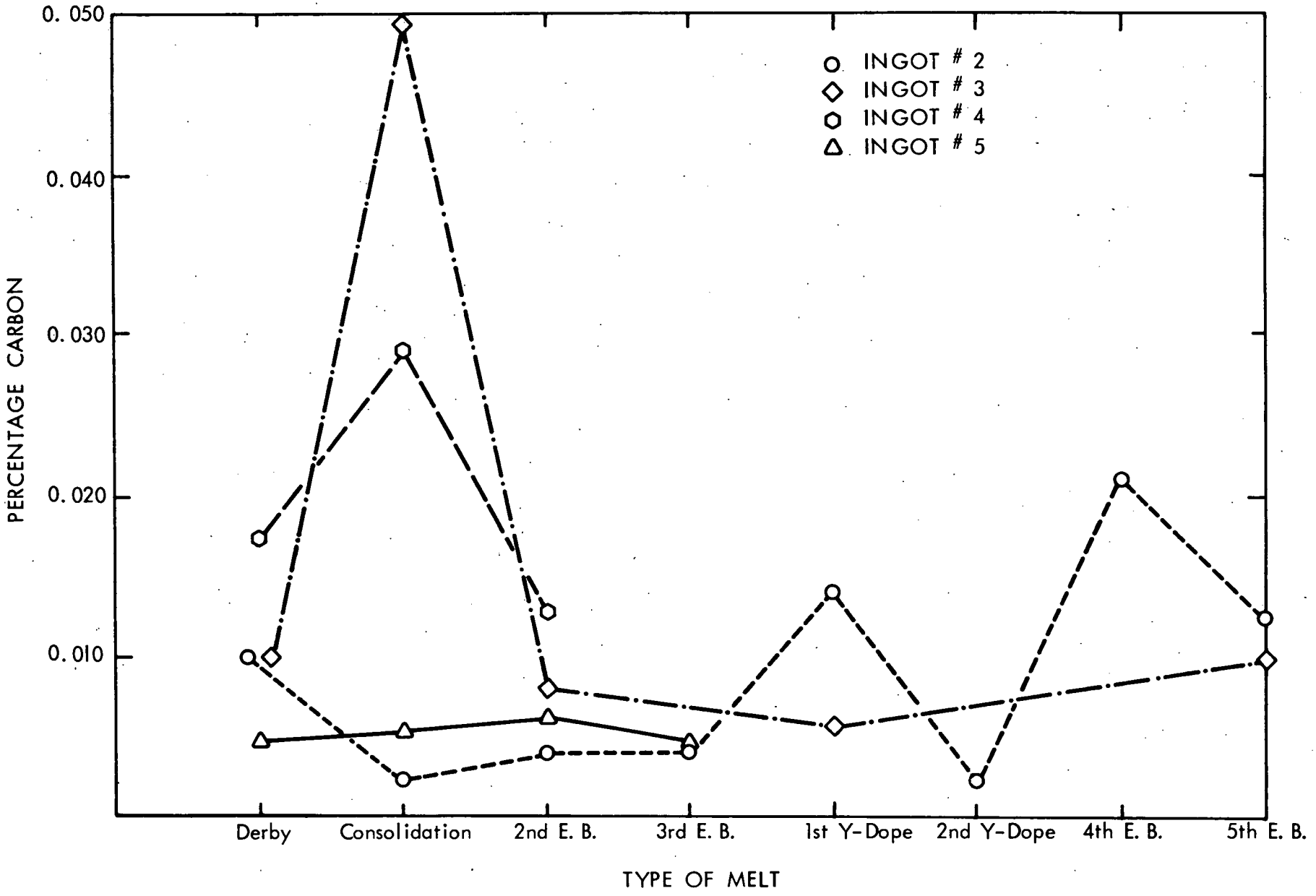

Figure 11: Effect Of Vacuum Melting On The Nitrogen Content In Vanadium

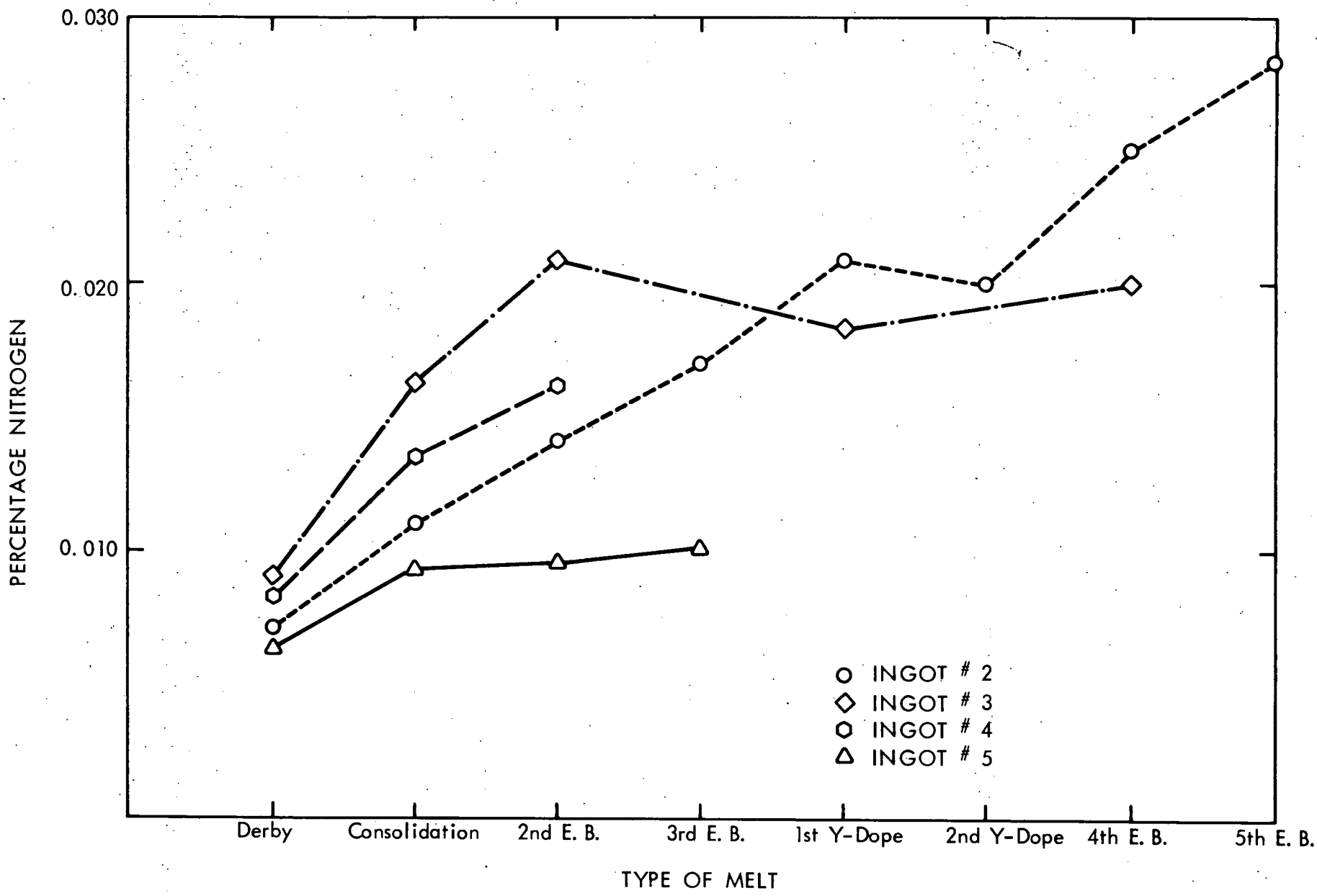

Figure 12: Effect Of Vacuum Melting On The Si Content In Vanadium

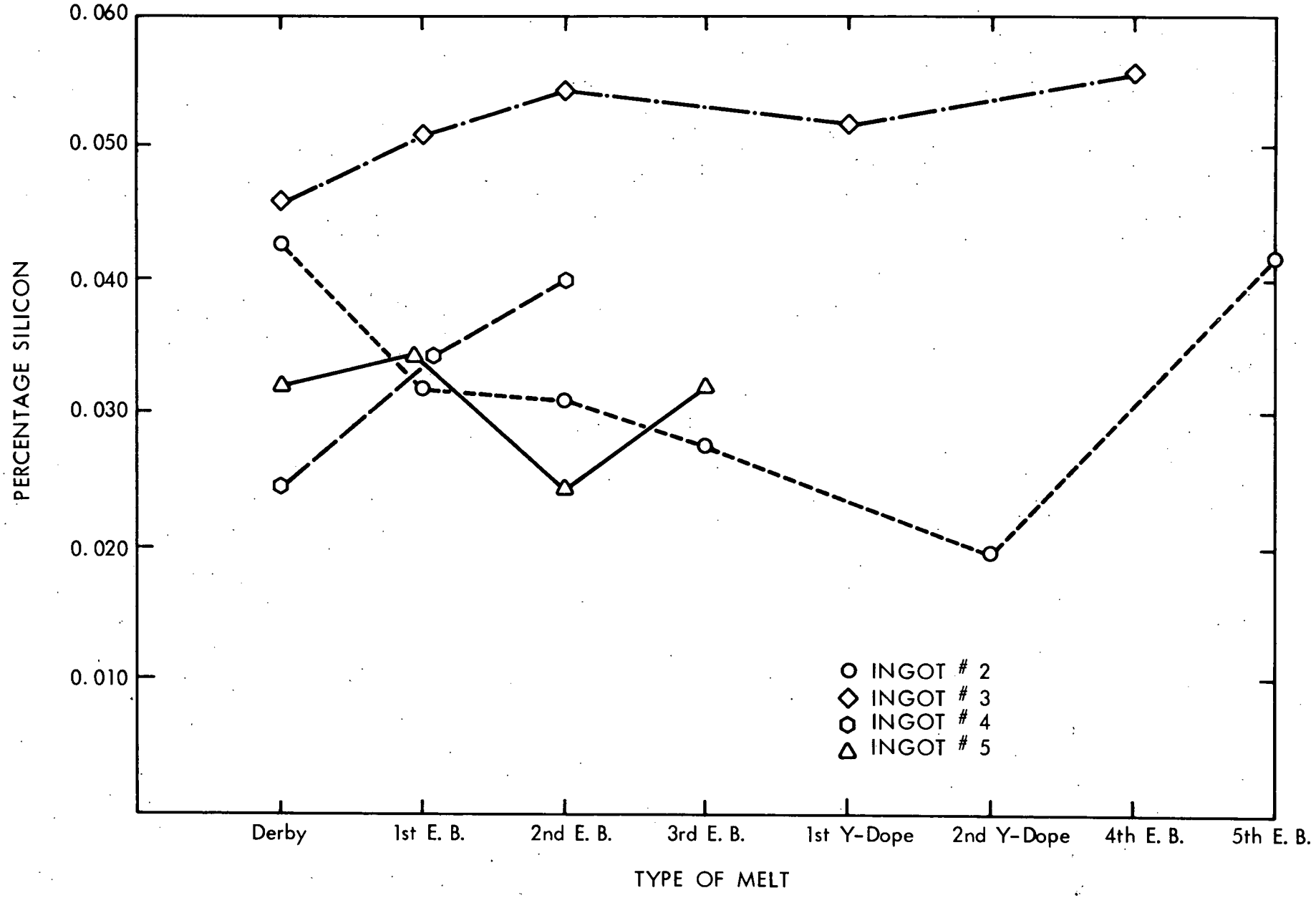

Figure 13: Effect Of Vacuum Melting On The Hardness Reading Of Vanadium

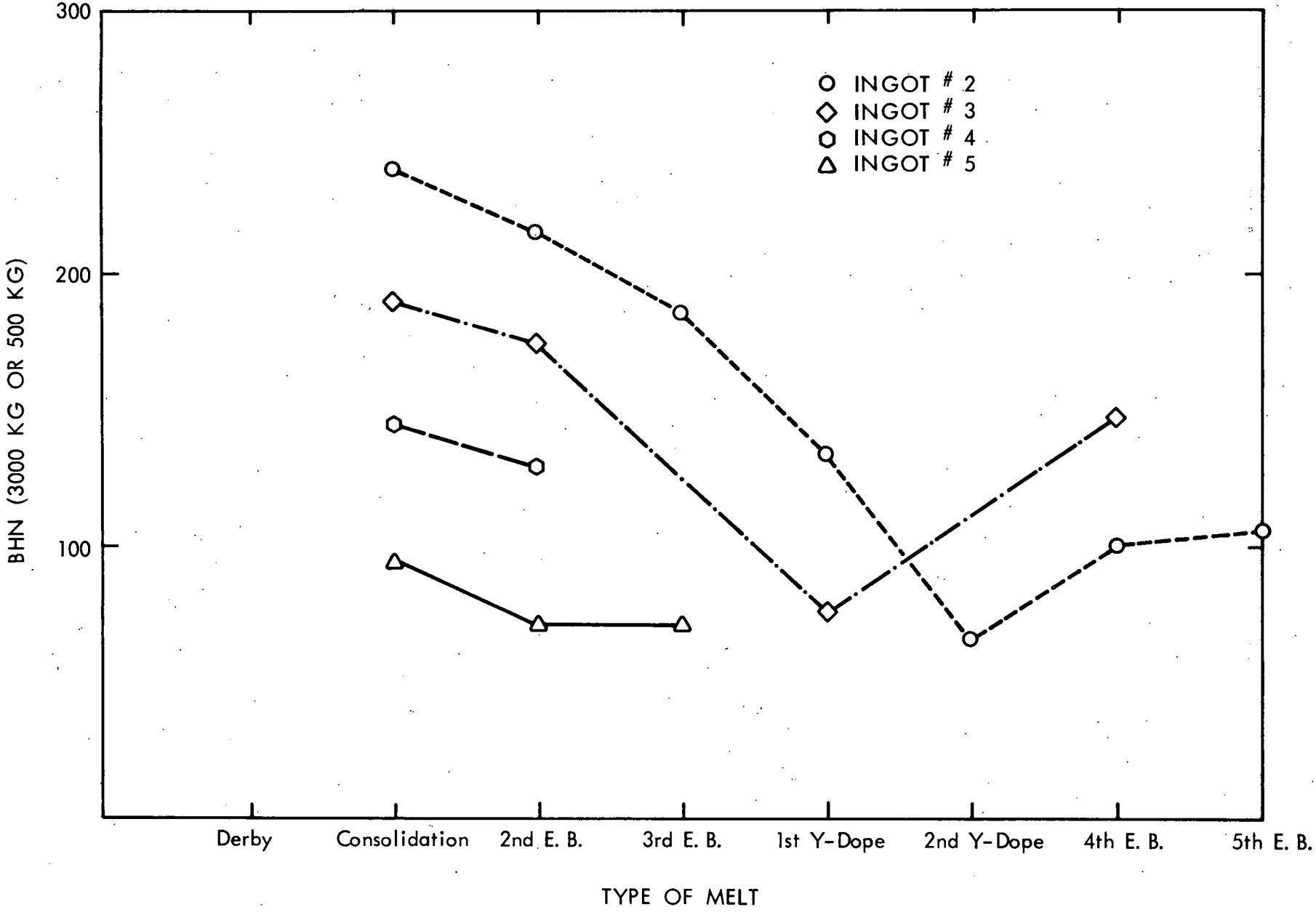

Figure 14: Plot Of Percent Of Yield Versus Melts