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Argonne National Laboratory

IMPROVEMENTS IN CONSOLIDATION AND FABRICATION OF VANADIUM-20 w/o TITANIUM (TV-20)

by

W. R. Burt, Jr., W. C. Kramer,
F. J. Karasek, and R. M. Mayfield

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May 1966

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ABSTRACT

Improvements in consolidation and fabrication techniques for V-20w/oTi are presented. Major emphasis is placed on tubing fabrication with significant progress having been made in tube-blank extrusion, tube reducing, and drawing. A newly designed and built three-roll tube-reducing machine shows much promise. Use of a plastic base-coat in conjunction with the drawing lubricant has allowed carbide draw dies to replace aluminum-bronze dies, with subsequent improvement in dimensional control. Commercial efforts in drawing V-20w/oTi tube-blanks furnished by ANL have been highly successful.

I. INTRODUCTION

Since the advent of the initial report on the consolidation and fabrication of vanadium-20 w/o titanium (TV-20),¹ considerable progress has been made in improving the fabrication operations and increasing the yield of finished products. Major emphasis was placed on tube fabrication. This report covers improvements in processing, yields obtained in various operations, and the results of commercial tube-drawing efforts. For the most part, operations unchanged from the previous report are mentioned only briefly.

Significant improvements were incorporated in the charge briquetting, tube-blank extrusion, and drawing operations; deleterious or time-consuming operations such as swaging and annealing were greatly reduced. Additional commercial efforts at tube-drawing were evaluated; resultant tubing was found to be of high quality.

II. IMPROVEMENTS IN CONSOLIDATION OF V-20w/oTi INGOTS

Production of full-scale V-20w/oTi (TV-20) ingots at ANL involves compaction of vanadium chips and titanium sponge into drip rods, which were electron-beam (EB) melted into 5.8-cm-diameter ingots. The EB ingots are consumable-electrode arc (AM) melted into 9.7-cm-diameter finished ingots. A detailed description of this procedure as it was originally developed was reported.¹ At the time, three arc-cast ingots (AM21, AM25, and AM28) had been produced at ANL by this procedure. Since then, three additional V-20w/oTi ingots (AM37, AM41, and AM53) have been produced. The same basic procedures were used, but improvements incorporated in some of the steps resulted in faster processing, higher yields, and ingots of better quality.

An auxiliary study was made to determine the effect of iron content on fabricability of the V-20w/oTi alloy. The results of this study are given in Appendix A, and it is concluded that iron contents ≤ 1000 ppm are not detrimental. The current specification--for vanadium chip given in Appendix B--requires an iron content of ≤ 500 ppm, which is well below the acceptable level.

A. Preparation of Drip-rods and Consumable Electrodes

Drip-rods utilized in producing ingots AM37 and AM41 were made in the same manner as for the first three ingots. Briquets of vanadium chips and titanium sponge were cold pressed in a one-piece cylindrical die to 3.2 cm in diameter and 2.5 cm long. A total of 180 briquets were tack-welded together to form six drip-rods which were electron-beam melted into two 5.8-cm-diameter x 56-cm-long ingots. These two ingots, in turn, were welded together and consumable-electrode arc melted to produce a single finished ingot, 9.7 cm in diameter and approximately 30 cm long.

The sixth ingot (AM53) was made from drip-rods formed from 1.9-cm-diameter x 2.5-cm briquets cold pressed in a three-piece split die (see Fig. 1). A total of 393 briquets were required, as opposed to 180 for the briquet of larger diameter. The split die overcame the problem of galling and die buildup experienced with the one-piece die when pushing out the briquets. Elimination of the problem of scraping accumulated or galled material off the die greatly reduced compacting time even though the number of briquets was increased over twofold.*

*Although not used on this work, a 2.5 x 2.5 x 7.6-cm single-piece compaction die has been constructed and used in other alloy-development work. Galling was eliminated by using a small amount of stearic acid as a lubricant on the die walls prior to compaction. The compacts had good green strength, and the total compaction cycle required a shorter time due to the fewer number of required briquets.

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Fig. 1. Photograph of the Three-piece Split Die Used for Compacting V-20w/oTi Briquets

Even at a melt rate of 100 g/min, however, the metal loss was less than at a melt rate of 33 g/min. The overall yield of more than 96% at melt rates of 50 to 100 g/min was considered a practical maximum and confirmed the earlier reported results wherein a 60-g/min rate was considered acceptable.

Because of the reduced diameter of these briquets, three rods of tack-welded briquets were welded together in a bundle to form the drip-rod. As before, two EB melted ingots were produced and remelted in the arc furnace to produce the final ingot.

B. Electron-beam Melting

In the course of EB melting drip-rods for the last three ingots, the melt rate was varied to determine its effect on metal loss. While the beam power was held constant at 29 to 30 kW, melt rates were increased by improving the beam focus and position of the drip-rod in relation to the beam. The results are given in Table I.

A significant improvement, due to reduced vaporization losses, was realized by increasing the melt rate from 33 to 51 g/min as the weight loss dropped from 5.6 to 2.4%. Increasing the melt rate above about 50 g/min again resulted in an increasing metal loss, due to increased splattering of molten metal from the pool.

TABLE I. Charge-weight Losses as a Function of Melting Rate in Electron-beam Melting V-20w/oTi Drip-rods

Ingot Number	Melt Rate, g/min	Charge Weight Lost in Melting, %
EB35	33	5.6
EB36	51	2.4
EB39	73	2.6
EB40	80	3.3
EB51	88	3.7
EB52	100	3.9

Since the EB ingots were to be remelted, surface quality was not considered critical, but surface characteristics were found to be dependent upon the position of the molten pool in the mold. Other factors being constant, a pool lying too low in the mold results in excessive cold shuts, whereas a pool that is too high and lies in the water-cooled approach radius results in surface tearing as the ingot is retracted.

Internal porosity in the EB ingots still existed regardless of variations in melting parameters. As with surface quality, this caused little concern as the ingots were to be remelted into the final ingot form.

C. Consumable-arc Remelting

Parameters used in consumable-arc remelting of the EB ingots were essentially unchanged from those that had been established originally. A melting rate of 1 kg/min was achieved with straight polarity (consumable electrode negative) at 84 kW (2700 A and 31 V). A stirring coil was used to improve ingot quality, and homogeneity and ingots were "hot-topped" to reduce secondary pipe. Although arc melting was done under vacuums of 10^{-2} to 10^{-3} Torr, no loss of charge material was experienced.

D. Ingot Chemistry

Analyses of the interstitial element (C, O₂, N₂, and H₂) of starting materials and final ingots are given in Table II. The analysis (592 ppm) for

TABLE II. Analyses of the Interstitial Element of Starting Materials and Arc-cast V-20w/oTi Ingots

		Interstitial Elements, ppm				
		Carbon	Oxygen	Nitrogen	Hydrogen	Total
Starting Materials	Titanium	33	150	20	7	210
	Vanadium	317	480	592*	2	1391
Ingot	AM37	226	420	320	20	986
Starting Materials	Titanium	33	130	20	7	210
	Vanadium	311	488	346	7	1152
Ingot	AM41	305	580	243	5	1133
Starting Materials	Titanium	33	150	20	7	210
	Vanadium	300	740	146	17	1203
Ingot	AM53	253	830	300	3	1386

*Value suspected of being too high.

nitrogen in the vanadium used in producing ingot AM37 is suspected of being too high for two reasons. First, previous lots of vanadium received from the same supplier were found to have nitrogen contents ranging from 200 to 400 ppm. Secondly, previous melting trials with vanadium and V-20w/oTi had never shown a substantial (150 ppm or more) reduction in nitrogen content, whereas the reduction in this case was approximately 270 ppm. With this one exception, results were as expected from prior work.

Of interest is the higher oxygen content of vanadium chips recently procured and used in producing AM53. It is expected that all vanadium chips purchased in the near future will have similarly high oxygen levels (~800 ppm) since commercial suppliers are not able to guarantee consistently lower levels by present production techniques, although some work is under way by commercial firms to develop means for producing vanadium of high purity.*

Results of analyses for titanium in the arc-cast ingots are given in Table III. The drip-rods used in producing AM37 were prepared with 21 w/o titanium to compensate for preferential loss of titanium by vaporization during melting. Analytical results for AM37 indicated that the titanium loss was negligible. Accordingly, drip rods for AM41 and AM53 were prepared with 20 w/o titanium. The variations in titanium content of the ingots were minor and well within typical specification limits of $\pm 1.0\%$.

TABLE III. Analytical Results for Titanium in
Arc-cast V-20w/oTi Ingots

Sample Location in Ingot	Titanium Analyses, w/o		
	Ingot AM37 ^(a)	Ingot AM41 ^(b)	Ingot AM53 ^(b)
Top	20.9	20.7	19.9
Middle	21.0	19.9	19.7
Bottom	20.6	20.2	20.0
Average	20.8	20.3	19.9

(a) Drip-rods prepared with 21 w/o titanium.

(b) Drip-rods prepared with 20 w/o titanium.

*The use of higher-purity (more expensive) starting materials will have to be judged in terms of the properties of the end product. It is entirely possible--although at this time unverified--that higher-purity vanadium would produce a weaker alloy entirely unsuited for its intended purpose.

Because of their potential effect on reactor physics and irradiation behavior, boron and uranium analyses were requested. Although the material selected for analysis was produced commercially, the techniques used to produce the alloy were similar to those employed at ANL, i.e., compaction of vanadium chips and titanium sponge, EB melting, and consumable-electrode arc melting. Wet chemical analyses indicated 5.0 ppm boron and 8.5 ppm uranium.

E. Process Yields (Consolidation)

Process yields based on charge material, arc-cast ingot, and machined billet weights are shown in Table IV.

TABLE IV. Process Yields in Consolidation of V-20w/oTi

Ingot Number	Material Weight, kg			Process Yields, %		
	Charge	Arc-cast Ingot	Machined Billet	Arc-cast Ingot to Charge	Machined Billet to Ingot	Machined Billet to Charge
AM21	17.10	15.33	10.90	89.7	71.1	63.8
25	17.84	15.85	10.90	88.8	68.7	61.1
28	15.40	13.28	9.90	86.2	74.5	64.2
37	16.06	-	9.55	-	-	59.5
41	16.50	14.20	-	86.0	-	-
53	16.50	14.43	10.10	87.4	69.0	61.2
Average	16.57	14.62	10.27	87.7	71.0	61.9

Losses in melting are approximately 14% of the charge weight, and result from splatter and vaporization during EB melting (about one-third of total loss), unmelted portions of drip-rods and consumable-electrode stub, and machining operations to remove starting plugs and square EB-ingot ends for welding. In machining the arc-cast ingot into a billet suitable for extrusion, approximately 30% of the ingot weight is removed, with the overall yield of machined billet being roughly 60% of the starting charge weight. Losses in machining the arc-cast ingot result from removing the starting plug, cropping the secondary pipe from the top of the ingot, and machining the ingot diameter from 9.7 to 9.0 cm (3.53 in.) to remove surface defects and enable the billet to fit the stainless steel can used for ingot breakdown extrusion. This reduction in diameter removes about 15% of the ingot weight. The overall weight loss was 40% (approximately a 30% loss based on ingot weight). Much of the loss is represented by solid scrap that is suitable for direct remelting.

F. Reclamation of Scrap Material

Solid scrap pieces of TV-20 generated in various processing operations were reclaimed by EB melting. The scrap consisted of sections of extruded and rolled rod together with quartered sections of cropped ingot tops. Drip-rods were prepared by tack welding these sections together. A melt rate of about 53 g/min was maintained in casting two 5.8-cm-diameter ingots (EB42 and EB43). Chemical analyses on EB42 showed a total interstitial level of 1491 ppm, with individual elements being carbon--430 ppm, oxygen--430 ppm, nitrogen--620 ppm, and hydrogen--11 ppm. Considering the amount of processing undergone by the material comprising the melt stock, the degree of interstitial contamination is not excessive. Roughly, the carbon content increased by about 100 ppm and the nitrogen content by about 300 ppm. Reclamation experience and technology may lower these values considerably.

No attempts have been made as yet to recover machining chips. In all likelihood, the chips will have to be pickled to remove surface contamination picked up during machining. The chips would be compacted and tack welded into drip-rods for melting. Due to the cost of starting material--vanadium chips at approximately \$14/kg--reclamation of scrap material is a necessity if final product costs are to be minimized.

III. INGOT BREAKDOWN EXTRUSION

The parameters for the breakdown extrusion of arc-cast ingots AM37, AM41, and AM53 were essentially the same as used for earlier ingots.¹ The 9.0-cm machined ingots were canned in 304 SS to provide an overall billet diameter of 10.2 cm. The billets were heated to 1100°C and extruded at a 3.5:1 reduction ratio to yield a bar of nominal 5.6-cm diameter. The extruded bars were machined into TV-20 billets for re-extrusion into rod, sheet-bar, and tube-blanks.

A. Extrusion Parameters

Data on extrusion of the three arc-cast ingots (AM37, AM41, and AM53) are given in Table V along with data on earlier ingot breakdown extrusions. The average extrusion constant (excluding the one billet extruded at 1000°C) is approximately 6950 kg/cm².

B. Process Yields (Primary Extrusion)

The yield of machined billets for re-extrusion and solid bar for rolling is shown in Table VI. Billets were re-extruded into rod, sheet-bar, and tube-blanks, with two or more types of billets being realized from each

TABLE V. Summary of Extrusion Data for V-20w/oTi
Arc-cast Ingots^(a)

Extrusion Number	Arc-cast Ingot Number ^(b)	Billet Temp, °C	Reduction Ratio	Extrusion Pressure		Extrusion ^(c) Constant, kg/cm ²
				Breakthrough kg/cm ²	Running kg/cm ²	
261	AM21	1000	3.6	9,490	8,650	6,820
269	AM25	1100	3.6	8,450	7,730	6,050
289	UC-761-3	1100	3.6	NM ^(d)	NM ^(d)	NM ^(d)
290	UC-761-1	1100	6.5	11,250	9,630	5,130
303	AM28	1100	3.5	8,930	8,160	6,470
325	UC-761-2	1100	3.8	11,600	11,180	8,450
410	AM37	1100	3.5	10,820	9,350	7,450
431	AM41	1100	3.5	NM ^(d)	NM ^(d)	NM ^(d)
478	AM53	1100	3.3	8,990	7,870	6,540

(a) Ingots were canned in Type 304 stainless steel for extrusion.

(b) AM indicates ingots produced at ANL; UC indicates ingots procured from Union Carbide Co.

(c) Calculated from $P = K \ln (A_o/A)$, where P is the running extrusion pressure, K is the extrusion constant, and A_o/A is the reduction ratio.

(d) Not measured.

TABLE VI. Yield of V-20w/oTi Billets for Re-extrusion and
Rolling Based on Starting Billet Weight

Extrusion Number	Arc-cast Ingot Number	Component Weights, kg			Yield of Starting Primary Billet Weight, %	
		Primary Extrusion Billets	Actual ^(a) Re-extrusion Billets	Corrected ^(b) Re-extrusion Billets	Actual ^(a) Billets	Corrected ^(b) Billets
261	AM21	10.90	6.72	7.26	61.5	66.5
269	AM25	10.90	7.64	8.37	70.0	76.5
289	UC-761-3	8.55	4.90	5.65	57.5	66.0
290	UC-761-1	7.35	-	-	-	-
303	AM28	9.90	5.80	6.55	58.5	66.0
325	UC-761-2	8.85	-	-	-	-
410	AM37	9.55	5.64	7.19	59.0	75.5
431	AM41	-	6.14	7.87	-	-
478	AM53	10.10	6.69	6.97	66.0	69.0
Average	-	9.51	6.22	7.12	66.5	75.0

(a) Includes weight of material used for rolling and for re-extrusion.

(b) Weight of hollow billets for tube-blank extrusions recalculated as solid billets to correct for varying numbers of hollow billets machined from the different extrusions.

breakdown extrusion. A corrected weight is shown for re-extrusion billets to indicate the amount of solid billets that could be achieved from each extrusion. This correction was made to compensate for the varying number of hollow billets machined from the different bars.

The actual yield of 66.5% could be increased by extruding the ingot to a bar of smaller diameter. However, sufficient stock must be left to insure removal of the longitudinal striations. Additional recovery could be effected by eliminating machining losses due to the tapered billet nose, as is now the current practice. As seen by the corrected billet yield of 75.0% (assumes solid billets for re-extrusion), roughly 10% is lost in drilling the holes for tube-blank extrusion billets.

IV. RE-EXTRUSION

A. Rod Stock and Sheet-bar

No significant changes were made in the techniques used for extruding rod stock or sheet-bar from that described in ANL-6928.¹

The Type 304 SS jackets were mechanically stripped from extruded products, with the re-extruded TV-20 surface quality being good. No conditioning was necessary prior to secondary fabrication. The protective steel jacket used in re-extrusion precluded any surface conditioning for contamination removal.

No specific data were kept for rod or sheet-bar. Approximately 95% of the alloy was recovered. The loss mainly was due to cropping the tail-end extrusion defect.

B. Tube-blanks

Significant progress was made in the extrusion of V-20Ti tube-blanks in regard to mandrel performance, internal surface quality, and recovery of material. The normal practice for tube-blank extrusion consists of extruding the billet at 1100 to 1150°C with reduction ratios of approximately 8:1. After extrusion, the tube-blank was heated in a lead bath (at ~500°C) and straightened on a two-roll Medart straightener. The Type 304 SS jacket was removed from the outer surface by "peeling" or mechanical stripping. The steel liner on the inner surface was removed by pickling in hot concentrated (30 to 40%) HCl. After dejacketing, the V-20Ti tube-blank measured approximately 2.16 cm in OD x 1.61 cm in ID.

1. Mandrel Performance

Initially, billets were extruded over 1.27-cm-diameter H-12 hot-work tool-steel mandrels hardened to approximately 47-50 R_C . In the majority of cases, the mandrel failed when billets were extruded at 1100 to 1150°C and reduction ratios of 9 to 13:1. The failure was tensile in nature and always was accompanied by a large degree of necking. The failure occurred at the tail end of the billet, either late in the extrusion (which allowed some recovery of extruded tube-blank) or at the onset of deformation of the V-20Ti alloy. Neither glass coating the mandrel to reduce heat buildup (and improve lubricity, as mentioned later) nor increasing the mandrel size alleviated the problem of mandrel failure. Subsequently, a "Clarite" (T-1) high-speed tool-steel mandrel hardened to 58-62 R_C was used and tube-blanks extruded successfully. No mandrel deformation (necking) was observed, and hardness measurements after extrusion showed no evidence of softening due to overheating. Since that time, a total of twenty tube-blank extrusions have been made over "Clarite" mandrels, and only in one case did the mandrel fail, at the end of extrusion, with the break being typical of a brittle fracture with no evidence of necking. Since the mandrel had been used a number of times, this fracture most likely was associated with a scratch or pit remaining from prior extrusions.

It is apparent from the above results that increasing the mandrel hardness and thereby the strength (as a rough guide, the room-temperature tensile strength would increase from 16,900 kg/cm² to 20,700 kg/cm² in going from 50 R_C to 60 R_C) resulted in sufficient strength to carry the stress during extrusion. The H-12 hot-work tool-steel has a higher softening temperature than the T-1 high-speed tool steel, yet no evidence of softening was found for the T-1 mandrels after repeated use.

2. Internal Surface Quality

Initial tube-blank extrusions were processed with a MoS₂-graphite (95:5) mixture in a synthetic oil as the mandrel lubricant. This lubricant generally resulted in a rough internal surface with occasional evidence of inner surface tearing. To improve lubricity and surface quality, mandrels were precoated with a glass slurry and dried prior to extrusion. Corning 7052 borosilicate glass powder with a softening temperature* of 708°C was used. The -325 mesh powder was mixed with water and "Alginate" to form a suspension, which was painted on the polished mandrel. The coating was approximately 0.25 to 0.38 mm thick.

The adoption of this lubrication technique significantly improved the inner surface quality. Rarely, if ever, were there indications of tearing, and the overall roughness was reduced.

*The temperature at which glass will deform under its own weight.

3. Process Yields (Re-extrusion)

The above-described extrusion technique for tube-blanks resulted in an overall material recovery of greater than 92% as seen in Table VII. The major factor in the high recovery was the use of the "canned" billet technique which eliminated any need for conditioning the extruded product to remove contamination or surface roughness prior to secondary fabrication.

TABLE VII. Material Recovery in Extruding V-20w/oTi Tube-blanks

Extrusion Parameters			Extruded V-20Ti	Material Recovery		
Number	Temp, °C	Reduction Ratio	Tube-blank Length, cm	Billet Wt, g	Tube-blank Wt, g	Yield, %
363	~1150	8.5:1	87	830	796	95.9
364	1125	8.5	79	830	738	88.9
366	1150	8.3	69	706	638	90.4
373	1150	8.0	70	796	697	87.6
374	1150	8.0	74	833	701	84.2
375	1150	8.2	84	814	767	94.2
376	1150	8.2	81	813	755	92.9
386	1150	8.2	87	833	832	99.9
417	1150	8.5	102	849	805	94.8
418	1150	8.5	99	835	769	92.1
419	1150	8.5	99	831	794	95.5
421	1150	8.5	84	843	~800	~94.9
437	1150	8.4	89	868	864	99.5
438	1150	8.4	91	867	863	99.5
439	1150	8.4	89	857	847	98.8
440	1150	8.2	89	873	866	99.2
441	1150	8.1	56	860	563	65.0
442	1150	8.0	87	837	~830	~99.2
480	1150	8.1	122	1175	1004	85.4
Average = 92.4						

A floating-mandrel technique was used in the extrusion of tube-blanks since the extrusion press design did not incorporate a mandrel shift mechanism to maintain a controlled mandrel position with respect to the die. The floating mandrel is held by the dummy block and both advance with the ram during extrusion. At the end of extrusion, the mandrel extended past the die, and the frictional force of the tube-blank on the mandrel prevented the tube-blank from sliding off the mandrel when the billet had cleared the die. As a result, a sleeve (or portion of the extruded tube) often remained on the mandrel after extrusion. This sleeve contained the tail end of the

V-20Ti billet and resulted in nonrecoverable material. In these cases, yields ran from 84 to 96%. The use of a tapered mandrel resulted in a tapered tube wall and did not materially improve the situation. Later extrusions (see extrusions 437 to 440 and 442 in Table VII) incorporated a longer Type 304 SS end plug in the billet design so that the TV-20 portion of the tube-blank would clear the mandrel and only a stainless steel sleeve would remain on the mandrel.

Based on improved billet design, it should be possible to recover approximately 99% of the starting billet weight as an extruded tube-blank suitable for secondary fabrication.

V. SECONDARY FABRICATION OF TUBING

A. Swaging

The improved surface quality of as-extruded tube-blanks has decreased surface conditioning to a minimum. In fact, current practice requires no conditioning prior to swaging. The increased tube-blank OD of 2.16 cm with the same wall thickness as before of 0.28 cm resulted in a D/t (OD/wall thickness) ratio of 7.9:1, which was significantly higher than the 6.5:1 ratio originally realized. This increased ratio minimized the amount of swaging necessary to prepare the tube-blank for ductile-core drawing. Basically, a single swaging operation was utilized mainly for rounding the tube-blank to provide better concentricity and a uniform wall. Since heavy swaging operations require in-process anneals after approximately 25% reduction in area to avoid crack formation, the reduced swaging schedule reduced the number of necessary in-process anneals.

For swaging, a 1.519-cm-diameter hardened tool-steel mandrel lubricated with Aldraw J-1 was inserted in the bore of the tube-blank and the assembly swaged in one pass. This reduced the 2.16-cm-OD x 1.61-cm-ID tube-blank to 2.03 cm OD x 1.52 cm ID. Total area reduction was approximately 15%, with the wall reduction being about 8%. This reduction was sufficient to "true up" the tube-blank and was within the maximum swage reduction limits of 25% previously reported.¹

After swaging, the tube-blank was cleaned and vacuum annealed for one hour at 900°C prior to drawing.

B. Tube Reducing

In addition to commercial results on tube reducing V-20Ti, tube reduction on a three-roll tube reducer of unique design (see Fig. 2) has been evaluated at ANL. Basically, three rolls converge on the tube and iron the tube wall thickness over a hardened mandrel. Since the rolls are guided by

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Fig. 2. Schematic Drawing of Three-roll Tube Reducer

an inclined (1.6°) backup channel, they converge on the tube as the forward stroke progresses. After the return stroke, the tube is indexed 60° before the next pass. After three passes, the tube is advanced approximately 1.2 to 2.5 cm and the ironing passes repeated.

While the tube reducer is still in the development stage, it has been used successfully for tube reducing the V-20Ti alloy. In the first trial a 1.48-cm-diameter mandrel was used, with the annealed tube being reduced from the starting size 2.032-cm OD x 1.524-cm ID to 1.943-cm OD x 1.478-cm ID. The mandrel was removed and a new, 1.38-cm-diameter mandrel inserted in the tube. The tube then was reduced to a final size of 1.631-cm OD x 1.382-cm ID. In the two passes, the wall thickness was reduced from 0.254 to 0.124 cm while the tube length increased from 30.7 to 75.2 cm. The overall reduction in area was 58%; the wall thickness was reduced 51%. Both the external and internal surfaces were excellent, although the OD was not perfectly round owing to the shape of the rolls. The variation in the OD was roughly 0.013 cm and could easily be eliminated in a subsequent drawing operation. Other roll designs for improving OD roundness are being evaluated. Ultrasonic inspection of the tube did not reveal cracks or other discontinuities.

Attempts to reduce further a 1.943-cm-OD x 1.478-cm-ID tube--the size after the first tube-reducing pass--to a final size of 1.631-cm OD x 1.478-cm ID by using a 1.478-cm-diameter mandrel resulted in severe splitting of the resultant 0.076-cm-thick tube wall. If, after the first pass, the tube was vacuum annealed for one hour at 900°C , the second reduction to the 1.631-cm-OD x 1.478-cm-ID final size was easily accomplished. In this second reduction, the overall reduction in area was 70% while the wall thickness was reduced 67%.

While operating parameters for this tube-reducer have not been fully evaluated as yet, the machine shows much promise. Also, the V-20Ti alloy has been shown to be quite amenable to tube-reducing operations provided excessive reductions (>75 percent) are not attempted.

C. Drawing Methods

1. Ductile-core Drawing

Ductile cores of mild steel and copper have been successfully used in the initial stages of drawing. In addition to supporting the tube ID, the OD/wall thickness ratio can be changed during drawing by judicious selection of core materials. Cores that are softer than the tube will result in a decreased D/t ratio whereas harder cores will increase the ratio. It has been noted, however, that when longer lengths (about 2 m) of tubing are drawn with a ductile core, a large variation in wall thickness of as much as

0.025 to 0.051 cm existed between the lead and tail end of the tube. Although the precise reasons for this behavior are not fully understood as yet, it is believed to be a result of uneven deformation of the tube/core composite over long lengths compounded by such factors as interface lubrication, severity of reduction, and core material. The problem is under study to provide a better understanding of the ductile-core drawing technique. Correction of this condition was necessary at an intermediate stage by removing the ductile core (reeling or swaging) and inserting a hardened mandrel in its place. The tube then was either drawn or swaged over the mandrel to produce a uniform wall thickness.

2. Mandrel (Bar) Drawing

Commercial drawing of tubing involves the use of bar mandrels in many cases. This is a sink-iron technique. After each pass, the tube is reeled off the mandrel, a new mandrel (smaller in diameter) inserted, and another draw pass is made. This is repeated with successively smaller dies and mandrels until the final size is reached. After the tube has been reeled off the last mandrel, the empty tube is drawn through the final die in a sinking or plug-draw pass to remove the ripples generated in reeling and to provide the proper tube size.

Only a limited amount of mandrel drawing of V-20Ti tubing has been done at ANL. These attempts were successful, and no ID defects were generated from the sink component of the drawing operation.

3. Plug Drawing

Plug drawing of TV-20 has been used mainly for the finishing passes, although it also has been used at intermediate stages to correct variations in wall thickness and/or to adjust the D/t ratio resulting from the ductile-core drawing operations. Advantages for plug drawing are close control of ID and better surface finish of the ID. In addition, the reeling operation in mandrel drawing is eliminated as is the final sink draw to provide the proper OD size and remove any surface rippling.

However, for TV-20 (and other materials as well) successful plug drawing is dependent upon many factors, including tube homogeneity, wall thickness and hardness uniformity, plug and die design, lubrication, and degree of reduction. Variations in any of the above factors can result in tube fracture, chatter or neckdown, tool breakage, or variations in drawn size. For smaller diameters, pass reductions must be decreased because of strength limitations of the plug-holding mechanism as well as the tubing itself. In the V-20Ti alloy, occasional variations in tubing hardness (possibly due to variations in chemistry or improper annealing) resulted in corresponding variations in wall thickness and diameter. Similarly, draw loads had to be reduced for long lengths of tubing as the force necessary to pull

the tube through the die sometimes exceeded the elastic limit of the tube in the longitudinal direction (along the drawn length) and the tubing would be plastically stretched with resultant dimensional changes.

D. Tooling Evaluation

1. Dies

Aluminum-bronze (Die-Bronze 25) dies were used to fabricate most of the V-20Ti tubing produced at ANL. The design and sizes of the dies have been mentioned previously.¹ The Al-Bz dies eliminated the major problem of galling from inadequate lubricants when drawing through standard carbide dies. Also, the die material was relatively easy to machine, thus enabling various die designs to be evaluated while producing the required amount of tubing.

Initially, die wear was no problem, but increased quantities and lengths made size control more difficult. Varying the die angle and/or bearing length did not materially reduce wear. Decreasing the reduction per draw pass did help to reduce die wear, but the increased number of dies and draw passes thereby needed were impractical. Dies having an approach angle of 30° were found to be suitable for ductile-core and plug drawing. Drawing was further improved by decreasing the bearing length to 25% of the die size.

Although carbide dies have excellent wear resistance, as evidenced by their widespread usage in commercial tube mills, they were not satisfactory in initial drawing attempts at ANL, mainly because of lubrication difficulties and improper die design. Recently, a few carbide dies were obtained with a 30° included approach angle and a bearing length of 25% of the die size. Although only a limited amount of tubing has been drawn through these dies, results have been very encouraging. Surface quality has not approached, as yet, the finish generated when aluminum-bronze dies were used, but it is expected to improve with more efficient lubrication.

2. Plugs

As with aluminum-bronze dies, draw plugs of hardened beryllium-copper alloy (Berylco 25) had limited wear resistance. Wear increased with decreasing tube size, increasing severity of reduction, and increased tubing lengths. Decreasing the severity of the reduction allowed longer lengths to be drawn within dimensional tolerances required, but required the replacement or conditioning of the plug after each draw.

A limited amount of plug drawing has been tried with standard carbide plugs, but the results were less promising than expected, due to

lubrication problems. With further study, gradual improvement in lubrication should ultimately permit usage of carbide plugs for better wear and dimensional control.

3. Mandrels

Mandrel (bar) drawing was done with Latrobe mandrel steel through-hardened to approximately 55+ R_C. The surface was ground and finished by polishing. The mandrels were coated with a medium-oil lubricant prior to insertion in the tube for drawing.

Although a rigorous evaluation of mandrel materials was not made, brief studies indicated that through-hardening was preferable to case-hardening. The latter type of mandrel is subject to damage in swaging or reeling operations to remove the mandrel after the draw. Surface finish must be good--preferably a polished surface--to prevent defect formation and facilitate mandrel removal. Mandrel lubrication is necessary to permit unrestricted flow of the tube over the mandrel as the wall is thinned and to ease removal.

E. Lubricant Evaluation

Suitable lubricants should facilitate the drawing of high-quality V-20Ti tubing. The lubricant should be easy to apply both to the outer and inner surfaces of tubes, ranging in lengths from 0.6 to 3 m and in sizes as small as 0.381 cm in diameter. Removal of the lubricant after drawing should be readily accomplished by vapor degreasing, dissolution, or washing. If base coatings are necessary, they should last for several draws prior to recoating.

Evaluation of lubricants has been underway for some time. Tubing has been drawn by both ductile-core and mandrel techniques with carbide dies and reductions of approximately 10% per pass. To date, three lubricants have been evaluated with the results as outlined below:

1) Molykote 523X (Teflon aerosol spray). Tubes were spray coated and drawn with and without secondary lubrication. Results were erratic and unpredictable. Varying amounts of galling occurred when drawing long lengths. Inability to coat the tubing inner surface negated the use of plug drawing. Further evaluation of this lubricant was discontinued.

2) Houghtodraw 431. This is a heavy oil used quite successfully in drawing stainless steel tubing. Trial draws on V-20Ti were complete failures. Severe galling would occur almost immediately. Vapor blasting of the tube to provide better lubricant retention did not reduce galling. Further evaluation was discontinued.

3) Hangsterfer #5 P.C. Base Coat and Aldraw J-1 Chlorinated-wax Lubricant. The liquid base-coat compound air dries to form a thin plastic coating. This coating in conjunction with a secondary lubricant has been used with success on both stainless steel and titanium. The base coat and lubricant combination proved successful in drawing V-20Ti tubing. The occurrence of occasional minor galling, due to a tendency of the plastic base coat to strip away from the tube, made redraw without recoating risky. A series of draws were made with various thicknesses of the plastic base coat (achieved by diluting the compound with a suitable thinner prior to application). The thinner coatings did not strip off and more draws between recoating were possible. In addition, light vapor blasting of the tube surface improved adhesion of the base coat and improved the drawn surface finish. Occasional galling has occurred but has been very minor. The base coat is easily removed in a suitable solvent such as chloroethene.

The following techniques were not extensively evaluated for the reasons indicated:

Conversion coatings such as the phosphates, oxalates, etc., are difficult to apply, and the process is generally recommended only for long production runs. Removal is usually accomplished by chemical means.

Plating and anodizing are somewhat involved processes that would require development for a specific alloy. The application of this technique to small tubing bores over long lengths appears somewhat remote. Again, removal is generally by chemical means.

Molybdenum disulfide is easy to apply, but special alkaline baths are required for its removal. (The possibility of incomplete removal might result in material contamination in subsequent annealing operations.)

F. Typical Production Schedule for V-20w/oTi Tubing

Changes in tube-blank starting size and drawing procedure have simplified the production schedule for drawing V-20Ti tubing to sizes of approximately 0.53 cm in OD x 0.45 cm in ID. The typical current schedule is shown in Fig. 3. Contrasted with the previously reported schedule,¹ one vacuum anneal and one swaging operation have been eliminated, even though the starting extruded tube-blank size is larger than before.

Since increased reductions are feasible in mandrel drawing, use of this technique would allow further simplification of the production schedule. If mandrel draws were utilized after annealing the swaged tube-blank, only one swaging operation would be necessary (to "true" the as-extruded tube-blank) and another annealing operation could be deleted (making a total of four anneals from the as-extruded tube-blank). Also, the number of draw passes could be reduced via a heavier reduction/pass, but it is necessary to incorporate a reeling operation to remove the mandrel after each pass.

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Fig. 4. Photograph of "Long-John" Vacuum-annealing Furnace

For operation, the desired annealing temperature (900 to 950°C for V-20Ti) is preset on the recorder-controller. Power is initially applied manually with the table (and induction coil) drive and automatic control being energized when the temperature is within about 50°C of the desired temperature. The Ray-O-Tube is sighted at the tail end of the 15-cm-long induction coil, as this represents the hottest region of the tube. Temperature control is normally within $\pm 5^\circ\text{C}$ of the desired temperature.

The speed at which the coil moves is governed by the size of the tube to be annealed and the annealing behavior of the material itself. The speed and temperature must be matched to provide similar hardness and microstructural changes observed for annealing in conventional furnaces where the complete structure is held at a specific temperature for a given time. For example, swaged TV-20 tube-blanks measuring 2.03 cm in OD x 1.52 cm in ID are annealed at 950°C with the coil moving at a speed of 7.6 cm/min. It is estimated that a given point on the tube is in the temperature range of 850 to 950°C for one minute under these conditions. This provides a hardness and microstructure equivalent to that obtained for annealing the same tube for one hour at 900°C in a conventional furnace. For tubes of thinner wall thickness, the time to reach heat is significantly reduced, thereby allowing either a reduction in temperature or an increase in speed of coil travel.

Fig. 3. Current Production Schedule for V-20w/oTi (TV-20) Tubing

VI. TUBE ANNEALING

A major problem in the fabrication of refractory metal tubing is the availability of suitable high-vacuum, high-temperature furnaces for annealing long lengths of tubing--lengths of 2.4 m or more. At ANL, this problem was alleviated by constructing an induction-heated, vacuum-annealing furnace provided with a moving coil. The "Long-John" vacuum-annealing furnace is shown in Fig. 4. Basically, it consists of a quartz tube attached to a high-vacuum system which is valved to permit reloading and pump-down while the high-vacuum pumps remain on. The quartz tube is capable of handling 3.0-m tubes, and its size can be varied to match coil dimensions and tubing sizes for better coupling. An ionization gauge is located in the tail end of the tube. A variable-speed motor drives a table on which the induction coil and a Leeds and Northrup Ray-O-Tube temperature detector are located. The table can be driven at speeds ranging from 1.3 to 51 cm/min. The temperature detector signals a Leeds and Northrup Current-adjusting Type (C.A.T.) control system which, in turn, controls the output of a 30-kW motor-generator set.

Two problems are inherent in the design of this furnace. The first is that of reduction in light transmission of the quartz tube due to "fogging." This has the effect of reducing the temperature seen by the Ray-O-Tube, thus calling for increased power output by the motor generator. In many trials, however, this has not been a serious problem. In fact, the system balances sufficiently well that the control system can be set at a given power output (independent of the Ray-O-Tube signal) and maintain the proper tube temperature.

The second problem is that of vacuum control should the tube outgas during annealing. Use of an induction coil surrounding the quartz tube vacuum chamber requires that the quartz tube be only slightly larger than the metal tube to be annealed to promote coupling between the coil and work-piece. For the V-20Ti swaged tube-blank measuring 2.03 cm in OD x 1.52 cm in ID, the quartz tube is of 3.18-cm OD x 2.54-cm ID. Since the small-diameter quartz tube is about 36 m long, it is difficult to maintain high dynamic vacuums (10^{-4} Torr or better) if outgassing occurs. To eliminate this problem, a rapid traverse of the tube at a lower temperature will outgas the tube with no accompanying fogging of the tube. Following this, the tube is given the standard anneal.

To date, the furnace has performed in fine fashion in annealing V-20Ti tubes ranging in size from 2.03-cm OD x 0.25-cm wall to 0.95-cm OD x 0.076-cm wall.

VII. COMMERCIAL EFFORTS IN FABRICATION OF V-20w/oTi

With a successful fabrication technique for high-quality tubing having been established at ANL, commercial vendors were asked to participate to insure commercial capability should the need for large quantities of tubing arise. As no single source could perform all operations, various separate phases were evaluated.

A. Ingot Production

In ANL-6928¹ the production and resultant quality of three V-20Ti ingots produced by the Union Carbide Corporation were discussed. These ingots were of comparable quality to those produced at ANL and were fabricated into various products (sheet, rod, and tubing) with no difficulty. No additional ingots have been procured from commercial sources since that time.

B. Breakdown Extrusion

The procurement of 4.8-cm-diameter V-20Ti bar stock from the Union Carbide Corporation required an ingot breakdown extrusion of the 15.2-cm arc-cast ingots produced by the vendor. This extrusion was performed by the Canton Drop Forge Company on a 2500-ton horizontal extrusion press. Two ingots were jacketed in a single, mild-steel can, heated to 1105°C in a salt bath, and extruded at a 7.3:1 reduction ratio to 5.1-cm-diameter bar stock. No difficulties were encountered in the extrusion although a faulty weld on the mild steel can permitted molten salt to contact the V-20Ti ingots. Removal of the steel after extrusion showed that no attack of the V-20Ti by the salt had occurred.

While only one breakdown extrusion has been attempted by commercial sources, all indications are that this operation can be successfully accomplished.

C. Tube-blank Extrusion

No commercial attempts have yet been made to extrude tube-blanks of V-20Ti. Two firms (Nuclear Metals, Inc., and the DuPont Company Metals Center) have indicated they are willing to extrude tube-blanks of approximately 2.54-cm OD x 0.32-cm wall thickness. Suitable billet material is not yet available to proceed with these trials.

D. Secondary Fabrication of Tubing

Two firms--Wolverine Tube Division of Calumet and Hecla and Superior Tube Company--have attempted to fabricate high-quality, small-diameter, thin-walled tubing of V-20Ti from tube-blanks furnished by ANL.

Wolverine Tube utilized tube reducing for the majority of their fabrication schedule. Starting with a 1.880-cm-OD x 0.292-cm-wall x 30.5-cm-long tube-blank, their fabrication schedule was as follows:

- 1) Tube reduce to 1.587-cm OD x 0.203-cm wall.
- 2) Vacuum anneal for one hour at 900°C.
- 3) Tube reduce to 1.270-cm OD x 0.130-cm wall.
- 4) Tube reduce to 0.953-cm OD x 0.076-cm wall.
- 5) Vacuum anneal for one hour at 900°C.
- 6) Tube reduce to 0.635-cm OD x 0.051-cm wall.
- 7) Vacuum anneal for one hour at 900°C.

8) Plug draw to 0.572-cm OD x 0.046-cm wall.

9) Plug draw to 0.531-cm OD x 0.038-cm wall.

Approximately 2.6 m of finished tube were received for inspection. Wolverine concluded that the alloy could be cold worked by tube reducing and plug drawing with relative ease. The material exhibited room-temperature fabricability similar to that of the columbium-base alloy D-43 (Cb-10W-1Zr-0.1C). The small yield of finished-size tubing resulted from difficulties in maintaining mandrel position during tube reducing from 0.953-cm OD x 0.076-cm wall to 0.635-cm OD x 0.051-cm wall. This was in no way related to the properties of the alloy. However, the difficulties encountered in the tube-reducing operation necessitated some free sinking (no internal support) in the following drawing operation which usually has an adverse effect on quality.

Eddy-current inspection of this tubing at ANL revealed that the tubing possessed numerous areas with defects greater than 10% of the wall thickness. Of the 2.6 m received, only 1.7 m were free of defects at this level, representing a 65% yield.

Although the overall quality of the tubing was not the best, it was established that the V-20Ti could be tube reduced successfully.

A great deal of the commercial tube-drawing of V-20Ti has been done by the Superior Tube Company. A typical specification for fabrication of finished tubing is given in Appendix C. To date, they have processed four orders ranging in size from 0.754-cm OD x 0.051-cm wall to 0.498-cm OD x 0.038-cm wall. Details on material recovery from these orders is given in Table VIII.

TABLE VIII. Results of Tube Drawing V-20w/oTi at the Superior Tube Company

ANL Order Number	Starting Tube-blank		Finished Tubing Received ^a			Acceptable Tubing ^b Length, m	Tubing Yields, %	
	Size, cm	Weight, g	Size, cm	Weight, g	Length, m		Received Tubing to Starting Tube Blank	Acceptable Tubing to Received Tubing
458947	1.905 OD x 1.270 ID x 66 long	584	0.526 OD x 0.038 wall	340	10.1	9.9	58.2	98.0
484294	2.032 OD x 1.524 ID x 259 long	2,054	0.754 OD x 0.051 wall	1,589	23.2	22.6	77.4	97.4
506668	2.032 OD x 1.524 ID x 297 long	2,339	0.559 OD x 0.041 wall	1,573	43.0	42.5	67.2	98.9
507820	2.032 OD x 1.524 ID x 117 long	960	0.498 OD x 0.038 wall	566	18.3	18.0	59.0	98.3

^aDoes not include pieces shorter than required minimum length. On order 484294, additional 1.3 m of acceptable tubing in lengths less than 51 cm received, representing an additional 5.1% yield. On order 506668, an additional 3.0 m of acceptable tubing in lengths less than 91 cm received, representing an additional 4.1% yield. On order 507820, additional 2.1 m of acceptable tubing in lengths less than 51 cm received, representing an additional 6.8% yield.

^bAcceptable tubing has defects less than 10% of the wall thickness as determined by X-ray and eddy-current inspection.

Although the specific schedules used for drawing are proprietary, area reductions of approximately 35% maximum per pass were taken with in-process anneals at 875°C after about 50% total reduction. The "as drawn" hardness ranged from 98 to 103 R_B, while the annealed hardness ranged from 86 to 93 R_B.

No difficulties were encountered in processing the tubing, and the yield of finished tubing from starting material of approximately 60 to 75% is considered good. All tubing received from Superior Tube Company possessed good dimensional control and was of excellent quality as evidenced by yields of acceptable tubing from received footage of 97% or more.

VIII. DISCUSSION

This report covers additional fabrication development on the V-20Ti alloy over that described in ANL-6928.¹ Much of the work reported herein represents process improvements which subsequently have improved material yields and/or quality. Two major improvements merit attention.

The alloy in tube form has proved amenable to heavy reductions by tube-reducing. A cursory trial by a commercial vendor with a conventional two-roll tube-reducing machine indicated that the alloy can be successfully tube reduced. However, final results were marred by difficulties in maintaining mandrel position, which affected the quality and quantity of finished tubing.

A tube-reducing machine employing a unique three-roll principle was designed and built at ANL. Although the machine is still in its trial period, it has proved successful in tube-reducing the V-20Ti alloy. Reductions up to 70% have been achieved in a single pass while maintaining tube quality.

The second area of improvement is the use of a thin, plastic base coat on the tubing which, in conjunction with a chlorinated-wax lubricant, has enabled carbide draw dies to replace aluminum-bronze dies previously used. This has greatly extended die life and significantly improved dimensional control of drawn tubes. Unfortunately, a suitable coating technique or lubricant for the internal tube surface has not been found which will enable the hardened beryllium-copper plugs used in plug drawing to be replaced by carbide plugs. Further work in this area is needed.

In addition to process improvements at ANL, commercial capability has been established for drawing V-20Ti tubing. Approximately 94.6 m of finished tubing has been processed from tube-blanks supplied by ANL, which represents a highly acceptable 68% yield, not including an additional 5% of acceptable tubing shorter than the required minimum length. From received tubing, an average 98% yield was acceptable through X-ray and eddy-current nondestructive testing.

IX. CONCLUSIONS

An integrated fabrication technology for producing high-quality, small-diameter, thin-walled tubing of the V-20w/oTi alloy has been established. Processes have been improved and demonstrated successful by repeated processing. Additional processes or modifications could be introduced based on capabilities of other fabricators.

Redrawing of tube-blanks by a commercial vendor has been successfully demonstrated on a number of occasions with excellent results. Other areas of processing on a commercial basis--especially extrusion of tube-blanks--need to be developed.

X. ACKNOWLEDGMENTS

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XI. REFERENCE

1. W. R. Burt, Jr., et al., *Consolidation and Fabrication Techniques for Vanadium-20 w/o Titanium (TV-20)*, ANL-6928 (Feb 1965).

XII. APPENDIX A

Effect of Iron Content on the Fabricability of V-20w/oTi

In initial fabrication operations of V-20Ti, difficulties were encountered that were thought to be influenced by the level of iron impurity in the alloy. To evaluate the effect of iron, several small arc-cast buttons of V-20Ti were prepared containing varying amounts of iron.

Vanadium chips containing varying amounts of iron and titanium sponge served as charge material. The charge materials were selected to provide arc-cast buttons containing approximately 200, 700, 1500, 3000, and 5000 ppm iron. Pure iron was added to the charge for the last three alloys to provide the proper iron content.

The charge materials were melted in a small nonconsumable-electrode arc furnace. The resultant buttons were canned in mild steel and hot rolled at 900°C to sheet. After dejacketing, the sheet was cold rolled to a 50% reduction in area and vacuum annealed for one hour at 900°C.

The effect of iron was evaluated by noting differences in hot and cold rolling characteristics and from hardness measurements in the as-cast, cold-rolled, and annealed conditions. The hardness measurements are summarized in Table IX.

TABLE IX. Hardness Values for V-20w/oTi with Iron Content

V-20Ti Alloy	Approximate Iron Content, ppm	Hardness		
		As Cast	Cold Rolled 50%	Annealed One Hour at 900°C
A	200	95 R _B	96 R _B	90 R _B
B	700	96 R _B	95 R _B	88 R _B
C	1500	96 R _B	96 R _B	90 R _B
D	3000	21 R _C	20 R _C	95 R _B
E	5000	25 R _C	24 R _C	98 R _B

For the alloys containing up to 1500 ppm, no noticeable differences were observed in fabrication of sheet. However, the iron alloys containing 3000 and 5000 ppm were slightly more difficult to cold roll, and there was a marked propensity for edge cracking in the alloy containing 5000 ppm iron. As seen from the hardness data, no marked increase in hardness is observed until the iron content reaches 3000 ppm. It was apparent that an iron content ≤ 1000 ppm would cause no fabrication difficulty. (The iron impurity limit presently used in specifications for vanadium chips is ≤ 500 ppm.)

XIII. APPENDIX B

Current Specifications for Vanadium Chips1. Scope

This specification covers vanadium chips in the $-1/4$ in. $+20$ mesh size range. Chip surfaces shall be free of excessive oxidation as evidenced by obvious discoloration.

2. Preparation

Chips may be prepared by any technique that will produce vanadium satisfying the size and purity requirements of this specification.

3. Chemistry

Chips shall be 99.75% (minimum) vanadium. Total interstitial (C, O₂, N₂, H₂) impurity content shall be <0.15 w/o (1500 ppm). Total other impurities shall be <0.10 w/o (1000 ppm). Maximum limits for individual impurities are: oxygen--700 ppm, nitrogen--400 ppm, carbon--400 ppm, hydrogen--50 ppm, iron--500 ppm, and silicon--500 ppm. The vendor shall supply certified chemical analyses, on samples taken from the lot(s) of chips supplied, for oxygen, nitrogen, carbon, hydrogen, iron, silicon, and any other significant impurities. The purchaser will perform chemical analyses to confirm analytical results supplied by the vendor. All individual analyses shall meet the requirements of this specification.

4. Rejection

Material not conforming to this specification or to authorized modifications shall be subject to rejection. Unless otherwise specified, rejected material may be returned to the vendor at the vendor's expense, unless the purchaser receives, within 3 weeks of notice of rejection, other instructions for disposition.

XIV. APPENDIX C

Typical Specification for Fabrication of V-20w/oTi Tubing

1. ACKNOWLEDGEMENT. The vendor shall refer to this specification number in all quotations, correspondence, and acknowledgements of purchase orders.
2. STARTING MATERIALS
 - a) Description. Starting material for producing seamless V-20w/oTi tubing shall consist of extruded and swaged tube-blanks furnished by Argonne National Laboratory. The tube-blank will be in a fully annealed condition with a hardness of approximately 95 RB.
 - b) Size. The tube-blanks are 0.800 ± 0.005 in. in OD x 0.600 ± 0.005 in. in ID x ~38 in. long. Ovality of the tube-blanks (maximum wall--minimum wall around a circumference) is within 0.007 in.
 - c) Quantity. In the quotation the vendor shall state the number of tube-blanks required to produce the required footage of finished tubing detailed in section 3a.
3. REQUIRED TUBING
 - a) Size and Amount. The following items shall be supplied under this purchase order:
 - i) 120 ft of seamless tubing: $0.220 +0.000, -0.001$ in. in OD x 0.188 ± 0.0005 in. in ID x $36 \pm 1/8$ in. long or multiples thereof (6 ft, 9 ft, etc.).
 - ii) 36 ft of seamless tubing: $0.220 +0.000, -0.001$ in. in OD x 0.180 ± 0.0005 in. in ID x $36 \pm 1/8$ in. long or multiples thereof.
 - b) Otherdimensional tolerances. Ovality of the tubing shall be held within the OD tolerance of $+0.000, -0.001$ in. Eccentricity as measured by the difference between maximum and minimum wall thickness around the circumference shall not exceed 0.003 in. The tubes shall be free of bends and/or kinks, and the maximum bow shall not exceed one part in 1200.
4. CONDITION. The delivered tubing shall be in a fully annealed condition with the heat treatment being done in a vacuum of 10^{-4} Torr or better. Pieces of finished tubing longer than 6 in. but shorter than the required 36 in. length shall also be annealed and delivered.

The annealing heat treatment shall consist of one hour at 900°C. The tubing shall have been cold worked to a minimum reduction in area of 35% between the last in-process anneal and the final full anneal heat treatment.

5. TUBING QUALITY. The delivered tubing shall be uniform in quality, condition, and cleanliness. The surfaces shall be mechanically or chemically cleaned, and free of residual drawing lubricants, cutting compounds, cleaning fluids, etc.

The tubing shall be free from scratches, dents, laps, seams, inclusions, and other surface imperfections. External and internal cracks, laps, and other defects shall be held to a depth not exceeding 10% of the wall thickness as determined by ultrasonic or eddy-current nondestructive testing.

The purchaser reserves the right to inspect the material by any and all methods (e.g., eddy current, dye penetrant, etc.) which may reveal nonconformance with the quality requirements.

6. IDENTIFICATION. Finished tubing shall be tagged or marked with an alcohol-soluble ink corresponding to the original tube-blank number.
7. PACKAGING. The finished tubes shall be individually wrapped in paper or placed in paper tubes to prevent marring of the surface. The tubes shall be boxed for shipment so as to prevent denting, bending, etc.