FABRICATION OF CREEP TUBING FROM THE US AND NIFS HEATS OF V-4Cr-4Ti –
A.F. Rowcliffe, (Oak Ridge National Laboratory), W.R. Johnson (Rocket Science & Materials Engineering Services), D.T. Hoelzer (Oak Ridge National Laboratory)

OBJECTIVE

Utilize commercial–scale processing to fabricate small-diameter, thin-wall tubing from plate stock of V-4Cr-4Ti for both the US program heat no.832665 and the NIFS –HEAT-2 and produce sufficient tubing to meet foreseeable programmatic needs for investigating creep behavior in both lithium and vacuum environments and for carrying out irradiation creep measurements

SUMMARY

Commercial-scale fabrication of thin-walled tubing for thermal and irradiation creep testing of V4Cr-4Ti has been initiated at Century Tubes Inc, San Diego. In an effort to minimize the occurrence of the surface flaws and cracks which characterized the previous batch of tubing, the technical specifications have been extensively modified. In particular improvements have been made in the cleaning procedures and in the control of the vacuum during all stages of the annealing cycle; the levels of cold work applied during the final stages have been substantially reduced. Both the US program heat and the Japanese NIFS-HEAT-2 are being processed; interim examination shows significant increases in oxygen content for both heats in spite of the added precautions introduced. Surface defects similar to those observed in the previous batch of tubing were detected in one section of tubing from the US heat after 4 draw cycles while the NIFS heat was relatively free of surface defects.

PROGRESS AND STATUS

Introduction

The pressurized tube creep specimens adopted by the US and Japanese programs measures 25.4 mm long with a 4.57mm outside diameter, and a wall thickness of 0.25mm. In 1995, Argonne National Laboratory coordinated a campaign to fabricate ~ 6 meters of tubing from the US program heat no 832665 with Century Tubes Inc of San Diego as the primary contractor [1]. This campaign met with mixed success with a large fraction of the tubing developing cracks on both the inside and outside surfaces. These cracks were frequently linked together through the wall thickness. Since they were long enough to be visible to the naked eye, it was possible to select relatively sound segments of tubing to prepare a sufficient number of creep specimens to meet short term program needs. This tubing was used for an initial series of testing both in vacuum and in lithium environments [2] and also used to develop preliminary irradiation creep data in experiments conducted in the ATR[1] and in the HFIR [3]. It was also found that during processing, the level of interstitials elements increased significantly, with carbon increasing from 80 to 300 wppm, oxygen increasing from 310 to 700 wppm and nitrogen going from 85 to 95 wppm, a total increase in interstitial level of ~130%. The increase in oxygen was of particular concern since creep rate is sensitive to oxygen concentration in this range. However, in subsequent creep measurements using this tubing it was observed that the oxygen content decreased to ~100wppm in approx. 1500hrs during tests carried out at 800ºC in a Li environment. Thus it is not entirely clear that the in-Li creep data were compromised by the initially high oxygen level of the ANL batch of tubing since the oxygen concentration reverted to very low levels during testing. However, it has been suggested that the presence of the defects in the tube wall could be responsible for inconsistencies in the measured strains to failure [2].

Two small heats of V-4Cr-4Ti with low levels of interstitials have been produced in Japan under the direction of the National Institute for Fusion Science (NIFS). A small quantity of creep tubing was prepared from the NIFS-HEAT -2 material using a three-directional rolling process [4]. Problems were also encountered in the NIFS campaign with interstitial pick-up and the development of surface defects. Based upon the processing records from the previous batch of tubing from the US heat and

the experience gained from processing the NIFS heat, a new procedure was developed which incorporated a series of changes designed to minimize interstitial pick-up, improve initial surface quality, and to reduce the probability of surface cracking by reducing stress levels in the final drawing stages. Using the new procedure, fabrication of ~10 meters of finished tubing from both heats was initiated at Century Tubes in April 2003.

This report documents the new procedures and discusses the status of the tubing fabrication as it enters the final stages; a full evaluation of the finished tubing will be presented in the next semi-annual report.

Previous Tubing Fabrication Experience

US HEAT 832665

Tubing from the first campaign at Century Tube (subsequently referred to as Batch A tubing) has been used to provide the initial sets of biaxial creep data in vacuum, lithium, and lithium plus neutron radiation environments. During the final stages of drawing and sinking, difficulties were experienced with surface cracking and sometimes complete fracture of tubes. Metallography of several sections was carried out at ORNL. Five out of six randomly selected sections of tubing contained branching cracks at both the ID and OD surfaces penetrating inwards to depths from 20 to 200 microns**.** Longitudinally, these cracks extended from 2 to 200mm in length and so were often visible to the naked eye. Cracks penetrating from both surfaces were often connected by a band of severe macroscopic deformation in which grains were rotated and elongated in a direction 45 degrees to the through –wall radial direction as shown in Fig.1. Many smaller cracks simply penetrated 10-20 microns in from both surfaces. Chemical analysis of the finished tubing indicated that the carbon and oxygen concentrations increased during processing by 220 wppm and 389 wppm respectively. Because archival samples were not secured at each drawing stage it is not possible to say when the cracks developed. One plausible scenario is that the large defects in the OD developed first in the oxygen-contaminated surface region and that as the wall thickness was reduced, the cracks penetrated an ever greater fraction of the wall. The decrease in effective load –bearing cross section resulted in localized shear stresses sufficient to create a band of severe macroscopic deformation accompanied by grain rotation and elongation. Intersection of this band with the surface resulted in the development of a corresponding ID crack. The summary of the processing provided by ANL [1] and the notebook records at Century Tubes Inc. suggested a number of factors that could have resulted in the observed defects; these are discussed in the following.

The source material for Batch A tubing was the rectangular cross- section (63.5mmx190mm) as-extruded bar supplied by Teledyne Wag Chang Co. This material was used to produce the initial tube blank (27.9mm OD x 19.1mm ID x 200mm long) with the long direction parallel to the extrusion direction. The initial microstructure for Batch A was consequently very inhomogeneous and characterized by a mixture of small grains (20-60 microns),coarse equiaxed grains (100-200 microns) and banded regions of deformed (unrecrystallized) grains [7]. There is considerable evidence that Ti segregates strongly during ingot solidification and during extrusion at 1150ºC these regions become drawn out parallel to the extrusion direction and eventually give rise to the bands of Ti (CON) particles that characterize the subsequent cold worked and annealed microstructure.

During the processing of Batch A, the cold drawn tubing was encased in Ti foil and recrystallized at 1025ºC in a vacuum of ~7 x 10^{-5} torr. Furnace records show that because of the rapid heating rate employed, the vacuum quality often deteriorated into the 10^{-4} torr range as a result of out-gassing. The lack of control of the vacuum quality during heat-up is the most likely reason for the significant oxygen pick-up observed. Examination of the cleaning procedures used following each draw cycle suggested that the acrylic lubricant used was not completely removed during cleaning and was probably the source of the observed carbon pick-up.

The tube drawing cycle utilized in the production of Batch A is summarized in Table 1. A set of 10 draw cycles were used with intermediate recrystallization at 1025ºC for 1 hour; each draw cycle

consisting of 3 approximately equal passes. The reductions in area for the final 5 cycles were in the range 40-45%.and is it possible that these relatively high levels of work hardening coupled with oxygen pick-up could have led to the conditions which resulted in the development of the observed surface cracking. In addition, cycles 2-4 were drawn with a constant diameter mandrel so that the ID did not change ; this procedure necessitated a fairly heavy reeling operation to remove the tube from the mandrel prior to annealing and it is possible that this also contributed to the development of the surface cracks.

Table 1. Summary of drawing schedule used for Batch A tubing from US heat 832665 of V-4Cr-4Ti

Cycle No.	OD (ins)	ID (ins)	Wall (ins)	RA/Cycle (%)	Anneal No.
Tube blank	1.100	0.770	0.165		1
1	0.981	0.750	0.117	40.6	2
2	0.920	0.750	0.085	33.3	3
3	0.863	0.750	0.057	41.1	4
4	0.830	0.750	0.040	34.4	5
5	0.755	0.700	0.029	37.6	6
6	0.688	0.650	0.020	41.5	7
7	0.606	0.580	0.015	41.1	8
8	0.520	0.500	0.010	45.9	9
9	0.288	0.264	0.012	40.0	10
10	0.178	0.157	0.011	45.4	11

NIFS-HEAT-2

Before the second campaign at Century Tube was initiated, researchers at NIFS reported on the fabrication of a small quantity of pressurized creep tubing and some larger size tubing for tritium permeation studies using plate from the high purity NIFS-HEAT-2. A tube blank with a 25.0mm OD and 19mmID was machined from a cold-worked 26mm square section and initially recrystallized at 1000ºC. Tube reduction was carried using a 3-directional rolling process with intermediate annealing at 825ºC. This temperature was selected to minimize oxygen pick-up but is apparently sufficiently high to effect recrystallization with a final grain size of ~20 microns [4]. Creep tube fabrication was accomplished in 7 rolling passes, utilizing four anneals at 850ºC and one final anneal at 1000ºC resulting in a final grain size of 39 microns. The processing resulted in an increase in oxygen concentration of 200 wppm and an increase in carbon and nitrogen by 70 and 30 wppm respectively. Early problems with cracking were eliminated by a) lowering the heat treatment temperature of the initial plate to 1000ºC (finer grain size) and b) limiting the reduction in area per cycle to below 40%. The ID surface quality was improved by honing following gun drilling. The final creep tubing contained surface defects up to 20 microns deep and it was suggested that this cracking is related to the intersection of bands of Ti(CON) particles with the tube surfaces.

REVISED PROCEDURE FOR TUBING FABRICATION

Based upon the prior experience summarized above, a number of changes were introduced into the procedure for fabricating thin-wall tubing which were designed to decrease the magnitude of interstitial pick-up and to minimize the probability of generating the large surface defects which developed during the fabrication of Batch A of the US heat. The complete technical specification that is being followed in the current campaign to produce creep tubing from both the US and the NIFS heats of V-4Cr-4Ti is presented in the Appendix. The major changes introduced compared to the Batch A procedure are summarized below.

a) For the US heat, the initial as-extruded plate was cold worked 50-55% prior to recrystallizing at 1000ºC to give an average grain size of ~30 microns. Although this treatment did not improve chemical inhomogeneity or alter the distribution of Ti(CON) particles, the uniformity of the grain structure was greatly improved compared with Batch A. Gun drilling of tube blanks was followed by honing to a surface finish of 16 rms or better

b) A more rigorous cleaning operation was introduced after each draw cycle involving successive treatments in Alconox, acetone and ethyl alcohol followed by an acid cleaning.

c) Prior to annealing, the vacuum furnace was baked out at 100ºC above the annealing temperature to remove sources of contamination. Tubes were completely wrapped in tantalum foil and annealed in a vacuum of better than $2x10^{-5}$ torr; this vacuum was maintained during all out-gassing stages during heat-up. The first 4 annealing treatments were carried out at GA San Diego. Subsequently operations were transferred to a furnace that could accommodate up to 5ft tubing lengths at Bodycote Inc. where the vacuum was maintained in the 10^{-6} torr range.

d) To minimize stresses the drawing cycle was selected so that for the final 6 stages , the reductions in area did not exceed 30%. All draw cycles consisted of 3 approximately equal passes of mandrel draws using a series of mandrels of reducing diameter so that both the ID and OD were reduced in each cycle. Decreasing the reduction in area per cycle necessitated an increase in the number of annealing treatments with the corresponding risk of increase the amount of oxygen pick-up.

e) Archive samples for metallography and chemical analysis were removed after each draw and after each vacuum anneal.

INTERIM QUALITY ASSESSMENT

Tube Blanks

The starting material for the US Heat was a 28mm square cross-section measuring 230 mm long which had been cut from an extruded plate which had been cold rolled ~55%. A tube blank was machined measuring 25.7 mm OD and 15.7 mm ID. Ultrasonic testing and X-radiography failed to detect any defects in the tube blank. The source material for the NIFS-HEAT -2 was a 26mm thick plate, 28.6 mm wide and 200mm long from which a similar tube blank was machined. Ultrasonic testing of the NIFS tube blank indicated that several areas contained defects. Subsequent examination under low power stereo showed possible surface cracks 0.5-1.0mm long parallel to the rolling direction, several possible cracks at 45 degrees to the tube axis and several short 0.1-0.3mm circumferential defects. Nagasaka et. al also detected defects in the 26t plate material using ultrasonic methods and presented metallographic evidence for elongated cavities ranging up to 0.5mm long parallel to the extrusion direction [5].

Interstitial pick-up

Archive samples were removed after each draw cycle and after each annealing treatment. The results of chemical analysis for carbon oxygen and nitrogen up to the seventh drawing/eighth annealing cycle are shown in Table 3. At the end of the eighth anneal, the oxygen content of both heats has approximately doubled with very little change occurring in either nitrogen or carbon concentrations. With 5 more annealing treatments to complete the processing schedule, (Table 2), the increasing oxygen concentration is a concern. If the increase continues at the average rate of ~40 appm per cycle for the US Heat and ~30 wppm per cycle for the NIFS heat then the oxygen concentrations of the finished tubing could approach 850 and 530 wppm respectively. During the course of this study several anomalously high analyses were obtained for both oxygen and carbon and the origins of these results are difficult to pin down. It was established however that cutting specimens with a high speed saw produces surface oxidation and is a leading source of anomalously high oxygen analyses. This is probably not the only means of contaminating chemical analysis specimens and careful cutting

Table 2. Summary of drawing schedule for US heat 832665 (Batch B) and NIFS-HEAT-2 of V-4Cr-4Ti.

Cycle No.	OD (ins)	ID (ins)	Wall (ins)	RA/Cycle (%)	Anneal No.
Tube Blank	1.010	0.626	0.192		1
1	0.940	0.600	0.170	17.3	2
2	0.818	0.542	0.138	30.4	3
3	0.723	0.499	0.112	31.3	4
4	0.588	0.420	0.084	44.4	5
5	0.495	0.375	0.060	44.7	6
6	0.395	0.299	0.048	42.5	7
7	0.318	0.240	0.039	39.0	8
8	0.288	0.224	0.032	29.2	9
9	0.262	0.212	0.025	29.0	10
10	0.246	0.208	0.019	30.2	11
11	0.229	0.199	0.015	31.0	12
12	0.203	0.179	0.012	32.0	13
13	0.188	0.168	0.010	21.0	
SIZING	0.180	0.160	0.010	5.5	
				Final	

Table 3: Chemical analysis of tubing

Cycle No.	Wall (ins)	US HEAT			NIFS HEAT		
		C wppm	O wppm	N wppm	C wppm	O wppm	N wppm
Tube Blank	0.192	119	331	88	59	156	130
ANN 1	0.170	138	329	93	67	132	135
ANN 5	0.060	175	426	89	124	312	150
ANN 6	0.048	119	511	94			
ANN 7	0.039	155	494	84			
ANN 8	0.032	179	637	101	90	378	148

and cleaning procedures need to be rigorously followed to avoid contamination with oxygen or carbon.

Surface defects

At the end of the fifth drawing cycle numerous longitudinal surface defects were observed on the OD of the US heat tubing which at this stage had a wall thickness of 0.060 ins. One of the LT surfaces of the original plate was designated as the top surface (zero degrees), and the corresponding position on the tubing was tracked throughout the drawing sequence. The defects occurred in a band parallel to the drawing direction at a location 90 degrees from the original top surface of the plate. The defects appeared to consist of very shallow regions,1-2mm long, which had separated from the tube surface. Such defects are not uncommon in drawing tubes from BCC alloys and it is normal practice to remove them by local mechanical polishing before each draw. After the fifth draw the tube from the US heat was cut into 2 sections to fit into the annealing furnace. The region containing the defects

was confined to one tube (Tube A) while the second tube appeared to be defect-free (Tube A1). Despite careful polishing to remove the defects, they continued to appear in Tube A after each subsequent draw. After the tenth draw, it was possible to cut a sample from a region containing OD surface defects and a cross-section is shown in Fig.2. Comparison with Fig.1 shows that the surface defects are very similar to the type of defect frequently encountered in Batch A tubing from the US heat. Since these defects develop at an early stage in the drawing/annealing sequence and at a particular circumferential location, they are probably related to some macroscopic feature of the initial plate microstructure such as regions of highly banded microstructure, internal porosity or inclusions.

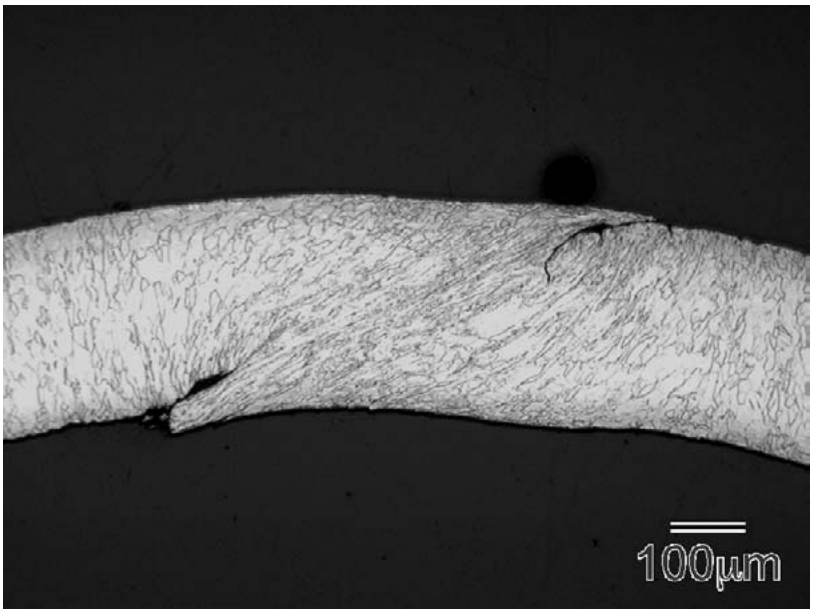

Figure 1. Surface defects in final tubing from US heat Batch A.

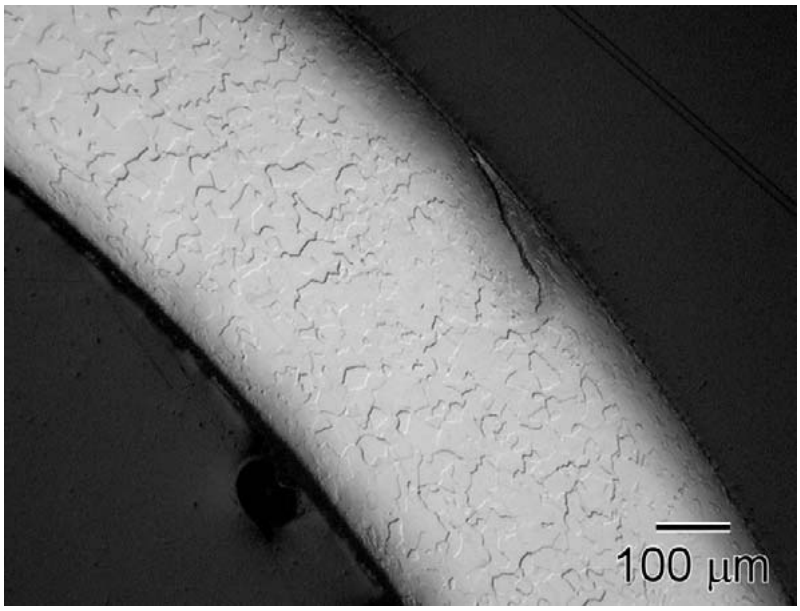

Figure 2. Surface defect in Tubing from US heat Batch B following the fifth drawing cycle.

Microstructure

Representative micrographs from both heats of tubing following the fourth draw cycle and fifth anneal are shown in Fig 3. The average grain size of the NIFS heat is very uniform with an average grain size of ~ 50 microns. The US heat contains a much higher fraction of small grains (5-10 microns) with an overall average size of ~ 28 microns. These differences are almost certainly related to the differences in initial interstitial content which is linked to the initial volume fraction and distribution of Ti(CON) particles. A cross section of the US heat following the third draw /fourth anneal is shown in Fig 4. This sample was stained during etching and at low magnification shows the macroscopic aspects of the deformation process during tube drawing. During etching, staining occurred along the bands of Ti(CON) particles revealing the significant macroscopic rotation of the grain structure which occurs as the outer surface is approached; the bands of particles apparently rotate with the deforming /rotating grains A similar phenomenon has been reported in the fabrication of NIFS heat tubing by 3-directional rolling and during cross-rolling of NIFS plate material [6]

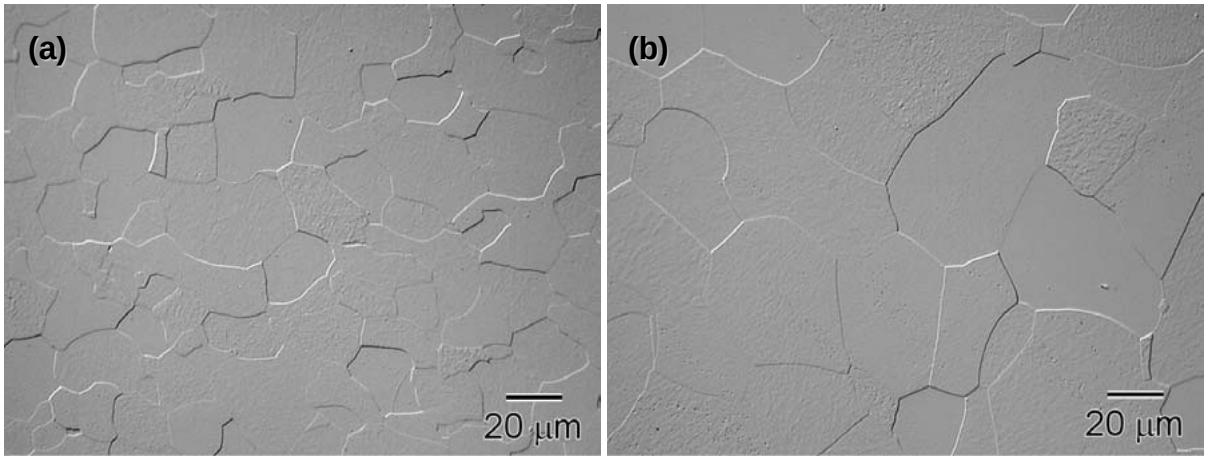

Figure 3. Grain structure of (a) US heat tubing (Batch B) and (b) NIFS heat tubing following the fifth annealing treatment.

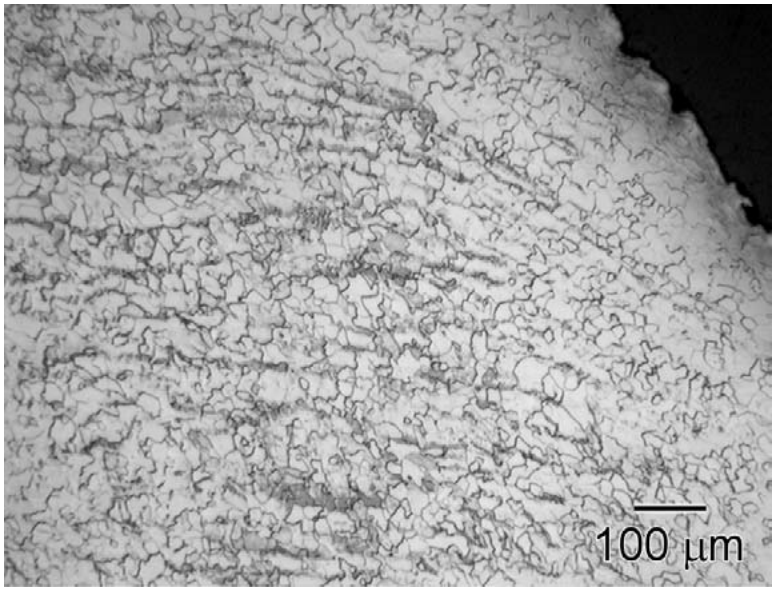

Figure 4. Optical micrograph with stain etch of the US heat (Batch B) following the 4th annealing treatment.

Future work

Fabrication of both heats will continue in accordance with the schedule shown in Table 2, paying close attention to the section of the US heat containing the visible defects and eliminating this section if necessary. Because of the development of different grain structures in the two heats it may be necessary to adjust the final heat treatment of the NIFS heat to reduce the grain size and ensure that there is a sufficient number of grains across the wall of the final creep tubing. A full evaluation of the finished tubing will be carried out including dimensional tolerance inspections, optical microscopy, TEM, chemical analysis, hardness, texture measurements and possibly ultrasonic and eddy current inspection.

REFERENCES

1. H. Tsai, M.C. Bilone, R.V. Strain, and D.L. Smith; Fusion Materials Semi-annual Progress Report DOE/ER-0310/23, December 1997, p149.
2. M.L. Grossbeck, R.J. Kurtz, L.T. Gibson and M.J. Gardner; Fusion Materials Semi-annual Report DOE/ER-0310/32, June 2002, p6.
3. H. Tsai, M.C. Billone, T.S. Bray and D.L. Smith; Fusion Materials Semi-annual Progress Report DOE/ER-0310/27,December 1999, p 65.
4. T. Nagaska, T. Muroga, T. Iikbo; “Development of Tubing Technique for High –Purity Low Activation Vanadium Alloys”, ANS Winter Meeting, Washington DC, Nov.2002, (to be published).
5. A.F. Rowcliffe and D.T. Hoelzer; Fusion Materials Semi-annual Progress Report DOE/ER-0310/25, December 1998, p42.
6. T. Nagasaka et. al, “Fabrication of High-Purity V-4Cr-4TiAlloy Products”; 2nd Workshop on the Development of Low Activation Vanadium Alloys for Fusion, NIFS, March 2002.

APPENDIX

TECHNICAL SPECIFICATION FOR FABRICATION OF THIN-WALL V-4Cr-4Ti ALLOY TUBING

TABLE OF CONTENTS

1.0 Background

2.0 General Information

3.0 Fabrication Requirements

- 3.1 Definition of Responsibilities
- 3.2 Fabrication Procedures
- 3.3 Material Contamination Precautions

4.0 Quality Assurance Requirements

- 4.1 Inspection Rights
- 4.2 Notification Points
- 4.3 Sampling and Hold Points
- 4.4 Records Maintenance

5.0 Fabrication Procedures

- 5.1 Characterization of the Raw Material Stock
- 5.2 Manufacture of Tube Blanks
- 5.3 Cleaning of Tube Blanks and Drawn Tubing
- 5.4 Heat Treatment of Tube Blanks and Drawn Tubing
- 5.5 Tube Fabrication (Drawing/Expansion/Reduction)

6.0 Packaging and Shipment

- 6.1 Identification and Marking
- 6.2 Packaging and Package Marking

7.0 Acknowledgement

1.0 BACKGROUND

A V-4Cr-4Ti alloy is being studied as a possible structural material for first wall and blanket applications in Li-cooled fusion energy systems. Oremet Wah Chang of Albany, Oregon (OWCA), have produced a 500-kg heat (OWCA Heat 832665), for the United States Department of Energy (DoE)-funded Fusion Materials Sciences Program and a 1200-kg heat (OWCA Heat 832864) for General Atomics (GA), San Diego, California. A 30-kg heat (NIFS-HEAT-1) and a 166-kg. heat (NIFS-HEAT-2) with lower oxygen and nitrogen contents have been produced in Japan for the National Institute for Fusion Sciences. Thin-walled tubing of these alloys is needed to fabricate pressurized tube specimens for making creep property measurements in vacuum, liquid lithium, and neutron irradiation environments.

2.0 GENERAL INFORMATION

This specification establishes the requirements for the fabrication and heat treatment of thin-wall tubing to be used in the fabrication of specimens for irradiation creep experiments for the DoE Fusion Materials Sciences Program. Any additions or deletions to this specification shall be specified in applicable Oak Ridge National Laboratory (ORNL) service requests, memoranda, and/or drawings, and/or ORNL purchase orders/attachments. Should any conflicts develop between this specification and other technical documents referenced for the procurements or services indicated herein, this specification shall control. All such conflicts shall be resolved with the assigned ORNL representative before implementation. Any deviations to this specification by ORNL service organizations and/or outside contractors performing processes identified herein shall not be initiated without approval in writing by the assigned ORNL representative.

3.0 FABRICATION REQUIREMENTS

3.1 DEFINITION OF RESPONSIBILITIES

The assigned ORNL representative shall provide the raw material stock for fabricating the tubing. and provide certification of the chemistry of the raw material stock and any available fabrication history relative to same.

The assigned ORNL representative shall be responsible for the oversight of all processing steps in the fabrication of the tubing including characterization and preparation of the raw material stock, machining and dimensioning of the starting material (tube blank) for the initial tube fabrication, and fabrication, heat treatment, and dimensioning of the tubing .The ORNL representative shall also be responsible for the oversight of the characterization of the tubing at the designated hold points during processing and shall identify those ORNL service organizations or outside contractors who will be assigned to perform the characterization.

.

3.2 FABRICATION PROCEDURES

The specific procedures and requirements for the processing of tubing covered under this specification are defined in Section 5.0

3.3 MATERIAL CONTAMINATION PRECAUTIONS

The organizations identified herein for performing the various process steps in the fabrication of the tubing shall use necessary precautions to prevent exposure of the tubing material, during any process step, to any of the following:

The material shall not be exposed to any chlorine- or sulfur-bearing liquid, vapor, or grease during processing unless deemed specifically required for successful tubing fabrication, and which is known or can be shown to be removable by the cleaning processes designated herein prior to exposure to elevated temperatures (i.e. vacuum annealing or vacuum bakeout). The chemical nature of such required liquids, vapors, and/or greases with respect to the specific chemical species indicated herein shall be identified by the outside contractor prior to performing the work, and their specific use shall require approval by the assigned ORNL representative.

Except during tube drawing, expansion, and reduction (and mandrel removal) operations, the material shall not be allowed to come in contact with any metallic or non-metallic surface which may allow foreign particles of same to become embedded in its surface.

4.0 QUALITY ASSURANCE PROCEDURES

4.1 INSPECTION RIGHTS

The assigned ORNL representative shall have the right, during the working hours of the organization or outside contractor performing the work element, to witness inspections, perform product inspections, witness work operations in progress, and review quality documentation and records pertaining to the work indicated herein.

4.2 NOTIFICATION POINTS

Notification points shall be as a minimum the start of all processes designated in Section 5.0 Each notification point shall be witnessed at the option of the assigned ORNL representative who will be given two (2) working days advance notice. The outside contractor performing the work element shall not be required to hold up work once proper notification has been received by the assigned ORNL representative.

4.3 SAMPLING AND HOLD POINTS

At the end of each annealing cycle samples of tubing, to be specified by the ORNL representative, will be removed and placed in an archive. Samples will also be removed for chemical analysis and metallographic examination at ORNL. At the discretion of the ORNL representative, a hold time may be imposed until the results of chemical and microstructural analysis have been evaluated.

4.4 RECORDS MAINTENANCE

Outside contractors performing any of the work elements indicated herein shall submit inspection documentation, time-temperature and time-vacuum furnace charts, and other operations documentation and reports for inspection by the ORNL representative

5.0 FABRICATION PROCEDURES

5.1 CHARACTERIZATION OF THE RAW MATERIAL STOCK

The raw material stock to be used for fabrication of the tubing will be sections of cold–rolled plate measuring approximately 1.1 inches square and approximately 9 inches long. ORNL will provide material for three (3) sections of the US heat no.832665 and one (1) section of the Japanese heat NIFS-HEAT-2. The initial plate material will be characterized at ORNL in terms of optical microstructure, hardness and chemical analysis

5.2. MANUFACTURE OF TUBE BLANKS

The raw material stock shall be prepared for fabrication into material suitable for tubing (tube blank) as follows:

1) The raw material stock for the tubing shall be initially shaped and final machined using conventional machining methods to produce a tube blank (thick-walled tube) of dimensions suitable for processing into tubing of the required outside diameter (O.D.) and wall thickness. The method of machining and dimensions (O.D., wall thickness, and length) of the tube blank shall be agreed upon by the assigned ORNL representative and the vendor contracted to perform the processing of the tubing,. After machining, the finished tube blank shall be measured to record all physical dimensions and to determine its conformance to the specified dimensions. All measurements of these physical dimensions shall be documented. The finished tube blank shall be degreased in a solvent suitable

for removing visible traces of cutting debris and lubricants, and placed in a clean, dry, appropriately identified plastic bag.

2) The tube blank shall be cleaned in accordance with the procedure described in Section 5.3 and then annealed in accordance with the procedure described in Section 5.4 .

5.3 CLEANING TUBE BLANKS AND DRAWN TUBING

The starting material for fabrication of the tubing (tube blank) and subsequent as-fabricated tubing shall be cleaned prior to tube fabrication processes (drawing/expansion/reduction). Cleaning of the finished tube blank and as-fabricated tubing shall consist of solvent pre-cleaning, cleaning in a series of aqueous and solvent baths, and acid cleaning/etching (pickling), and shall be performed as follows:

5.3.1 SOLVENT CLEANING

The raw material stock for fabrication of tubing and as-fabricated tubing shall be solvent cleaned as follows:

1) Pre-clean the outer surfaces of the material to remove visual traces of any machining oils, marking ink, machining debris, etc., using a bristle brush or cloth and any suitable water-based or organic solvent cleaner (acetone, alcohol, etc.). Pre-clean the inner surfaces of the material to remove visual traces of any machining oils, machining debris, etc. by pulling or pushing multiple clean white solvent-soaked cotton or linen cloths through the internal bore of the material.

2) Clean external and internal surfaces of the material by immersion for a minimum of 5 minutes in each of the following series of solvent baths to remove all residues. Air dry material by placing on laboratory tissue (Kimwipes or equivalent) after each cleaning step. Verify removal of residues by surface wiping with a clean laboratory tissue moistened with ethyl alcohol.

   a) Bath 1 – Aqueous commercial cleaner (at >120ºF) [Alconox]

   b) Bath 2 - acetone (room temperature)

   c) Bath 3 - ethyl alcohol (room temperature)

3) If material has been saw cut (bandsaw, abrasive wheel, etc.), and saw-cut surfaces contain burrs, remove burrs by abrasive grinding saw-cut edges with #240 grit SiC paper (new).

4) If material has been electric discharge machined, abrasive grind machined surfaces with 600 grit SiC paper (new) to remove any discoloration and all evidence (by visual exam using 10X magnifier) of any brass residue from possible electrode interaction during discharge machining. Grind off a minimum of 0.0002 in. of surface material.

5) Wipe ground surfaces of material with ethyl alcohol-moistened tissue to remove all visual evidence of surface grinding debris. For tubular product forms, clean I.D. of tube by pulling or pushing ethyl alcohol-moistened cotton or linen cloth through tube to remove all visual evidence of surface grinding debris. Ultrasonically clean material in ethyl alcohol at room temperature. Air dry material on laboratory tissue (Kimwipes or equivalent). Handle cleaned material only with clean un-powdered rubber gloves or clean cotton or linen gloves.

6) Unless material is to proceed immediately through another process, e.g., pickling, annealing, etc., wrap material in laboratory tissue (Kimwipes or

equivalent) and place in a clean plastic bag appropriately identified as to sample identification, alloy designation, and material condition.

5.3.2 ACID CLEANING (PICKLING)

As soon as practical after solvent cleaning, all materials shall be acid cleaned as follows

1) Acid* clean/etch (pickle) material (maximum removal of 0.0005 in. of thickness) by immersion with agitation in a room temperature acid solution of 50 vol% deionized water - 30 vol% nitric acid - 20 vol% hydrofluoric acid for 30 seconds, followed by multiple rinses in deionized water at room temperature. Handle material with plastic tongs or by hand with clean un-powdered rubber gloves.

2) Wipe cleaned/etched and water-rinsed surfaces of material while still wet with deionized water-moistened cotton or linen cloth to remove all visual evidence of residue from acid cleaning/etching. For tubular product forms, clean I.D. of tube while still wet by pulling or pushing deionized water-moistened cotton or linen cloth through tube to remove all visual evidence of residue. For electric discharge machined materials remove all evidence of brass residue from prior machining (verify by visual exam using 10X magnifier). Ultrasonically clean material in ethyl alcohol at room temperature. Air dry material on laboratory tissue (Kimwipes or equivalent). Use clean un-powdered rubber gloves or clean cotton or linen gloves during etching/cleaning/wiping/handling of material. For electric discharge machined materials, repeat cleaning/etching/ rinsing/wiping procedure one additional time if visual evidence of brass residue persists. If repeat procedure is unsuccessful, hold at this point for review by the assigned ORNL representative.

3) Unless material is to proceed immediately through another process, e.g., vacuum annealing, etc., wrap fully-rinsed and dried material in laboratory tissue (Kimwipes or equivalent) and place in a clean plastic bag appropriately identified as to sample identification, alloy designation, and material condition.

**Caution: The acid etchant will generate some heat during its exposure to the material, and may cause severe burns from contact with unprotected tissue. A face shield and acid-resistant gloves should therefore be used at all times during handling of the acid and acid container(s).*

5.4 HEAT TREATMENT OF THE TUBE BLANK AND DRAWN TUBING

As soon as possible after cleaning as described above ,vacuum annealing should be carried out as follows:

1) Bake out vacuum furnace at a temperature at least 100ºC higher than the specified annealing temperature [see 4) below], and not more than 24 hours prior to the anneal.
2) Prior to annealing, wrap or shroud the material in a clean tantalum foil blanket such that no line of sight exists between the components of the vacuum furnace and any surface of the material to be annealed. Tantalum foil which has been previously used to anneal material in accordance with Section 5.4 or which has been baked out during or in a manner similar to the furnace bakeout step indicated in 1) above shall be acceptable. Support the material in the furnace to prevent distortion, or bowing. The material may be placed in the furnace either horizontally, vertically, or at an angle as long as proper support is provided.

3) Vacuum anneal the material using the following procedure:

a) Heat the material from room temperature to 525ºC at a rate commensurate with maintaining a vacuum of 2 x 10^{-5} torr or better. Hold at 525 ± 10ºC for 1 hour.

b) Heat the material from 525ºC to 1000ºC in a vacuum of 2 x 10^{-5} torr or better, and at a rate of 400ºC - 600ºC/hour. Hold at 1000 ± 10ºC for 1 hour.

c) Cool material to 50ºC in a vacuum of 2 x 10^{-5} torr or better, and at a rate of >1000ºC/hour (nominal). Do not remove material until temperature of material is <50ºC. Use clean cotton or linen gloves during handling.

d) Monitor the temperature and temperature uniformity of the material using a minimum of two (2) thermocouples (e.g., Type K, with Inconel 600 sheath) attached directly to the material or inserted into the tantalum-wrapped material/ package. If the uniformity of the furnace has been previously established and accepted by the assigned ORNL representative, only one (1) thermocouple attached to the material or inserted into the Ta-wrapped material/package is required.

5) Record temperature and vacuum level readings in a continuous fashion, or manually every 10 minutes, during heat up, elevated-temperature holds, and cool down of the material.

6) After removal of the wrapped material from the vacuum furnace, unwrap material. Wrap material in laboratory tissue (Kimwipes or equivalent) and place in a clean plastic bag appropriately identified as to sample identification, alloy designation, and material condition. Place tantalum foil in a clean plastic bag and provide to the assigned ORNL representative.

5.5 TUBING FABRICATION (TUBE DRAWING/EXPANSION/REDUCTION)

Tube blank material prepared in accordance with Sections 5.1 through 5.5 shall be fabricated into tubing of the required outside diameter (O.D.) and wall thickness. The dimensions of the tube blank required to produce the required finished tubing dimensions shall be agreed upon by the assigned ORNL representative and the vendor contracted to perform the fabrication of the tubing. The tube blank and finished tubing dimensions shall be specified in the ORNL purchase order for the processing of the tubing. Fabrication of the tubing shall be performed as follows:

1) The finished, cleaned, and annealed tube blank shall be fabricated into tubing by drawing at room temperature in accordance with the dimensions (O.D. and wall thickness) specified in the ORNL purchase order for the processing of the tubing. A step-by-step fabrication schedule to be followed in drawing the tubing, including die and mandrel sizes for each draw pass, shall be provided to the assigned ORNL representative by the outside contractor processing the tubing prior to the initiation of processing. The tolerance on the tube O.D. shall be ± 0.001 in. and the tolerance on the tube I.D. shall be ± 0.001ins.Finished tube ovality shall not exceed 0.002ins and eccentricity shall not exceed 0.0005ins. The fabrication of the finished tubing will require several draw cycles, of several draw passes each, and several intermediate cleanings/anneals.

2) Prior to initial drawing, one end of the tube blank shall be swaged (pointed) to facilitate entry into the drawing die. The swaged length (point) shall be minimized in order to facilitate the fabrication of the longest length of tubing possible, but shall not be made so short as to compromise the fabrication of the tubing itself. The tube blank shall be reduced through successively smaller dies until both its O.D. and wall thickness reach the final tube dimensions specified, or

reduced through successively smaller dies until its wall thickness reaches the specified wall thickness, and then expanded or reduced in diameter to the specified O.D. The reduction in area for each drawing pass shall be limited to 15 ± 2%. The total reduction for the first draw cycle shall be limited to 30 ± 5%; the total reductions for succeeding draw cycles up until the last two draw cycles shall not exceed 45%; and the total reductions for the last two draw cycles shall be limited to 30 ± 5%. The outside diameter, wall thickness, length, and hardness (near trailing end) of the tubing shall be measured and recorded after each draw (pass) to assess the progress of the processing. A report of the tubing dimensions and hardness values recorded after each pass shall be provided with the finished tubing.

3) After each draw cycle, and prior to cleaning/annealing, two (2) cross-sections (each ~1/4 in. in length) shall be taken from the trailing end (non-pointed end) of the tubing) using conventional cutting methods (abrasive wheel, bandsaw, EDM, etc). After cutting, the tubing sections shall be degreased in a solvent suitable for removing visible traces of machining debris and lubricants, and placed in a clean, dry, plastic bag appropriately identified as to sample identification, alloy designation, and material condition. The tube blank sections shall be provided to the assigned ORNL representative.

4) The remaining tubing shall be cleaned and vacuum annealed in accordance with the procedures described in Sections 5.3 and 5.4.

5) The step-by-step fabrication schedule may be necessarily changed or altered by the tube processing vendor during the actual fabrication process depending on the actual final dimensions of the starting tube blank, possible failure of the material to achieve the specified deformation levels, etc. Such changes shall require approval in writing by the ORNL representative prior to initiation.

6.0 PACKAGING AND SHIPMENT

6.1 IDENTIFICATION AND MARKING

The finished tubing shall be marked with the supplier's identification, lot number, nominal thickness or diameter and purchase order number. Markings shall be made on one surface of each product piece. Characters shall be of such size as to be clearly legible, and shall be applied using a suitable marking fluid capable of being removed with conventional cleaning solutions without excessive rubbing. The markings or their method of removal shall have no deleterious effect on the product or its intended performance, and shall be sufficiently stable to withstand normal handling.

6.2 PACKAGING AND PACKAGE MARKING

The product shall be prepared for shipment in accordance with commercial practice and in compliance with applicable rules and regulations pertaining to the handling, packaging, and transportation of the product to ensure carrier acceptance and safe delivery. Each piece of product form shall be individually wrapped in a protective material to preclude exposure to atmospheric conditions and/or improper handling which could inadvertently damage the material. Packaging shall conform to carrier rules and regulations applicable to the mode of transportation utilized for shipment of the product.

7.0 ACKNOWLEDGEMENT

The supplier shall specifically mention this specification number and revision letter in all quotations and when acknowledging contracts or purchase orders.