Report No. EMI-1417

UC-25 Metallurgy and Ceramics (TID-4500, 15th Ed.)

Contract No. W-7405-eng-92

ŧ

`49

Ŧ

幕

DEVELOPMENT OF HIGH-STRENGTH NIOBIUM ALLOYS FOR ELEVATED-TEMPERATURE APPLICATIONS

by

John A. De Mastry Frederic R. Shober Ronald F. Dickerson

February 22, 1960

BATTELLE MEMORIAL INSTITUTE 505 King Avenue Columbus 1, Ohio

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

TABLE OF CONTENTS

۶

٩,

*9

8

ø

		<u>Paye</u>
ABSTRACT	a o	. 1
INTRODUCTION	• •	. 1
ALLO' PREPARATION	а о	. 2
ALLOY FABRICATION	e a	. 5
Forging of Alloys in the First Series	0 0 0 0	. 5 . 11
SHORT-TIME TENSILE PROPERTIES	• •	. 13
WELDING STUDY	6 B	. 13
CONCLUSIONS		. 21
RECOMMENDATIONS	a a	. 21
ACKNOWLEDGMENTS	a o	. 22

John A. De Mastry, Frederic R. Shober, and Ronald F. Dickerson

A study to improve the elevated-temperature strength of niobium by alloying has resulted not only in greatly improved strengths at 1200 and 1470 F but also in the development of improved fubrication techniques for these alloys.

The most important step in the fabrication procedure of niobium and niobium-base alloys is the initial breakdown of the cast structure. The cast structure of 1.84 u o chromium, 3.21 v o chromium, 4.33 w o zirconium, and 20.5 u o titanium-4.28 chromium alloys and unalloyed niobium was broken down by forging ingots (protected from oxidation by molybdenum cans) at 2550 F and rolling at 800 F. After the initial breakdown of the cast structure, the alloys were cold rolled to a total of 95 per cent reduction with no difficulty.

A second fabrication technique was employed for a second set of alloys. Unalloyed niobium and 1.29 w o chromium, 2.74 w o zirconium, 4.5 w o molybdenum, and 10 w o titanium-3 w o chromium alloys were forged and rolled at 1000 F to break down the cast structure and then cold rolled to 0.030-in. sheet. The sheet obtained by this technique showed moderate edge cracking.

Tensile tests on the cold-worked materials at 1200 and 1170 F indicate that chromium and zirconium are both potent strengtheners of niobium: the 1.84w 'o chromium allov had a 0.2 per cent offset vield strength of 107,000 psi at 1200 F and 69,000 psi at 1470 F, and the 1.33w 'o zirconium alloy had a 0.2 per cent offset vield strength of 69,000 psi at 1470 F.

Limited welding studies indicate that strong and reasonably ductile welds can be produced both by arc and spot welding.

INTRODUCTION

The need for an alternate cladding material capable of withstanding the high operating temperatures (1200 to 1470 F) of advanced designs of reactors of the EBR type has motivated a consideration of alloys of the refractory elements. The formation of the iron-uranium eutectic at 1340 F precludes employment of the Type 347 stainless steel cladding used in the EBR-I. The development of niobium alloys for the EDR-II appears attractive because niobium, which is already adequate in mechanical strength, thermal conductivity, and corrosion resistance to high-temperature sodium for the operating conditions of the EBR-I, can be improved markedly by alloying. The present investigation was undertaken to develop a niobium-base alloy which would be superior in strength to stainless steel at 1200 to 1500 F, be resistant to attack by 1500 F sodium, and be fabricable by standard techniques, and in which ductile welds could be produced.

Selection of alloying elements was based on their probable strengthening effect or on their ability to improve fabricability. Chromium, molybdenum, and zirconium were used as binary additions, and aluminum, chromium, tantalum, and titanium were used for ternary alloys. Except for titanium, all of these elements should strengthen niobium by solid-solution or age-hardening mechanisms. Titanium additions tend to improve the fabricability of alloys containing aluminum, chromium, and tantalum.

To insure that promising alloys would be readily fabricable, considerable attention was given to fabrication procedures, particularly the initial breakdown of the cast structure. Two series of alloys were prepared to study this problem. In the evaluation of fabricable material, elevated-temperature short-time tensile tests on sheet specimens were used to obtain strength data, while tension-shear and bend tests were employed to study the weldability of the alloys. Both spot and arc welding were investigated.

ALLOY PREPARATION

Two series of alloys were prepared by consumable-area melting for this study. The first series consisted of unalloyed niobium, six niobium alloys, and vanadium alloy. The compositions were as follows:

> Unalloyed niobium Niobium-1.84 w/o chromium Niobium-3.21 w/o chromium Niobium-4.33 w/o zirconium Niobium-9.95 w/o tantalum-3.31 w/o chromium Niobium-39.8 w/o titanium-10.6 w/o aluminum Niobium-20.5 w/o titanium-4.28 w/o chromium Vanadium-11.7 w/o titanium-2.6 w/o niobium.

The second series consisted of:

Unalloyed niobium Niobium-1. 29 w/o chromium Niobium-2. 74 w/o zirconium Niobium-4. 5 w/o molybdenum Niobium-10.0 w/o titanium-3.0 w/o chromium.

The melting procedure was identical for both series of alloys although they were prepared at different times.

Tables 1, 2, and 3 list results of chemical analyses for the major impurities in niobium, vanadium, and all alloying additions used as melting stock in alloy preparation. The niobium used was received in the form of sintered rod to which the desired alloying additions were welded. Chromium losses due to volatilization during melting were expected and chromium was overcharged 100 per cent. The vanadium-base alloy was prepared by forming a compact of niobium, titanium, and vanadium granules under pressure. The above-mentioned welded rods and pressed-compact electrodes were then consumably arc melted (argon atmosphere) into a water-cooled copper crucible.

Impurity Elements	Amount, ppm
Aluminum	20
Chromium	<20
Iron	<100
Manganese	<20
Molybdenum	<20
Nickel	20
Silicon	<100
Tantalum	630
Titanium	30
Tungsten	<300
Vanadium	<20
Zirconium	60
Carbon	37
Hydrogen	33
Nitrogen	235
Oxygen	200

TABLE 1. CHEMICAL ANALYSIS OF NIOBIUM USED FOR ALLOY PREPARATION

Ŗ

TABLE 2. VENDOR'S CHEMICAL ANALYSES OF VANADIUM USED IN ALLOY PREPARATION

Impurity Elements	Amount, ppm
Carbon	610
Hydrogen	160
Oxygen	500
Nitrogen	800

T	A 1	Chromium	dicated Additior Titanium ^(a)	Zirconium	· start and the second seco
Impurity	Aluminum	Chromium	11tanium(~)	Zirconium	Molybdenum
Aluminum	anti). 6500	4764 Mille	<300		and they
Carbon	620 Grai.	10	50	50	
Chlorine	ins en	6000 (Č00	800		2023 0000
Chromium	and spin		<50	مقتب محقت	200
Copper	20	100 Lan	<100	- 50	20
Hafnium	640 STR	the star	800 ipsi	<200	
Hydrogen	ama 1470	1000 AUTO	70	and soft	tore sum
Iron	10	2	<50	200	50
Magnesium	10		20	aana 1999	20
Manganese	angen Steph		200	2010 Ballio	mme Seel
Nitrogen	and and	100 488	20	60	985. CHA
Oxygen		15	190		
Silicon	10	<20	100		30
Tin	print 933	<20		65. 574	
Vanadium	au an	<3	200	gran, spine	
Zirconium	grap Antik	<20			1000 (100)

TABLE 3. CHEMICAL ANALYSES OF ALLOYING ADDITIONS FOR NIOBIUM- AND VANDIUM-BASE ALLOYS

(a) Titanium was supplied by U.S. Bureau of Mines.

ſ

After melting, the ingot was turned on a lathe, cut into quarters, welded into an electrode, and remelted. The final ingots weighed between 3/4 and 2 kg. After the second melt, the ingots were cropped and lathe turned to remove surface imperfections. All ingots appeared sound with the exception of the alloys containing titanium; these exhibited mild piping and poor surface quality, which was probably due to the high gas content of the titanium.

ALLOY FABRICATION

The initial breakdown of the cast structure is the key step in the fabrication of niobium-base alloys. If the coarse as-cast structure can be sufficiently refined without cracking, then many niobium-base alloys can be readily fabricated at room temperature. In this study, the cast structures were broken down at both elevated (2550 F) and low temperatures (1000 F). The first fabrication was accomplished by upset hammer forging after enclosing the alloys in molybdenum packs at 2550 F. All alloys of the first series were fabricated in this manner. Fabrication of a second set of alloys was accomplished by upset forging unclad ingots at 1000 F. All alloys of the second series were fabricated following the second method. For the sake of simplicity the fabrication of alloys in each series will be reviewed separately.

Forging of Alloys in the First Series

1

The first method of initial cast-structure breakdown consisted of upset forging of the alloys in molybdenum cans at 2550 F. The cans were needed to prevent contamination of the ingots by the atmosphere. The ingots were cropped and lathe turned to remove surface impurities prior to encapsulation. A top and bottom of molybdenum plate were welded onto the ends of the molybdenum can in a tank under a partial pressure of helium. Figure 1 shows a typical can assembly. The ingots were upset hammer forged at 2550 F with reductions of 10 per cent per pass in 15 to 20 sec in order to prevent excessive heat losses. All ingot assemblies were reheated between forging passes for 10 min. The ingots were forged only in their axial direction, which resulted in a final plate shape resembling a pancake. Results of fabrication are shown in Table 4. Edge views of the plates are shown in Figures 2 and 3. The end caps of the molybdenum container which can be seen adhering to the vanadium-base alloy in Figure 3 were not bonded and peeled off during rolling.

Sections were cut from these forged ingots for additional rolling studies and metallographic examination.

Microstructures of the hot-worked alloys were examined. The alloys exhibited clean microstructures. Figures 4, 5, and 6 show representative microstructures of the alloys being studied. The structures of the unalloyed niobium and niobium-1.84 w/o chromium alloys are quite similar. The niobium-20.5 w/o titanium-4.28 w/o chromium alloy appears to be recrystallized.



FIGURE 1. MOLYBDENUM CAN ASSEMBLY

Left side shows molybdenum lids 0.100 in. thick which were welded onto center tube to produce finished can at right.

Cold-Rolling Study of Alloys in the First Series

Small rectangular specimens of each alloy were cut from the forged slabs, machined, and rolled at room temperature. The niobium-3.21 w/o chromium, and niobium-9.95 w/o tantalum-3.31 w/o chromium specimens fractured after 5 per cent reduction. The remaining alloys were reduced 70 to 80 per cent with only minor edge cracking. After evaluating the above result, attempts were made to cold roll a portion of the forged slabs of all cold-fabricable alloys. Cold rolling of the larger size slabs was not satisfactory. The cold-rolled sheet exhibited severe edge cracking. The surface of the unalloyed niobium also showed a network of fine cracks. Two factors were considered as being potentially responsible for the apparent brittleness in these alloys, contamination by interstitial elements (especially oxygen) or insufficient breaking down of the cast structure during forging. Chemical analyses were performed on all alloys to determine if contamination had occurred. The results of these analyses are shown in Table 5. When compared with the analyses of the niobium melting stock it appears that only the unalloyed niobium absorbed more oxygen. This probably occurred during forging due to a crack in a weld or the molybdenum pack. This lack of evidence of contamination of the alloys indicated that perhaps the as-cast structures were not sufficiently broken down during the initial forging at 2550 F, and further reduction was attempted at 800 F.

TABLE 4.RESULTS OF UPSET FORGING OF NIOBIUM-BASE ALLOYS IN
MOLYBDENUM PACKS AT 2550 F

Analyzed Composition, w/o	Reduction(a), per cent	Remarks
Niobium	84	No edge cracks
Niobium-1.84 chromium	76	Slight edge cracking
Niobium-3.21 chromium	69	Slight edge cracking
Niobium-4.33 zirconium	76	No edge cracks
Niobium-9.95 tantalum-3.31 chromium	68	Severe edge cracks
Niobium-39.8 titanium-10.6 aluminum	84	No edge cracks
Niobium-20.5 titanium-4.28 chromium	79	No edge cracks
Vanadium-11.7 titanium-2.6 niobium	79	No edge cracks

(a) Based on reductions from ingot size.

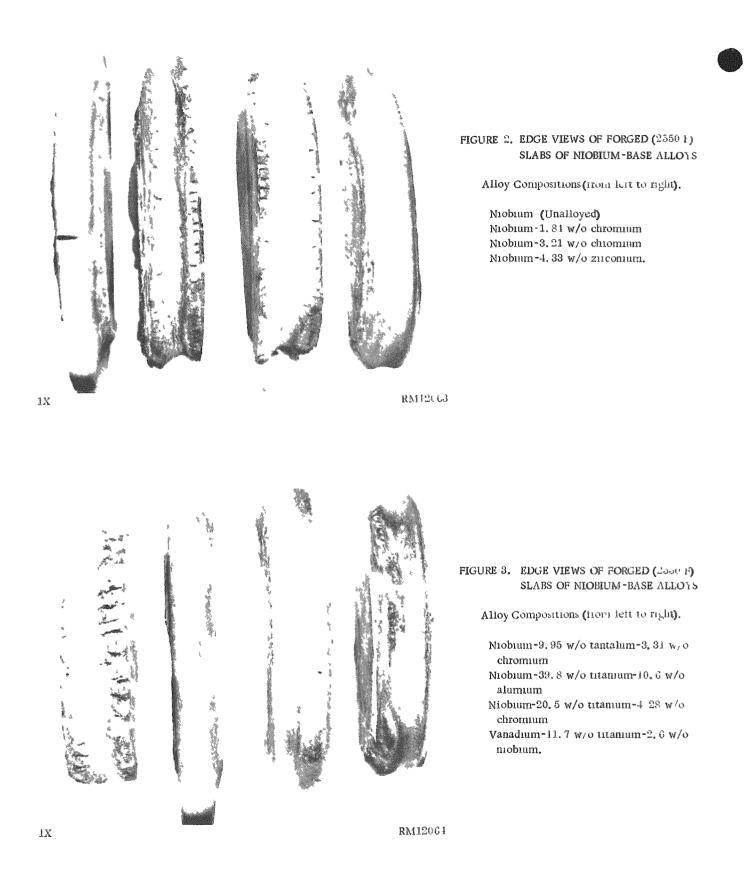
1

*

4

.

Ъ



ş

8

FIGURE 4. NIOBIUM (UNALLOYED) HOT-WORKED STRUCTURE SHOWING STRESS-FREE GRAINS AFTER FORGING AT 2550 F

9

RM12428

혛

RM12429

FIGURE 5. SINGLE-PHASE STRUCTURE ON MOBIUM-1.84 w/o CHROMIUM AFTER FORCING AT 2550 F

FIGURE 6. MICROSTRUCTURE OF NIOBIUM-20.5 w/o TITANIUM-4.28 w/o CHROMIUM AFTER FORGING AT 2550 F

This structure appears to be recrystallized.

100X

ъ

-

R.

250X

 $250 \mathrm{X}$

ş

TABLE 5.	RESULTS OF CHEMICAL ANALYSES OF ALLOYS IN THE FIRST
	SERIES AFTER FORGING AT 2550 F AND COLD ROLLING

	Impurity Analysis, ppm				
Analyzed Composition, w/o	Oxygen	Hydrogen	Carbon	Nitrogen	
Niobium (unmelted stock)	200	20	250	300	
Niobium (unalloyed)	820	5	300	110	
Niobium-1.84 chromium	90	43	120	140	
Niobium-3.21 chromium	60	82	110	150	
Niobium-4.33 zirconium	392	01	110	220	
Niobium-9.95 tantalum-3.31 chromium	300	92	80	130	
Niobium-39.8 titanium-10.6 aluminum	313	146	80	120	
Niobium-20.5 titanium-4.28 chromium	350	54	120	110	
Vanadium (unmelted stock)	800	160	o10	500	
Vanadium-11.7 titanium-2.6 niobium	680	98	330	400	

ſ

Warm-Rolling Studies of Alloys in the First Series

Assuming that further breakdown of the as-cast structure was needed, all alloys were rolled from a furnace (argon atmosphere) at 800 F. These specimens, approximately 1 in. wide, had been cut from the as-forged slabs. All alloys except the niobium-9.95 w/o tantalum-3.31 w/o chromium and the niobium-39.8 w/o titanium-10.6 w/o aluminum were reduced about 70 per cent. The sheet fabricated at 800 F was sectioned into three separate pieces, and a fabrication study was made at room temperature. One piece of each alloy was cold rolled with no further treatment. This rolling was successful, but the sheet exhibited edge cracks which extended 0.062 to 0.125 in. from the edge toward the center of the sheet. The surface also had occasional remnants of cracks formed during forging or rolling. A second piece was belt sanded to remove surface and edge cracks produced by forging at 2550 F and rolling at 300 F. This piece was cold rolled and yielded sheet with only occasional edge cracks, extending less than 0.062 in. from the edge to the center, and rare surface defects. A third piece of sheet was belt sanded and vacuum annealed for 1 hr at 2550 F and furnace cooled. This technique produced sheet with smooth edges and a defect-free surface after cold rolling. In Figure 7 examples of the difference in appearance of sheet which was belt sanded and that which was belt sanded and annealed prior to cold rolling are shown.

The sheet which had been belt sanded and annealed was subsequently reduced to 0.010-in. material (without intermediate annealing) for welding studies.

Attempts to roll the niobium-9.95 w/o tantalum-3.31 w/o chromium and niobium-39.8 w/o titanium-10.6 w/o aluminum alloys were unsuccessful at 2000 F (material clad in stainless steel). No further attempts to fabricate these alloys were made.

The fabrication studies conducted with the alloys in the first series indicated that forging at 2550 F in a molybdenum can and subsequent rolling at 800 F in air breaks down the cast structure of niobium alloys studied (with the exception of the niobium-9.95 w/o tantalum-3.31 w/o chromium and niobium-39.8 w/o titanium-10.6 w/o aluminum alloys) sufficiently to allow cold rolling. The quality of the cold-rolled sheet can be improved if the sheet rolled at 800 F is surface conditioned and annealed.

Fabrication of Alloys in the Second Series

Since niobium can be worked at room temperature, it is reasonable to assume that certain niobium alloys may also be workable at room temperature or temperatures close to room temperature. Therefore, additional ingots were prepared and fabricated by upset forging and rolling at 1000 F.

This portion of the program was designed primarily to study the fabrication characteristics of niobium-base alloys at relatively low temperatures. The ingots were not cropped or lathe turned prior to fabrication; therefore, the sides of the ingots were rough which would be reflected as edge cracks in the forged slabs. The unalloyed niobium, niobium-1.29 w/o chromium, niobium-2.74 w/o zirconium, niobium-4.5 w/o molybdenum, and niobium-10.0 w/o titanium-3.0 w/o chromium were upset forged at 1000 F in air and, after about 40 per cent reduction, rolled at 1000 F. After warm

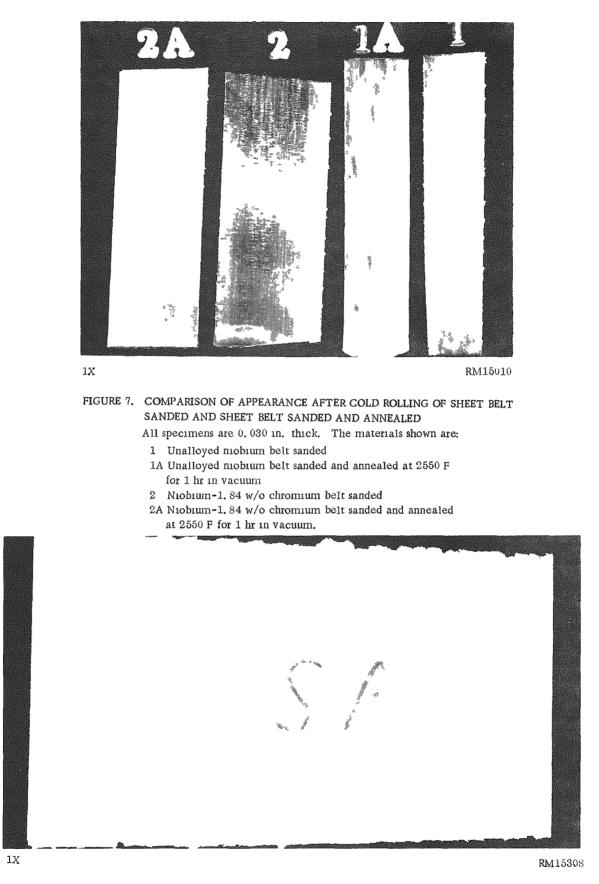


FIGURE 8. PORTION OF SHEET OF UNALLOYED NIOBIUM WHICH WAS REDUCED 95 PER CENT BY FORGING AND ROLLING AT 1000 F AND ROLLING AT 75 F rolling the sheet was vapor blasted to remove a slight oxide film and cold rolled to a total reduction of 95 per cent from the ingot. Table 6 shows the results of fabrication of these alloys. Figures 8, 9, and 10 show portions of the cold-rolled sheet. While edge cracks do exist, they are due to the poor surfaces of the ingots prior to fabrication.

It is apparent from this brief investigation that it is possible to break down the as-cast structure of certain niobium-base alloys by forging and rolling at 1000 F. The largest ingots fabricated at 1000 F weighed 2 lb, indicating that larger scale ingots can probably be handled in similar manner. Breakdown of the cast structure of these niobium alloys at 1000 F is a major accomplishment which could have far-reaching results in the development of niobium-base alloys. In the past it has always been believed that elevated temperatures were necessary to break down the cast structures of niobium alloys. This study has demonstrated that this is not true for the alloys discussed above. It is hoped that these results will stimulate a thorough investigation of low-temperature fabrication of niobium-base alloys.

SHORT-TIME TENSILE PROPERTIES

Tensile tests at 1200 and 1470 F were performed on sheet specimens cut parallel with the rolling direction of the sheet. The strain rate for all specimens in the elastic region was 0.002 in. per in. per min, which was increased to 0.013 in. per in. per min beyond the proportional limit. The specimens were tested in a vacuum of less than 10 μ , and showed no visual signs of oxygen contamination after testing. The results of testing of alloys from both series are shown in Tables 7 and 8.

All alloys studied showed greatly increased strength over that of unalloyed niobium at 1200 and 1470 F. The best strengthener at 1200 F was chromium, while both chromium and zirconium were extremely potent strengtheners at 1470 F.

WELDING STUDY

Weldability is an important consideration in the development of material for cladding applications, since seam-welded tubing or welded end closures are used with most core-cladding combinations.

In this study, three criteria were used to determine the weldability of the niobiumbase alloys being developed. The first of these methods consisted of spot welding two sheets of alloy together and then qualitatively evaluating the strength of the bond by peeling the sheet apart with pliers. A second method consisted of spot welding two sheets together and pulling them apart in a tensile machine. In the third method of evaluation, two sheets of material butt welded together longitudinally were bent across the weld.

		Fabric	ation at 1000 F		Fab	rication at 75 F
Alloy Composition, w/o	Upset Forging Reduction ^(a) , per cent	Remarks	Rolling Reduction ^(a) , per cent	Remarks	Rolling Reduction ^(a) , per cent	Remarks
Unalloyed niobium	70	Slight edge cracking	75	No additional edge cracking	80	No additional edge cracking
Niobium-1.29 chromium	50	Severe edge cracking	75	Edge cracks did not open	80	No additional edge cracking; cracks already present did not open
Niobium-2.74 zirconium	60	Moderate edge cracking	75	Edge cracks remained moderate	80	No additional cracking
Niobium-4.5 molybdenum	50	Severe edge cracking	75	Cracks did not open up	80	No additional cracking
Niobium-10.0 titanium-3.0 chromium	50	Moderate edge cracking	75	No propagation of cracks	80	No additional cracking

-

1

TABLE 6. RESULTS OF SUCCESSIVE STAGES OF FABRICATION OF NIOBIUM-BASE ALLOYS AT 1000 F AND 75 F

(a) Reductions are based on each succeeding reduction. Total reduction of all alloys is above 95 per cent.

4



1



FIGURE 9. PORTION OF SHEET OF NIOBIUM-1.29 w/o CHROMIUM ALLOY REDUCED 95 PER CENT BY FORGING AND ROLLING AT 1000 F AND ROLLING AT 75 F

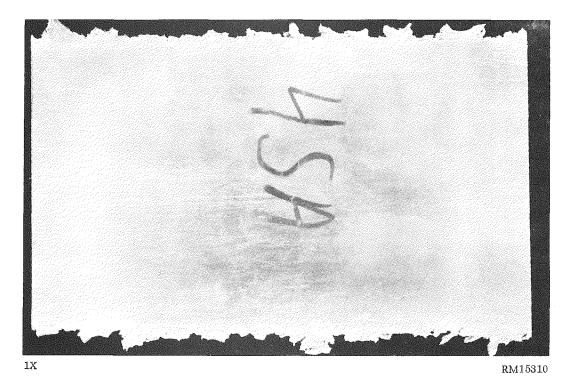


FIGURE 10. PORTION OF SHEET OF NIOBIUM -2. 74 w/o ZIRCONIUM ALLOY REDUCED 95 PER CENT BY FORGING AND ROLLING AT 1000 F AND ROLLING AT 75 F

ð,

8

Alloy Composition, w/o	0.2 Per Cent Offset Yield Strength, psi	Tensile Strength, psi	Elongation in l In., per cent	Reduction in Area, per cent
Niobium (unalloyed)	54,600	62,500	3	ganamar, ang
Niobium-1.29 chromium	95, 300	102,000	3	FEES Gaulo
Niobium-1.84 chromium	107,000	115,000	4	5
Niobium-3.21 chromium	120, 400	129,500	4	post stas
Niobium-2.74 zirconium	94, 100	96, 100	2	
Niobium-4.33 zirconium	-	82,400	4	7
Niobium-20.5 titanium-4.28 chromium	75,500	92,200	11	25
Vanadium-11.7 titanium-2.6 niobium	41,000	51,200	46	71

TABLE 7. TENSILE DATA FOR NIOBIUM- AND VANADIUM-BASE ALLOYSIN AS-WROUGHT CONDITION AT 1200 F

ſ

1

TABLE 8. TENSILE DATA FOR NIOBIUM- AND VANADIUM-BASE ALLOYS IN AS-WROUGHT CONDITION AT 1470 F

Alloy Composition, w/o	0.2 Per Cent Offset Yield Strength, psi	Tensile Strength, psi	Elongation in 1 In., per cent	Reduction in Area, per cent
Niobium (unalloyed)	39,900	46,100		989 688
Niobium-1.29 chromium	63,000	73, 100	11	1200 480
Niobium-1.84 chromium	68,700	81,600	14	37
Niobium-2.74 zirconium	79,000	85,800	4	3
Niobium-4.33 zirconium	69,000	76, 400	4	3
Niobium-20.5 titanium-4.28 chromium	30, 800	39, 700	40	100
Vanadium-11.7 titanium-2.6 niobium	34, 900	45,700	35	67

All welds were made on 0.010-in.-thick sheet. The welding study was designed to determine if sound, ductile welds could be made. No attempt to attach relative strengths to the welds was made, since no attempt was made to develop optimum welding conditions. All welds were made on cold-rolled material and were not annealed after welding; therefore, all welds were tested with the sheet in the cold-worked and the welds in the unannealed condition. The spot welds were made using a 50-kva singlephase a-c spot welder with synchronous controls. Resistance-welding electrodes were RW MA Group A Class 1 alloy, having a 10-in. face radius and a 5/8-in. limiting diameter. The welding conditions for making the spot welds are shown in Table 9.

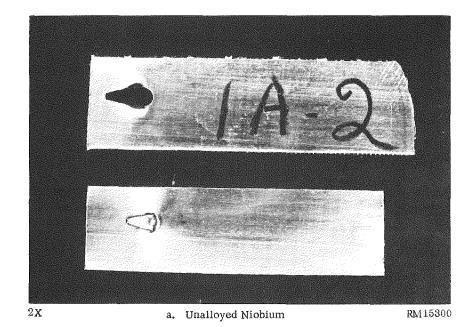
TABLE 9. WELDING CONDITIONS FOR SPOT WELDS

мадару жан часка алын часка кака кака кака кака кака кака как	n and a second secon A second
Electrode Force	200 1ь
Weld Time	4 cycles (60 cps)
Weld Current	4000 to 5000 amp (estimate)

The spot welds were evaluated for ductility by both peel and tension-shear tests. Failure of all spot welds occurred by tearing of the spot weldment out of one sheet. The failure occurred in the heat-affected metal surrounding the weldment, indicating that the welds possess some degree of ductility. The pullout-button diameters ranged from 0.078 to 0.093 in. in diameter. Figure 11 shows typical failures of the tension-shear test specimens.

In order to further evaluate the weldability of these alloys, two pieces 0.010 by 1/2 by 3 in. were arc welded along their 3-in. length. The welds were made without filler-metal additions, using automatic inert-gas tungsten-electrode arc-welding equipment in the open atmosphere. Weld joints were prepared by first forming a 0.020-in. flange along the edges that were to be welded. The flanges on each sheet provided filler metal for the weld. Heat was removed from the 0.010-in. sheet by a copper cooling fixture. Argon gas was used for shielding. The welding currents ranged from 30 to 45 amp. The welds were evaluated by both visual and guided-bend test procedures. In all cases, the appearance of the weld was satisfactory, and as shown by the bend tests, the welds were reasonably ductile since the specimens were not annealed after welding. The results of testing of these arc-welded specimens are shown in Table 10. Figure 12 shows typical weld specimens before bending, and Figure 13 shows specimens after the bend test.

It appears that welds with reasonable ductility can be produced in the cold-worked material. Annealing should make the welds even more ductile. No attempt was made to optimize the welding conditions since this study was intended only as a survey of the welding characteristics of the alloys.



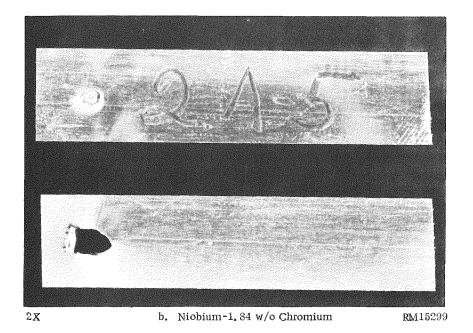


FIGURE 11. EXAMPLES OF TYPICAL FAILURES IN SPOT-WELDED TENSION-SHEAR TEST SPECIMENS

Tearing of the test coupons indicates ductility in the weldment. The specimens were not annealed after welding.

TABLE 10. RESULTS OF BEND TESTING OF ARC-WELDED NIOBIUM ALLOYS^(a)

Alloy Composition, w/o	Minimum Bend Radius ^(b) , 7
Niobium (unalloyed)	18
Niobium-1.84 chromium	18
Niobium-3.21 chromium	9
Niobium-4.33 zirconium	18
Niobium-20.5 titanium-4.28 chromium	1
Vanadium-11.7 titanium-2.6 niobium	12
Niobium-4.5 molybdenum	12

(a) All specimens were sound prior to testing.

(b) Minimum bend radius, T, is calculated as T = R/t,

where

٠

١

*

\$

٤

R = Radius of ram

t = Thickness of weld.

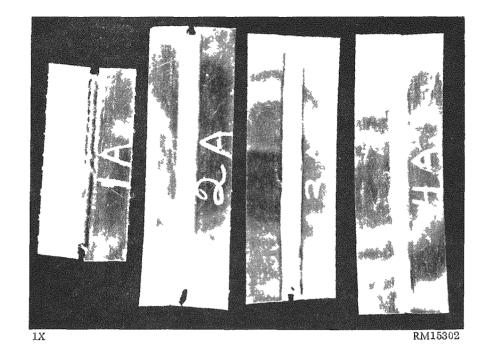
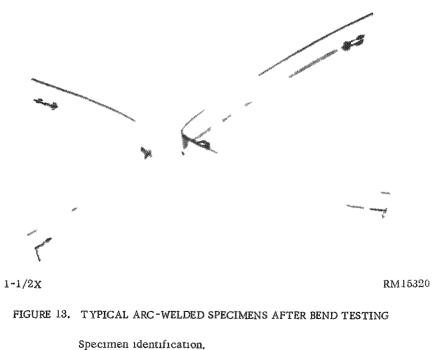


FIGURE 12. TYPICAL ARC-WELDED SPECIMENS BEFORE TESTING

Specimen identification

- 1A Niobium (unalloyed)
- 2A Niobium-i 84 w/o chromium
- 3A Niobium-3, 21 w/o chromium
- łA Niobium-4, 33 w/o zirconium



Left Niobium (unalloyed) Right Niobium-20.5 w/o titanium-4 28 w/o chromium.

CONCLUSIONS

As a result of the research discussed in detail in the preceding sections, it can be concluded generally that niobium-base alloys can be developed which exhibit very interesting properties at 1200 and 1470 F. In addition, the technology of niobium has been advanced by the development of techniques which allow niobium-base alloys to be fabricated at relatively low temperatures and, consequently, without the need for protection against oxidation during fabrication.

Specific conclusions reached as a result of the study are listed below.

٩.

- Of the alloys studied, the niobium-3.21 w/o chromium alloy exhibited the best strength properties at 1200 F (0.2 per cent offset yield strength of about 120,000 psi). In general, however, very large increases in strength at elevated temperature were obtained by the addition of moderate amounts of chromium and zirconium.
- (2) Although all alloys were fabricable, the niobium-zirconium alloys appeared to possess the best fabrication characteristics.
- (3) No difficulties in producing ductile welds are expected in any of the alloys studied.
- (4) The niobium alloys studied can be cold rolled with no difficulties once the as-cast structure has been broken down.
- (5) It is possible to break down the as-cast structures of many niobiumbase alloys by hammer forging at temperatures as low as 1000 F with only moderate edge cracking.

RECOMMENDATIONS

The excellent high-temperature tensile-strength data of these alloys should be supplemented with elevated-temperature creep data. It is expected that the minimum creep rate of these alloys will be extremely low.

It is strongly recommended that an additional study be made of the lowtemperature breakdown of cast structures of niobium-base alloys. The influence of ingot size should be studied, the effect of various alloying additions and amounts to which this technique is applicable should be ascertained, and the limiting oxygen content of the alloys which can be fabricated in this manner determined.

This study was conducted on cold-worked material; therefore, it is necessary to determine the stability of the cold-worked material at elevated temperatures and the effects of long-time anneals at temperatures below the recrystallization temperature on tensile properties.

ACKNOWLEDGMENTS

This research was conducted in support of the EBR program at the Argonne National Laboratory. Mr. Karl Smith coordinated the work for ANL. His assistance is gratefully acknowledged.

The authors wish to express appreciation to Battelle staff members John S. DiPesa and Ellis L. Foster, Jr., for suggestions and assistance given in the fabrication studies.

JAD:FRS:RFD/mln