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June 17, 1963
DMIC Memorandum 169

WELDABILITY STUDIES OF THREE COMMERCIAL COLUMBIUM-BASE ALLOYS

DEFENSE METALS INFORMATION CENTER
BATTELLE MEMORIAL INSTITUTE
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2. To supplement established Service activities in providing technical advisory services to producers, melters, and fabricators of the above materials, and to designers and fabricators of military equipment containing these materials.

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4. On assignment, to conduct surveys, or laboratory research investigations, mainly of a short-range nature, as required, to ascertain causes of troubles encountered by fabricators, or to fill minor gaps in established research programs.

Contract No. AF 33(616)-7747
Project No. 2(8-8975)

WELDABILITY STUDIES OF THREE COMMERCIAL
COLUMBIUM-BASE ALLOYS

P. A. Kammer and R. E. Monroe*

SUMMARY

At the present time, a group of columbium-base alloys is being considered for addition to the Department of Defense Refractory Metals Sheet Rolling Program. Good weldability is one of the requirements for inclusion of alloys in this program. Two alloys currently under consideration are B-66, produced by Westinghouse Electric Corporation, and FS-85, produced by Fansteel Metallurgical Corporation. A third alloy, C-129, produced by Wah Chang and submitted by Boeing Airplane Company, was also included in this evaluation. Samples of these three alloys were obtained for a brief weldability evaluation at Battelle to determine whether the alloys met the target properties established by the Materials Advisory Board, Refractory Metals Sheet Rolling Panel-Alloy Requirements and Selection Group. Room-temperature bend-ductility target properties for fabricable columbium-base alloys are 1T for base metal bends and 2T for welds bent with the weld transverse to the bend axis.

Bend tests made on base-metal samples showed that all alloys met the target properties for base metal. Bend tests of welds made in a vacuum-purged controlled-atmosphere chamber showed that only the welds in the FS-85 alloy possessed the desired target properties.

EXPERIMENTAL TECHNIQUES

This investigation was planned at the request of the Alloy Requirements and Selection Group of the Refractory Metal Sheet Rolling Panel to provide an evaluation by a single laboratory of the bend ductility of welds in selected columbium alloys. Request for the submission of candidate alloys was made by the Alloy Requirements and Selection Group to interested refractory-metal producers. The producers were asked to submit the alloys in a condition suitable for the welding studies, with restrictions being placed only on the thickness and quantity of material required. Sheet samples of the FS-85 (Cb-27Ta-10W-1Zr) and B-66 (Cb-5Mo-5V-1Zr) alloys were obtained from the metal producers. Samples of the C-129 (Cb-10W-10Hf) alloy were obtained from an aircraft producer who had purchased the sheet from the metal producer. Information on the fabrication history was available only for the FS-85 alloy. This material was stress relieved after a 50 per cent cold reduction from 0.080 inch. Available chemical analyses of the materials are given in Table 1; where actual analyses were not available, as for the FS-85 alloy, nominal compositions are shown in this table. The B-66 and FS-85 alloys were supplied as sheet 0.040 inch thick. The C-129 alloy was 0.030 inch thick.

*Metals Joining Research Division, Battelle Memorial Institute.

TABLE 1. COMPOSITION[(a)] OF COLUMBIUM-BASE ALLOYS EVALUATED

Alloy		Composition, weight per cent									
		Mo	V	Zr	C	W	Hf	Ta	O	N	H
B-66	Ingot, top	4.97	4.81	0.95	0.007	--	--	--	0.012	0.0055	--
B-66	Ingot, bottom	5.11	4.92	0.87	0.014	--	--	--	0.011	0.006	--
C-129	Ingot, top	--	--	--	--	10	10	--	0.019	0.0055	0.0038
C-129	Ingot, bottom	--	--	--	--	10	10	--	0.014	0.0025	0.0029
C-129	Sheet					10	10	--	0.0288	0.0049	0.0005
FS-85		--	--	1	--	10	--	27	--	--	--

(a) Underlined values are nominal composition; all others are actual analyses.

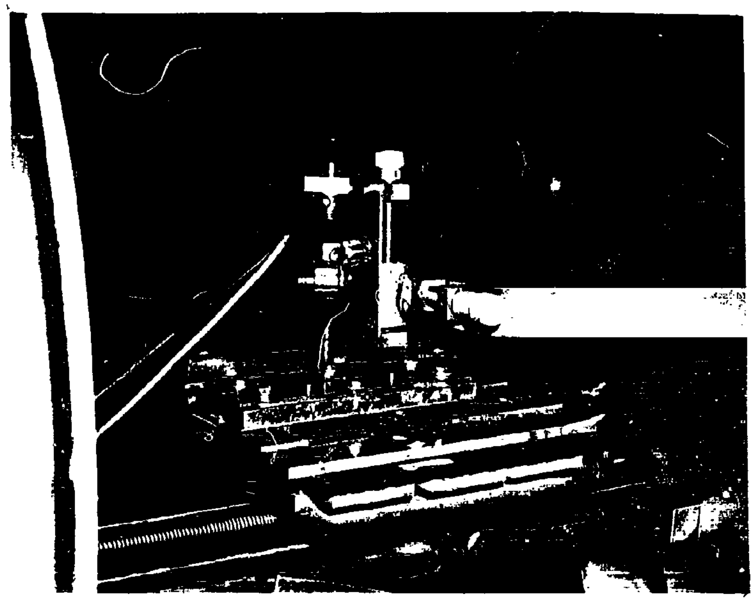
Specimens 1 by 4 inches were cut from the sheet material with the 4-inch dimension parallel to the apparent final rolling direction. Each specimen then was etched in a solution of 5 per cent HF, 40 per cent HNO_3, and 55 per cent water just prior to welding. The C-129 alloy was slightly discolored by this treatment. The other two alloys appeared bright and clean after etching.

Automatic tungsten arc welds were made on the sheet samples in an argon-filled chamber. Prior to making each weld, the chamber walls were wiped with clean acetone-soaked cloths. The chamber was then evacuated to a pressure of less than 1 micron of mercury (usually a pressure less than 0.2 micron was reached). The chamber leak rate was then determined and the chamber was again evacuated to less than 1 micron. No welds were made until a leak rate of less than 0.5 micron per minute was obtained. The chamber was equipped with a liquid-nitrogen cold trap to prevent backstreaming of the diffusion-pump oil. Figure 1 shows two views of the welding chamber.

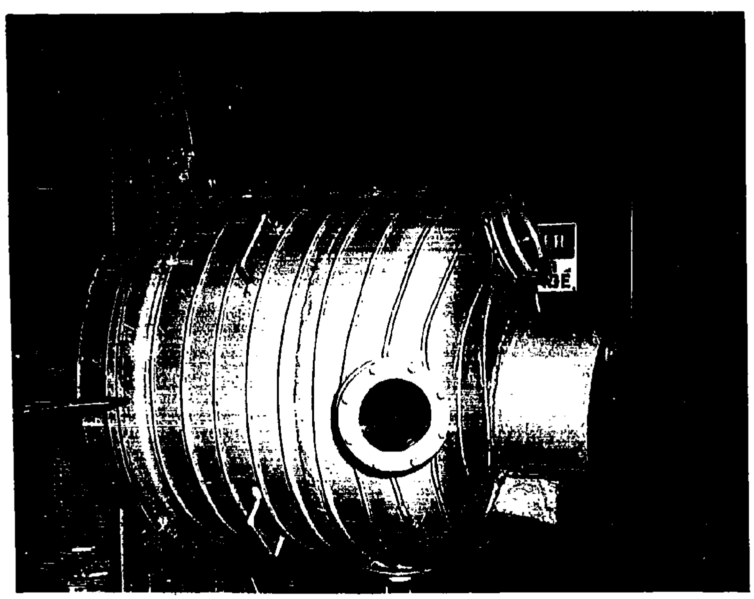
The procedure selected to accommodate changes in material thickness and alloy composition was to develop welding conditions that produced full penetration with a bead profile typical of refractory-metal weldments. Since it would not be possible to employ identical welding conditions for the alloys and thicknesses available, it was felt that this procedure would insure a fair test of each alloy. Welding conditions for the final welds were developed by making a series of short beads along the 1 by 4-inch specimens. The arc voltage and travel speed were held constant while the current was increased for each successive bead. Full-length trial welds were also made before the final specimens were welded. By using this approach, the conditions to produce full penetration were determined. The bead width in all cases was about 1/8 inch. Final welds consisted of a bead on the center of the specimens. Preliminary and final welding conditions are given in Tables 2 and 3, respectively. All welding was done with a 200-ampere 3-phase rectifier-type d-c welder. High-frequency-assisted starting was used.

The final welds in each material were examined visually and by radiography. All weld beads were clean and shiny in appearance and were found to be sound with no evidence of porosity, cracks, or other defects. The welds were then further evaluated by bend tests, hardness measurements, and metallographic examination.

Bend specimens were cut from the completed weldments on a cut-off wheel. These specimens were 1/2 by 2 inches for the B-66 and FS-85 alloys. The specimens were 5/16 inch by 2 inches for the C-129 alloy (the thinner material). The welds were parallel to the long dimension of the specimen as shown in Figure 2. In addition to the longitudinal bend specimens, base-metal bend specimens also were tested. The weld specimens were bent with the face of the weld in tension. Initially the bend tests were conducted in general accordance with the Materials Advisory Board "Tentative Specification for Bend Testing of Refractory Metals". Figure 2 shows the test fixture employed in such tests. As can be seen in this figure, a variable-radius tup is employed in this fixture. This radius, designated R_1 on the figure, ranged from 1/2 inch to a sharp radius in the tests conducted. The configuration of the sharp radius was that of a "V" with a flat 0.003 to 0.005 inch wide at the tip of the "V". The radius just larger than the sharp radius was



a. Chamber Interior N 98407



b. Chamber Exterior N 98406

FIGURE 1. AUTOMATIC TIG WELDING CHAMBER

TABLE 2. WELDING CONDITIONS FOR PRELIMINARY WELDS

Specimen Designation	Arc Voltage, volts	Travel Speed, ipm	Current, amp Set	Current, amp Meter	Remarks
B-66-1	10	20	100	89	Very narrow bead, complete penetration
	10	20	110	101	Bead about 9/64 inch wide, complete penetration
B-66-2	10	20	120	112	Burn-through
	10	20	115	108	Excessive penetration
B-66-3	10	20	105	100	Complete penetration
B-66-4	10	20	110	106	Insufficient penetration
B-66-5	10.5	20	125	120	Slightly excessive penetration
B-66-6	15	20	130	122	Good bead, slightly excessive penetration
C-129-1	10	20	100	90	Excessive penetration
	10.5	20	110	105	Ditto
	10.5	20	120	115	"
	11	20	130	120	"
C-129-2	10	20	90	84	Complete penetration
	9.5	20	80	73	Ditto
	9	20	70	59	Incomplete penetration
	9	20	60	46	Ditto
C-129-3	10	20	90	84	Complete penetration
	9.5	20	80	73	Ditto
	9	20	70	59	Incomplete penetration
	9	20	60	50	Ditto
C-129-4	10	20	85	77	Satisfactory, bead about 1/8 inch wide, complete penetration

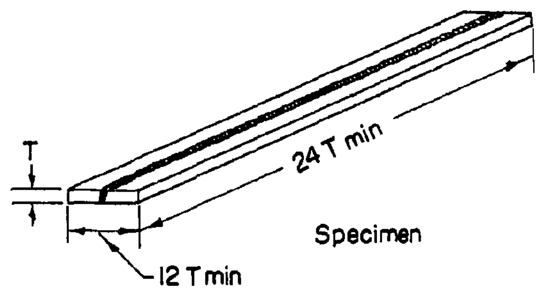
TABLE 2. (CONTINUED)

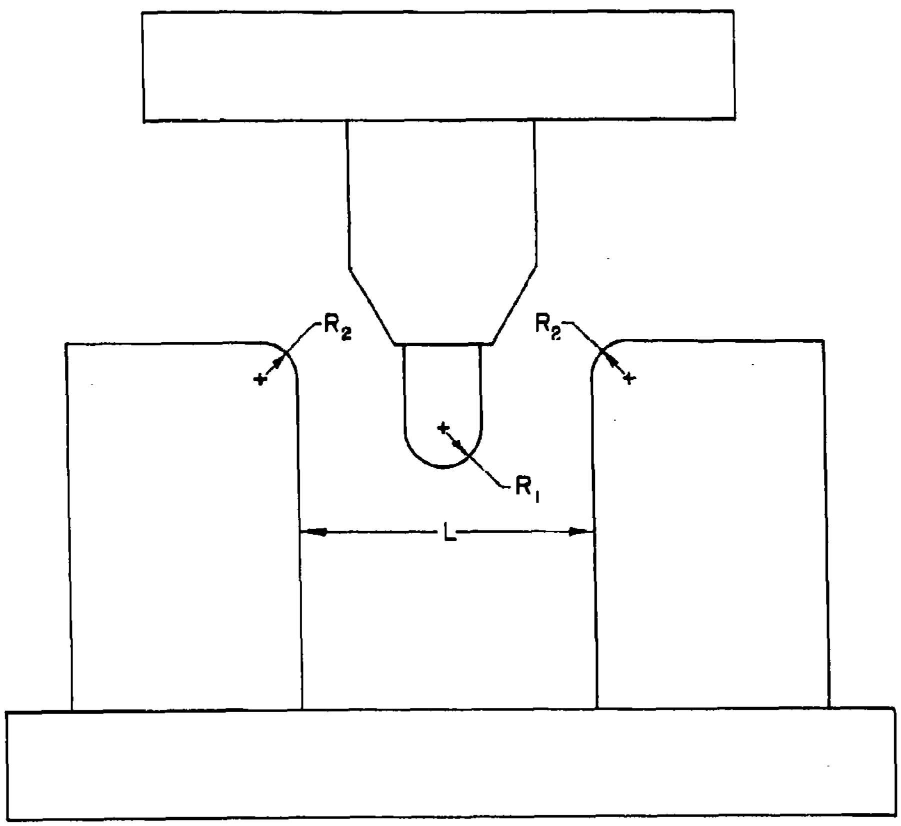
Specimen Designation	Arc Voltage, volts	Travel Speed, ipm	Current, amp Set	Current, amp Meter	Remarks
FS-85-1	9.5	20	95	85	Very narrow bead, incomplete penetration
	9.5	20	100	93	Ditto
	10	20	105	98	"
	10	20	110	104	Narrow bead, partial penetration
FS-85-2	10.5	20	115	108	Narrow bead, slight penetration
	11	20	120	113	Ditto
	10.5	20	125	117	Excessive penetration at start
	11	20	130	122	Ditto
FS-85-3	11	20	125	120	Good bead width, barely sufficient penetration
FS-85-4	11	20	130	122	Ditto
FS-85-5	11	20	137	125	"
FS-85-6	11.5	20	145	133	Good bead width, barely sufficient penetration
FS-85-7	11.5	20	155	147	Slightly undercut, almost excessive penetration

TABLE 3. FINAL WELDING CONDITIONS FOR B-66, C-129, AND FS-85 COLUMBIUM ALLOYS

Alloy	Current, amp Set	Current, amp Meter	Voltage, volts(a)	Travel Speed, ipm
B-66	130	122-123	11-11.5	20
C-129	85	73-77	10	20
FS-85	148	138-140	11-12	20

(a) Arc length was set at 0.030 inch for each weld.





Test Fixture

R_1 = Variable

R_2 = 1-1/2T Max

L = $3R_1$ + 2T or 15T, Whichever is Greater

T = Sheet Thickness

FIGURE 2. BEND SPECIMEN AND TEST FIXTURE

0.02 inch. A 1/32-inch radius was used for the shoulder supports of the fixture, designated as R_2. The time in seconds for completing each bend was less than the numerical value obtained by multiplying 200 times the sheet thickness in inches. To complete a bend within this time, a ram speed of 15 inches per minute was used. When the MAB-type fixture was used with loading tup radii smaller than 3/16 inch, the specimen did not conform to the test radii. Therefore, bends with a radius of less than 3/16 inch were completed by bottoming the specimen in a vee-block. This technique forces the specimen to conform to the radius of the tup.

RESULTS

The results of the room-temperature bend tests, summarized here, are given in Table 4. Base-metal specimens of all three alloys passed a sharp-radius bend. Welded specimens of FS-85 alloy also were adequately ductile, passing a sharp-radius bend. The welds in the B-66 alloy were not so ductile, failing at an average radius of 5T. Welds in the C-129 alloy were the least ductile, failing at an average radius of 7-1/2T. The B-66 alloy specimens failed with a complete fracture of the specimen; the C-129 specimens failed with a fracture in the weld, which was arrested in the heat-affected zone. This indicated that the weld was less ductile than the weld-heat-affected zone for this alloy, and that the heat-affected zone retained good crack-propagation resistance.

Both Vickers (5-kg load) and Knoop (200-g load) hardness measurements were made on welds in the three alloys. The results of the Vickers measurements are given in Table 5 and shown in Figure 3, which is a photomacrograph of typical specimens. The Vickers measurements present an "average" hardness of each section of the weldment as compared with the localized Knoop hardness measurements shown in Figure 4. Knoop hardness measurements were made across a transverse section of each of two welds for each alloy. Each point plotted in Figure 4 is an average of the hardnesses a given distance from the weld centerline on either side of the centerline for the two specimens of each alloy.

The hardness data correlated well with the bend-test results. The B-66 weldments were of almost uniform hardness across the weldment. The fusion zones were slightly harder than the remainder of the weldment. A marked decrease in the hardness of the heat-affected zones of the C-129 weldments was observed. The Vickers data indicated that the fusion zone was slightly harder than the base material, while the Knoop data indicated the converse. The low hardness of the C-129 heat-affected zones correlated well with the observation that the failures of the bend-test specimens of this alloy were arrested in the heat-affected zone. The Vickers data indicated that the softest portions of the FS-85 weldments were the heat-affected zones, while the base material was slightly harder than the fusion zones. The Knoop data indicated that the heat-affected zone/base material and fusion zone/heat-affected zone boundaries were the softest portions of the FS-85 weldments. The base material and fusion zones were about the same hardness, with the average heat-affected zone hardness slightly less.

TABLE 4. RESULTS OF BEND TESTS

Alloy	Specimen	Radius of Last[a] Tup Passed	Radius of Tup at Failure	Elongation[b] in Outer Fibers, per cent
B-66, Base metal	1	S	(c)	>50
	2	S	(c)	>50
	Avg	S	(c)	>50
B-66, Weld	1	4T	3T	10
	2	6T	4T	8
	3	3T	2T	14
	4	3T	2T	14
	5	(d)	12-1/2T	(d)
	6	(d)	8T	(d)
	Avg	>6T	5T	<8
C-129, Base metal	1	S	(c)	>50
	2	S	(c)	>50
C-129, Weld	1	6T	4T	7
	2	10-1/2T	8T	6
	3	10-1/2T	8T	6
	4	12-1/2T	10-1/2T	4
	Avg	10T	7-1/2T	6
FS-85, Base metal	1	S	(c)	>50
	2	S	(c)	>50
	Avg	S	(c)	>50
FS-85, Weld	1	S	(c)	>50
	2	S	(c)	>50
	3	S	(c)	>50
	4	S	(c)	>50
	Avg	S	(c)	>50

(a) Letter S designates the "sharp" radius tup (see text); radius is expressed as multiples of the specimen thickness, T.

(b) Calculated from $E = \frac{T}{2R+T} \times 100$,

where E = per cent elongation in the outer fibers

T = thickness of material

R = radius of last tup passed.

For those specimens which passed the "sharp" radius tup, E is given as being greater than the value calculated for an 0.020 radius.

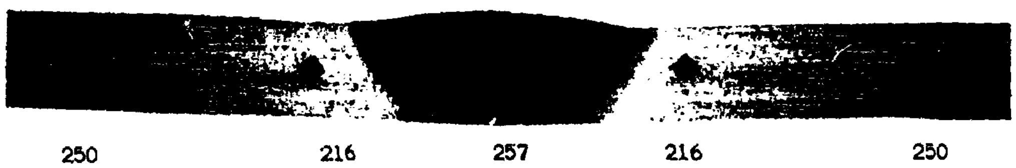
(c) No failure occurred (passed "sharp" radius tup).

(d) Failure occurred at first radius used.

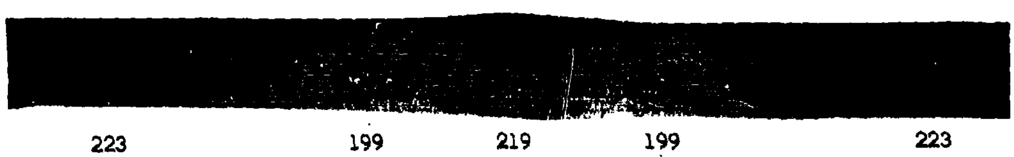
TABLE 5. RESULTS OF VICKERS HARDNESS MEASUREMENTS ACROSS WELDS

Alloy	Vickers Hardness Number (5-kg load) Base Metal	Heat-Affected Zone	Fusion Zone	Heat-Affected Zone	Base Metal
B-66	223	227	239	225	223
	227	236	232	232	225
C-129	251	214	251	214	244
	249	214	262	221	257
FS-85	225	201	232	206	221
	223	195	206	195	223

(a) B-66 Alloy



(b) C-129 Alloy



(c) FS-85 Alloy

15X N 97655

FIGURE 3. TRANSVERSE CROSS SECTIONS OF TUNGSTEN-ARC WELDS IN COLUMBIUM-BASE ALLOYS

Average Vickers Hardness is Given Under Each Impression

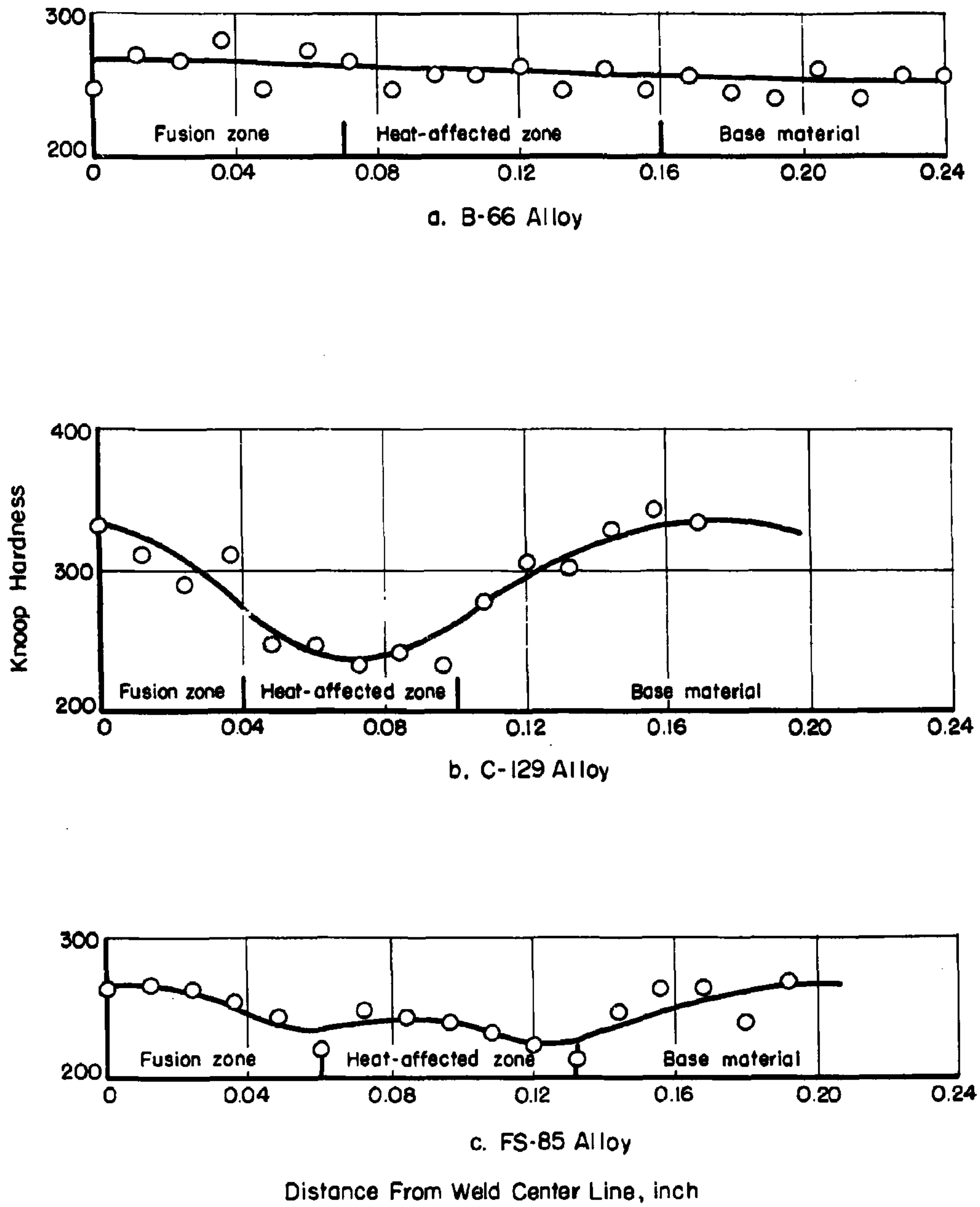


FIGURE 4. RESULTS OF KNOOP HARDNESS MEASUREMENTS (200 g load)

The results of the metallographic examinations conducted were found to explain most of the other observations made in this study. Examinations of the starting sheet microstructure confirmed the presence of a wrought structure in the FS-85 alloy sheet. The C-129 alloy sheet also exhibited a wrought structure but one which is indicative of a much greater final cold reduction than that employed for the FS-85 alloy. The structure of the C-129 alloy exhibited severe banding, suggesting considerable alloy segregation. In contrast to the other alloys, the B-66 alloy sheet was completely recrystallized.

Metallographic examination also revealed a probable surface contamination of the B-66 and C-129 alloys to a depth of about 0.001 to 0.002 inch. The contaminant particles appeared larger and more numerous in the weld-heat-affected zone. This region is heated to high temperatures during welding and is subsequently rapidly cooled. The appearance of the contaminant particles in the heat-affected zone is indicative of coalescence or precipitation. Typical examples of the surface regions in these alloys are shown in Figure 5. The surface contamination appears to be greater in the C-129 alloy (Figure 5a) than in the B-66 alloy (Figure 5b). Metallographic examination did not disclose any evidence of surface contamination on the FS-85 alloy sheet.

Photomicrographs showing typical regions of the weldments in the three alloys are shown in Figures 6, 7, and 8. In the fusion zones of the welds in all three alloys, a core dendritic structure was observed. This coring was most pronounced in the C-129 alloy. Small particles of a second phase also were observed in the fusion zones of the B-66 and C-129 alloys. More of these particles were present in the C-129 alloy than in the B-66 alloy welds. The second phase may have been the result of the surface contaminants observed on these alloys. Recrystallization and grain growth occurred in the heat-affected zones of both the C-129 and FS-85 alloys. Since the B-66 alloy was already recrystallized, only grain growth occurred in the heat-affected zone of this alloy. Wavy lines present in the heat-affected zones in the C-129 and FS-85 alloys are probably remnants of alloy segregation originally present in the wrought base material.

CONCLUSIONS

The results of this study showed that all of the three alloys evaluated met the desired target properties for wrought sheet material. However, only the FS-85 alloy met the desired target properties for weldment bend ductility. The samples of B-66 and C-129 alloys were representative of production materials, but surface contamination was detected on both alloys. This contamination could have occurred at any one of a number of steps in the fabrication of the alloys. It is apparent that a quality-control procedure is needed to detect such contamination. Since contamination might vary over the surface of a large sheet, a simple, nondestructive technique would be most desirable.

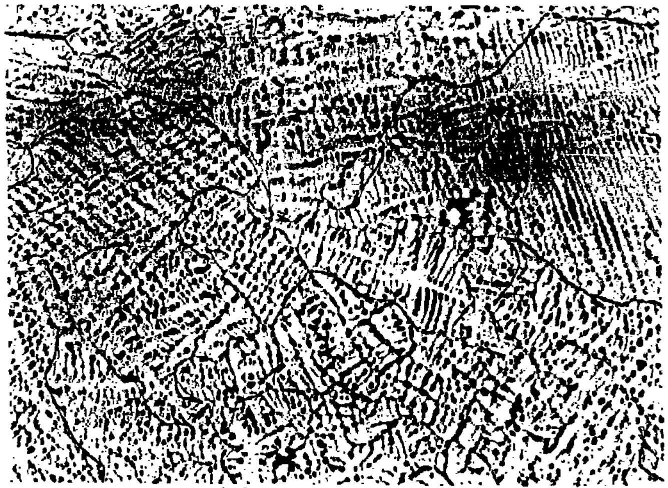
250X (a) C-129 N 97328
25 g NH_4FHF
50 ml HNO_3
100 ml H_2O

750X (b) C-129 N 97326
50 ml lactic acid
50 ml HNO_3
1 ml HF

250X (c) B-66 N 97334
25 g NH_4FHF
50 ml HNO_3
100 ml H_2O

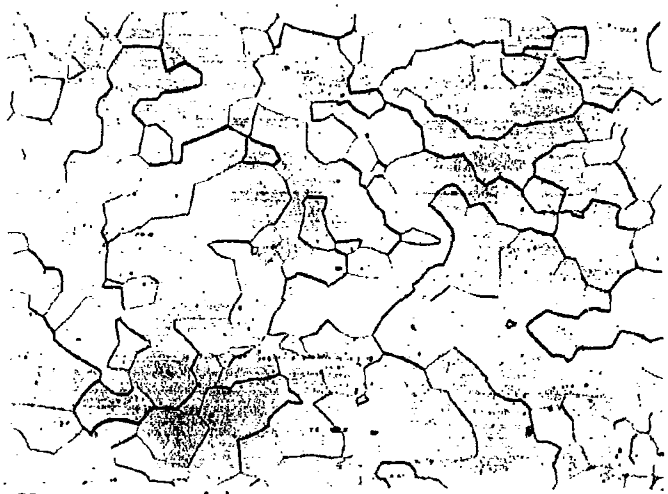
FIGURE 5. SURFACE CONTAMINATION IN C-129 AND B-66 COLUMBIUM ALLOYS

Areas Shown are Typical of Weld-Heat-Affected Zone



250X (a) Fusion Zone N 97333

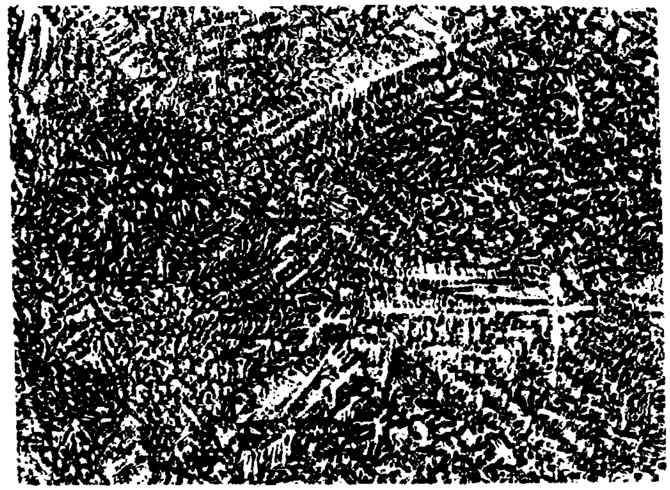
25 g NH_4FHF
50 ml HNO_3
100 ml H_2O



250X (b) Heat-Affected Zone N 97335

25 g NH_4FHF
50 ml HNO_3
100 ml H_2O

FIGURE 6. MICROSTRUCTURES OF WELDS IN B-66 COLUMBIUM ALLOY

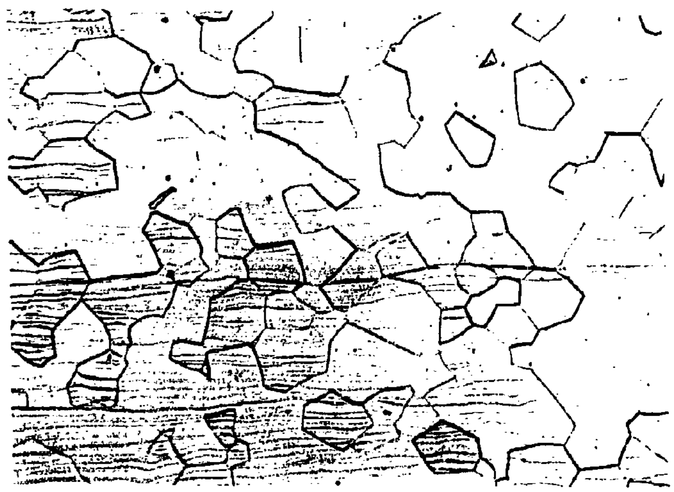


250X (a) Fusion Zone N 97327
25 g NH_4FHF
50 ml HNO_3
100 ml H_2O

250X (b) Heat-Affected Zone N 97329
25 g NH_4FHF
50 ml HNO_3
100 ml H_2O

FIGURE 7. MICROSTRUCTURES OF WELDS IN C-129 COLUMBIUM ALLOY

250X (a) Fusion Zone N 97337
25 g NH_4FHF
50 ml HNO_3
100 ml H_2O



250X (b) Heat-Affected Zone N 97339
25 g NA_4FHF
50 ml HNO_3
100 ml H_2O

FIGURE 8. MICROSTRUCTURES OF WELDS IN FS-85 COLUMBIUM ALLOY

Observations made during the bend tests conducted on this program indicate that the tentative MAB specification for this type of testing is not entirely adequate. Thin-sheet specimens will not properly conform to the smaller bend radii necessary to determine the bend ductility of relatively ductile materials. It is suggested that the specification for bend testing be modified to allow the use of test fixtures which insure that the bend sample conforms to the radius being employed in the test.

LIST OF DMIC MEMORANDA ISSUED
(Continued)

A list of DMIC Memoranda 1-164 may be obtained from DMIC, or see previously issued memoranda.

DMIC Memorandum Number	Title
165	Review of Uses for Depleted Uranium and Nonenergy Uses for Natural Uranium, February 1, 1963
166	Literature Survey on the Effect of Sonic and Ultrasonic Vibrations in Controlling Grain Size During Solidification of Steel Ingots and Weldments, May 15, 1963
167	Notes on Large-Size Furnaces for Heat Treating Metal Assemblies, May 24, 1963 (A Revision of DMIC Memo 63)
168	Some Observations on the Arc Melting of Tungsten, May 31, 1963
169	Weldability Studies of Three Commercial Columbium-Base Alloys, June 17, 1963