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Refractory Metal Alloys in Sheet Form: Availability, Properties, and Fabrication

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Introduction

AVAILABILITY of wide refractory metal and alloy sheet as a commercial mill product has become a reality during the past two years. As recently as four years ago, Mo-0.5 Ti was the only alloy available as large sheet, whereas today 23 different alloys are offered commercially by 11 producers. In the present paper, a commercially available alloy is defined as one in which sheet of 12-in. minimum width is marketed as a standard mill product. With few exceptions, each metal or alloy is available from at least one producer in widths of 24–36 in. and lengths of 72–96 in. Certain of the Cb- and Ta-base alloys can be produced as 40- × 96-in. flat sheet. Refractory metal and alloy foil (0.005-in. thick) have become available in widths up to 12-in. during the past year. Foil that is up to 24-in. wide is being produced experimentally under Air Force contract, and several Cb-base alloys are available as commercial products today. A major effort is being given to the production of wide foil, particularly in Cb- and Ta-base alloys, for use in lightweight sandwich-panel construction. In this paper the current status of such commercially produced sheets as columbium, molybdenum, tantalum, tungsten, and their alloys are summarized. The information and data presented have been obtained from the 11 domestic producers of these materials or from open published literature.

The availability section lists the maximum sizes of sheet produced at thicknesses of 0.060, 0.030, 0.010, and 0.002-in. The principal alloys of interest for aerospace applications are characterized in terms of composition, size availability, and cost. The current level of quality and activities designed to improve quality are discussed.

The properties section presents, in a tabular and graphical manner, a summary of the typical mechanical properties of these materials. Emphasis is placed on tensile strength and stress-rupture properties. Properties of alloys in one class, i.e.,

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Cb-base alloys, are reviewed and comparisons made with properties of the other classes of alloys. The purpose of this section is to illustrate the wide range of properties available in Cb, Mo, Ta, and W commercial alloys and to highlight areas in which further testing and evaluation are needed.

The fabrication section reviews the current status in production-shop forming of these alloys. The general types of parts produced and the forming practices used are discussed. Problems in production forming, such as brittle fracture, delamination, and dimensional tolerances, are mentioned. This section illustrates the complexity of the parts that can be made, the flexibility of forming processes that can be used, and the relative ease of manufacture of these materials.

Commercial Availability

The maximum-size refractory metal and alloy sheet and foil currently produced by the different domestic producers is given in Table 1, along with price-range data for 100-lb lots of material. Available sizes are limited more by the size of ingots and restrictions of annealing equipment than by the rolling-mill capabilities. Although keen competition exists between producers for the present limited market for wide sheet, there also is much cooperation in processing these materials. Most suppliers will produce on a mill-order conversion basis many of the refractory metals and alloys that they do not market commercially as standard mill products. Thus, although Table 1 lists the producers and trade designations for each alloy, it should be recognized that any of the producers will roll any of the listed alloy compositions within the broad area of his capabilities on a mill-order basis. The buyer today is in an excellent position to obtain the most favorable price and delivery by competitive bidding and generally is not restricted to the original alloy developer as a source for material. For a producer, exact cost of a given material will be strongly dependent on sheet size, quantity, tolerances (length, width, thickness, and flatness) and other restrictive specifications. Unalloyed and low-strength, lightly alloyed sheet can be obtained at least cost. The newer high-strength alloys command a premium price, which for Cb-base materials is about twice that of the lower-strength alloys. Cost per pound also is seen to increase significantly as sheet thickness decreases.

In the past, many users have not been particularly cost conscious with respect to components and structures manufactured from refractory metal sheet. Today, however, as the number of alloy compositions competing for a given application increases, considerable benefit may be realized by analyzing possible tradeoffs in materials and design with respect to cost as well as properties and performance. For this purpose, quoted prices in \$/lb can be misleading, since weight per unit area is not being considered. A more realistic basis for comparison is provided by expressing cost per unit area for a given thickness of material. This is illustrated in Fig. 1 in which the cost of 10 different sheet materials in $\$/ft^2$ is given as a function of sheet thickness for four different gages. These values were calculated using the minimum prices listed in Table 1 and the density of the particular metal or alloy.

Cost for 0.060-in.-thick sheet is shown to range from $\$48/ft^2$ for sintered molybdenum to $\$417/ft^2$ for a tantalum-base alloy (T-111). Cost decreases with reduced thickness due to the lesser weight per unit area, and at 0.010-in. thickness all of the materials are priced between \$15 and $\$75/ft^2$. The high-strength Cb-base alloys are comparable in price to high-strength Mo-base alloys in thin sheet, although the cost per pound of the former is three times that of the latter in heavier gage material. Comparisons such as this provide a sound basis for considering tradeoffs of properties and cost. In some cases, structures can be produced at greatly reduced cost by using a slightly heavier gage sheet of a moderate- to low-strength alloy in the place of a somewhat lighter-gage higher-strength material.

Estimates of industry capacity indicate that refractory metal and alloy sheet can be produced in sufficient quantity to meet anticipated applications. A Materials Advisory Board report of 1959 stated that 100 tons per year each of Mo, W, Cb, and Ta alloy sheet could be produced in existing facilities.[1] Production could be increased to 1000 tons per year of Cb, Mo, or W by expansion in ore reduction and processing facilities. Tantalum alloy production may be limited 500–600 tons per year by availability of workable ore deposits.

Most of the unalloyed metal and alloy sheet materials are available on a mill-order basis, with average delivery ranging

Table 1 Commercial availability of refractory metal and alloy wide sheet

MATERIAL	DOMESTIC PRODUCERS[a] AND TRADE DESIGNATIONS	MAXIMUM SIZE – W × L (in.)				APPROXIMATE PRICE RANGE[b]			
		.060"	.030"	.010"	.002"	.060"	.030"	.010"	.002"
COLUMBIUM									
1) Cb - 1Zr	2(D-11), 3(FS-80), 5(Cb-751), 7(SCb-990), 10(Cb-1Zr)	40 × 93	40 × 96	36 × 96	24 × COIL	50 - 85	50 - 90	60 - 100	80 - 120
2) Cb - 5Zr	2(D-14)	28 × 96	28 × 96	28 × 96	24 × COIL	75 - 80	75 - 80	95 - 100	-
3) Cb - 5V	11(B-33)	18 × 100 lb	18 × 100 lb	18 × 100 lb	12 × 100 lb	85 - 90	95 - 100	105 - 110	140 - 150
4) Cb - 10Ti - 5Zr	2(D-36), 3(D-36), 7(SCb-885)	30 × 108	30 × 108	28 × 96	24 × COIL	65 - 85	65 - 90	70 - 100	90 - 120
5) Cb - 10W - 2.5Zr	5(Cb-752)	40 × 96	40 × 96	36 × 96	12 × COIL	100 - 120	105 - 125	110 - 130	-
6) Cb - 10W - 10Ta	7(SCb-291)	30 × 100	30 × 100	24 × 300	12 × 70 lb	55 - 85	60 - 90	65 - 100	85 - 120
7) Cb - 10W - 10Hf	10(C-129)	24 × 72	24 × COIL	24 × COIL	4.5 × COIL	100 - 105	105 - 110	110 - 115	105 - 110
8) Cb - 33Ta - 1Zr	3(FS-82)	30 × 108	30 × 108	24 × 80	12 × COIL	70 - 75	80 - 85	100 - 105	120 - 125
9) Cb - 11W - 28Ta - 1Zr	3(FS-85)	30 × 108	30 × 108	24 × 80	12 × COIL	80 - 85	85 - 90	100 - 105	120 - 125
10) Cb - 5Mo - 5V - 1Zr	11(B-66)	18 × 100 lb	18 × 100 lb	18 × 100 lb	12 × 100 lb	120 - 125	125 - 130	130 - 135	190 - 195
11) Cb - 10W - 1Zr - 0.1C	2(D-43)	28 × 96	28 × 96	28 × 96	24 × COIL	115 - 120	115 - 120	135 - 140	-
12) Cb - 10Ti - 10Mo - 0.1C	2(D-31)	28 × 96	28 × 96	28 × 96	-	115 - 120	115 - 120	135 - 140	-
13) Cb - 10Hf - 1Ti - 0.7Zr	10(C-103)	30 × 120	30 × COIL	30 × COIL	-	95 - 100	95 - 100	100 - 105	95 - 100
MOLYBDENUM									
1) UNALLOYED - SINTERED	3(Mo), 4(Mo), 9(Mo-PM), 11(Mo), 8(Mo)	36 × 96	24 × 96	24 × 96	12 × COIL	15 - 20	15 - 25	20 - 35	55 - 60
2) UNALLOYED - ARC CAST	1, 9(Mo-AVC), 3(Mo)	36 × 96	24 × 72	16 × 48	12 × COIL	30 - 35	35 - 40	55 - 60	-
3) Mo - 0.5Ti	3(FS-40), 9(Mo-0.5 Ti), 8(MTC)	36 × 96	24 × 72	16 × 48	12 × COIL	30 - 35	35 - 40	65 - 70	-
4) Mo - 0.5Ti - 0.08Zr	1(TZM), 3(FS-42), 9(TZM)	36 × 96	24 × 72	16 × 48	12 × COIL	40 - 45	50 - 55	95 - 100	-
TANTALUM									
1) UNALLOYED	3(Ta), 5(Ta), 6(Ta), 7(STa1000)	40 × 96	40 × 96	36 × 96	12 × COIL	50 - 90	60 - 100	70 - 100	90 - 130
2) Ta - 10W	3(FS-60), 5(Ta-782), 6(Ta-10W), 7(STa-900)	40 × 96	40 × 96	36 × 96	12 × COIL	60 - 90	65 - 100	70 - 100	90 - 130
3) Ta - 8W - 2Hf	3(T-111), 6(Ta-8W-2Hf), 11(T-111)	30 × 100	30 × 100	24 × 300	12 × 100 lb	80 - 120	88 - 135	88 - 145	210
4) Ta - 12.5W	7(STa-880)	30 × 100	30 × 100	24 × 300	12 × COIL	60 - 90	65 - 100	70 - 100	90 - 130
TUNGSTEN									
1) UNALLOYED - SINTERED	3(W), 4(W), 9(W-PM)	24 × 48	24 × 60	24 × 36	6 × 24	20 - 32	24 - 45	48 - 76	120
2) UNALLOYED - ARC CAST	9(AVC-W)	12 × 24	18 × 30	16 × 36	-	63	78	110	-

[a] Identification of producer as numbered: 1. Climax Molybdenum Co. 2. DuPont Metals Co. 3. Fansteel Metallurgical Corp. 4. General Electric Co. 5. Haynes Stellite Co. 6. National Research Corp. 7. Stauffer Metals Co. 8. Sylvania Electric Products Co. 9. Universal-Cyclops Steel Corp. 10. Wah Chang Corp. 11. Westinghouse Electric Corp.

[b] \$/lb in 100-lb lots for each of 4 thicknesses listed.

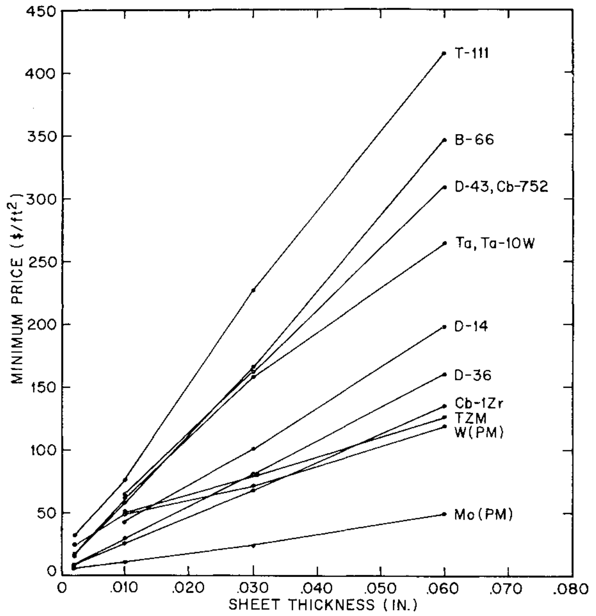

Fig. 1 Cost per unit area of refractory metal and alloy wide sheet as function of sheet thickness.

from 2 to 12 weeks. In a few cases, limited stocks of varying gages are maintained, and delivery can be obtained from stock in less than one week. Extensive stocking programs have not been undertaken because of the wide variety of sizes required, the limited demand for any given size material, and the dollar volume represented by large stocks of relatively expensive sheet-metal products. Producers report, however, that it would be completely feasible to stock material and to take advantage of accompanying economies, if a continuing demand existed for standard-size mill products.

Commercially produced refractory metal and alloy sheet is of variable quality and in some cases may not consistently meet mechanical properties achieved in carefully controlled pilot production. Because these materials have not been generally available until recent times, information on quality is general rather than specific. There are essentially no baseline data on the quality of current production materials. There are several reasons for this apparent shortcoming. First, producers receive numbers of orders for relatively small lots of varied material and, as a result, have not had the opportunity to determine the quality and the effects and interactions of the many processing variables; second, the users for the most part are conducting research and development programs and have been primarily concerned with manufacture of hardware for component testing. As any application nears a production phase, naturally more attention is given to quality and reproducibility.

In general, wide sheet will conform to tolerances of AMS-2242, "Corrosion resistant steel sheet, plate, and strip." Flatness and gage tolerances appear to be adequate for most proposed aerospace applications. Flatness usually can be maintained within $\pm 4\%$ (half-chord) and thickness tolerances generally are less than $\pm 10\%$ of nominal gage. Variations in mechanical properties, ductile-brittle behavior, and response to forming are known to exist; however, the statistical range of variations and minimum design properties have not been established for most alloys.

As a result of the apparent need to accelerate efforts to achieve high-quality consistent sheet products, the Department of Defense initiated a refractory metal sheet-rolling program in June 1959. The Navy Department BuWeps and Air Force Aeronautical Systems Division manage the contract phases of the program. The Materials Advisory Board of the National Academy of Sciences was requested to form an advisory panel to assist in technical aspects of the program.[2] Among specific contributions that will evolve from the program are manufacture of large sheet by consistent practices in sufficient quantity for evaluation and fabrication of components; standardization of quality and acceptance criteria for sheet materials; and establishment of adequate engineering design data and optimum methods of fabrication for each material.

The total cost of the program is estimated to be at least 10 million dollars, and the studies will be conducted over a 5-yr period. It should be emphasized that this is a long-term program designed to provide a more rational basis for producing high-quality refractory metal sheet suitable for the manufacture of airframe and missile components. Four phases are covered: 1) manufacturing development and sheet production; 2) mechanical testing and evaluation; 3) fabrication evaluation; and 4) design data. Sufficient sheet, at least 24-in. wide, will be manufactured in phase 1 programs for use in detailed fabrication, testing, and evaluation programs. A summary of past and current contracts that comprise the over-all sheet rolling program is given in Table 2. Only two materials, unalloyed tungsten and TZM alloy, have entered the phase 2 and 3 programs of testing and fabrication evaluation. Several of the more advanced high-strength Cb- and Mo-base alloys recently have been included in phase 1 programs.

Property Data

Although fairly complete tensile data are available for the various commercial alloys in the general temperature range of interest, stress-rupture data and particularly creep data under similar test conditions of temperature and stress which allow comparisons between the alloys are relatively scarce. In addition, the usual problem exists in that the available data were obtained under various test conditions using different testing techniques in different laboratories. For example, the tensile data summarized for the columbium alloys represents at least five different strain-rate schedules. An additional shortcoming in the available data is that not sufficient testing has been conducted on different lots of the same alloy to indicate the spread in mechanical properties which might be expected for a given alloy. The property data presented in this paper thus represent "typical" values only and are not guaranteed minimums.

Columbium Alloys

Early efforts in the development of columbium alloys were largely influenced by two major objectives: to develop maximum-strength alloys and to develop alloys with improved oxidation resistance over that of the base metal. Some 30 experimental columbium alloys were extensively evaluated during this early effort. The highest-strength, most oxidation-resistant alloys, which initially received great interest and attention, presented difficulties due to lack of sufficient ductility. Serious limitations were encountered with a number of the early alloys during processing into final sheet, during fabrication of the sheet, and due to loss of room-temperature ductility during welding. Furthermore, the maximum improvement in oxidation resistance due to alloying (about 100 times that for unalloyed columbium) was not nearly great enough for satisfactory service in the desired temperature range without a protective coating.

The net result today is that the early alloys that still remain of interest and the newer alloys that have been developed during the past few years (so-called second-generation alloys) have had as their basic requirement good high-temperature strength while still maintaining the low-temperature ductility, ease of fabricability, and ductility in the welded condition intrinsic in unalloyed columbium. Table 3 gives the nominal compositions of the columbium alloys that the various producers currently list as commercial alloys available in sheet form, and Table 4 summarizes the room-temperature

Table 2 Refractory metal sheet-rolling programs

Program Phase	Subject	Materials				Contractor[b]	Contract Number
		Cb	Mo	Ta	W		
I – Manufacturing Development and Sheet Production	1. Molybdenum alloy sheet rolling		TZM Mo-0.5Ti			15	NOAS 59-6142C
	2. Infab processing of molybdenum alloy sheet		TZM			15	AF 33(657)-8495
	3. Molybdenum sheet by powder metallurgy[a]		MTC			13	NOAS 60-6018C
	4. Co-reduction and consolidation of alloys		Mo-0.5Ti			1	NOW 61-0548C
	5. Consolidation of alloy ingots		TZM, TZC		alloys	5	AF 33(657)-8792
	6. Rolling large tungsten sheet[a]				W-PM	6	NOW 60-0621C
	7. Arc-melted tungsten sheet rolling				W-AC	15	AF 33(600)-41917
	8. Pilot production of high strength alloys		TZC		W-AC, alloys	3	NAVY BUWEPS
	9. Tungsten sheet by point deformation				W-PM	16	NOW 61-1046C
	10. Columbium alloy sheet rolling[a]	F-48, D-31				4	AF 33(600)-39942
	11. Columbium alloy sheet rolling	D-43				5	AF 33(600)-39942
	12. Pilot production of columbium alloy sheet	B-66, FS-85				17	N 600(19)59546
	13. Pilot production of columbium alloy sheet	B-66, FS-85				6	NOW 63-0231C
	14. Pilot production of columbium alloy sheet	Cb-752				8	AF 33(657)-7210
	15. Tantalum alloy sheet rolling			30Cb-7.5V		16	AF 33(657)-7015
	16. Pilot production of tantalum alloy sheet			T-111		17	N 600(19)-59762
	17. Refractory alloy foil rolling	D-43, B-66, Cb-752		Ta-10W, T-111		5	AF 33(657)-8912
	18. Refractory alloy foil rolling					14	AF 33(657)-9384
	19. Pilot production of tantalum alloys			alloys		2	AF 33(616)-7452
	20. Manufacture of flow-turned tungsten sheet				W-PM	16	AF 33(600)-43034
	21. Reduction and consolidation of molybdenum		Mo-AC			16	NOAS 60-6046C
II – Mechanical Testing and Evaluation	1. Physical and mechanical properties of sheet		TZM		W-PM	11	N 600(19)-59530
III – Fabrication Evaluation	1. Spinning evaluation of tungsten sheet				W-PM	12	NOW 63-0542C
	2. Forming and evaluation of molybdenum sheet		TZM			9	NOW 61-0653t
	3. Forming and evaluation of columbium sheet[a]	F-48, D-31				7/9	AF 33(616)-6578
	4. Forming evaluation of tungsten sheet				W-PM	10	NAVY BUWEPS

[a] Program complete, final report issued.

[b] 1. Armour Research Foundation (IIT) 2. Battelle Memorial Institute 3. Climax Molybdenum Co. 4. Crucible Steel Co. 5. E. I. Du Pont de Nemours Co. 6. Fansteel Metallurgical Corp. 7. General Electric Co. 8. Haynes Stellite Co. 9. McDonnell Aircraft Corp. 10. Solar Div., Int. Harvester Corp. 11. Southern Research Institute 12. Supertemp Corp. 13. Sylvania Electric Products Co. 14. Texas Instruments Corp. 15. Universal Cyclops Steel Corp. 16. Wah Chang Corp. 17. Westinghouse Electric Corp.

tensile properties, densities, and melting points of the same alloys. Several points of interest may be noted from these data.

First it is interesting to note the wide range of melting points of the various alloys. D-36 has a melting point of 3500°F, 1000°F below that of unalloyed columbium, whereas FS-85 has a melting point of about 4700°F or about 200°F above that of columbium. The alloys with the highest melting points can be expected to have greater superiority in terms of tensile properties and creep resistance at the very high temperatures. Also of interest is the variation in density of the various alloys. FS-85 with a density of 0.390, as compared with 0.305 for B-66, would have to have a higher-strength value at a given temperature of almost 30% in order to be equal to B-66 on a density-compensated strength basis.

Figure 2 summarizes the ultimate-tensile-strength to density ratio for all the columbium alloys listed in Table 3. All materials were in the recrystallized condition, with the exception of FS-85, which was in the 50% cold-worked condition, and D-14, which was in the stress-relieved condition. All tests were conducted in a vacuum. Of interest is the large spread in strength-to-density ratio for the various alloys: for example, at 2000°F the lowest to highest value represents a 125% increase, whereas at 2700°F a 350% increase exists from lowest-to-highest value. B-66 alloy exhibits the highest strength of all the alloys, at least over the temperature range 2000°–2600°F.

Referring to the curves of Fig. 2, it is apparent that FS-85, which was in the 50% cold-worked condition, showed a greater than normal drop between 2000° to 2400°F, crossing the curves for C-129 and Cb-752 and resulting in FS-85 moving from third to fifth position in the top five strongest alloys. Also of interest is the very abrupt drop shown for D-43 between 2400° to 2600°F. This decrease above 2400°F has been ascribed to over-aging of the zirconium-carbide precipitate. At the very high temperatures above 3000°F the high-melting-point alloys or those alloys containing large additions of W and Ta begin to show superior strength.

Table 3 Nominal compositions of commercial columbium sheet alloys, % weight

ALLOY DESIGNATION	PRODUCER	W	Mo	Ta	V	Hf	Ti	Zr	C
D-14	DU PONT	–	–	–	–	–	–	5	–
D-31	DU PONT	–	10	–	–	–	10	–	0.1
D-36	DU PONT	–	–	–	–	–	10	5	–
D-43	DU PONT	10	–	–	–	–	–	1	0.1
FS-82	FANSTEEL	–	–	33	–	–	–	1	–
FS-85	FANSTEEL	11	–	28	–	–	–	1	–
Cb-752	HAYNES-STELLITE	10	–	–	–	–	–	2.5	–
SCb-291	STAUFFER	10	–	10	–	–	–	–	–
C-103	WAH CHANG	–	–	–	–	10	1	0.7	–
C-129	WAH CHANG	10	–	–	–	10	–	–	–
B-33	WESTINGHOUSE	–	–	–	5	–	–	–	–
B-66	WESTINGHOUSE	–	5	–	5	–	–	1	–
Cb-1Zr	VARIOUS	–	–	–	–	–	–	1	–

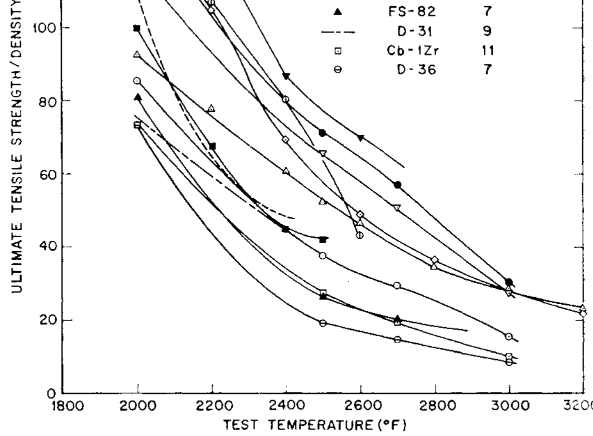

Fig. 2 Ultimate-tensile-strength to density ratio for commercial columbium alloys.

Table 4 Room-temperature tensile and other selected properties of commercial columbium sheet alloys

ALLOY DESIGNATION (Condition Normally Supplied)	0.2% YS (ksi)	UTS (ksi)	ELONG. (%)	MODULUS OF ELASTICITY (10^6 psi)	DENSITY (lb/in.^{3})	MELTING POINT (°F)
UNALLOYED Cb				15.1	0.310	4475
D-14 (S-R)	61	75	15	13.7	0.310	3950
D-31 (Rx)	90	98	–	16.5	0.292	4100
D-36 (Rx)	72	80	20	16.5	0.286	3500
D-43 (S-R)	70	87	21	16.2	0.326	–
FS-82 (S-R)	90	98	12	17.0	0.372	4550
FS-85 (S-R)	92	109	14	20.0	0.390	4695
Cb-752 (Rx)	70	84	22	15.0	0.326	–
SCb-291 (Rx)	60	75	25	–	0.347	–
C-103 (Rx)	50	59	26	12.6	0.320	–
C-129 (Rx)	72	88	26	16.0	0.343	–
B-33 (Rx)	54	78	32	15.8	0.306	4310
B-66 (Rx)	76	101	26	15.3	0.305	4300
Cb-1Zr (Rx)	35	48	15	11.5	0.31	4375

The order of decreasing strength-to-density ratio for the top five alloys at different test temperatures is listed below and presented graphically in Fig. 3.

2000°F	2400°F	2600°F
B-66	B-66	B-66
D-43	D-43	C-129
FS-85	C-129	Cb-752
C-129	Cb-752	FS-85
Cb-752	FS-85	SCb-291

Figure 4 summarizes the available modulus of elasticity vs temperature data for the various columbium alloys. Data for nine alloys are represented, whereas two years ago modulus data for only two of these alloys were available. Room-temperature values of modulus for unalloyed columbium have been reported by various investigators to vary from 14.3 to 17.8 × 10^6 psi, with temperature dependence similar to that for the alloy curves shown in Fig. 4. This fact gives an indication of how much variation from the values reported in Fig. 4 may be expected as more data are reported. Also, this indicates the desirability of having property data for different alloys evaluated at one laboratory using identical techniques, in order to have a meaningful comparison between the different alloys.

Figure 5 presents density-compensated stress-rupture curves for 1-, 10-, and 100-hr rupture life for the columbium alloys for which data were available. The data points shown for D-14, D-31, and D-36 were calculated from a Larson-Miller plot. Above 2200°F the data indicate that the temperature for equal 1-hr rupture-strength values is about 100°F higher for B-66 than that for the next strongest alloy. The 10-hr rupture data indicate that three alloys have about equal strength values: B-66, FS-85, and Cb-752. It is unfortunately not possible to make comparisons between these three alloys in terms of 100-hr rupture strength, as data were not available for FS-85 or B-66.

Only two sets of creep data were found for Cb alloys. Data were recently reported by Boeing for Cb-752 tested at 2000°, 2500°, and 3000°F. Stress vs creep-time curves for constant values of total strain of 0.2, 0.5, and 1.0% at 2500°F are shown in Fig. 6.

More recently a paper by Hall and Titran[21] reported results of some preliminary creep tests on nine Cb alloys. The objectives of the over-all program are directed to conducting very long tests (10,000 hr) in very high vacuums at temperatures between 2000° and 2600°F. Initial screening tests were conducted at 4000 psi on the nine alloys in the recrystallized condition for a 300-hr creep period to identify the more promising alloys. The results of these tests are summarized in Table 5 in terms of total strain after 300 hr at 2000°, 2200°, and 2400°F. These particular results at a creep stress of 4000 psi indicate FS-85 and D-43 to be most creep resistant at 2200°F and D-43 to be the most creep resistant at 2400°F.

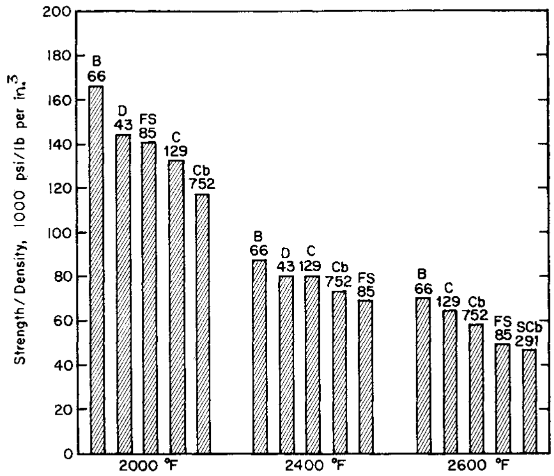

Fig. 3 Ultimate-tensile-strength to density ratio for five top columbium alloys, from Fig. 1, for test temperatures of 2000°, 2400°, and 2600°F.

Alloys FS-85, D-43, B-66, and Cb-752 were selected by Hall and Titran for more extensive study for longer creep times and other stress levels. These data have not as yet been reported.

Molybdenum Alloys

Although there are several molybdenum alloys in the experimental or developmental stages which show outstanding high-temperature strength properties, only two Mo-alloys are commercially available today: Mo-0.5Ti and Mo-0.5 Ti-0.08 Zr (TZM). Figure 7 shows the ultimate tensile strength of these alloys as a function of test temperature. Included for comparison purposes are data for both commercial powder-metallurgy Mo and arc-cast Mo. As is evident from Fig. 7, TZM has excellent high-temperature tensile strength, about twice that of Mo-0.05 Ti between 2400° and 3000°F. Problems still persist with Mo and Mo-alloys because of limited ductility at ambient temperature due to recrystallization during exposure at elevated temperatures or as a result of welding. Additional problems arising from ductile-to-brittle transitions are encountered during fabrication, application of protective coatings, and assembly of coated parts. These problem areas have placed Mo-alloys at a disadvantage for

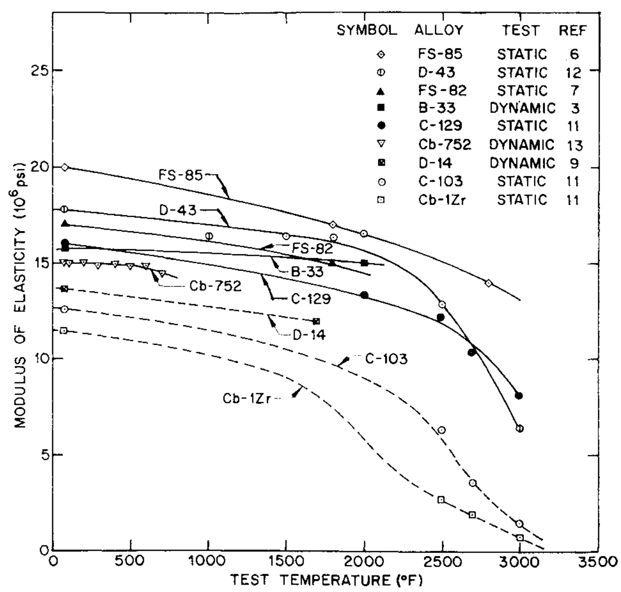

Fig. 4 Modulus of elasticity vs test temperature for commercial columbium alloys.

Table 5 Creep properties of columbium alloys[a]

Alloy	Test temperature, °F 2000°	2200°	2400°
FS-85	[b]	0.26	3.35
D-43	[b]	0.30	1.26
B-66	0.18	1.42	12.33, 124 hr
Cb-752	0.53	3.57	12.00
C-129	1.05	3.64	[c]
Cb-1Zr	1.10	15.11, 235 hr	[c]
D-14	3.05	8.40, 72 hr	[c]
B-33	8.60	33.9, 255 hr	[c]

[a] Values are for total strain after 300 hr at a stress of 4000 psi on 0.030-in.-thick recrystallized sheet material.
[b] Not evaluated at 2000°F.
[c] Not evaluated at 2400°F.

certain structural applications despite their superior strength at elevated temperatures.

Tantalum Alloys

Three tantalum alloys are available as commercial sheet products: Ta-10W, Ta-8W-2Hf, and Ta-12.5W. The ultimate-tensile-strength to density ratio for these alloys is plotted as a function of test temperature in Fig. 8. Three sets of data representing different material conditions are given for Ta-8W-2Hf alloy. At temperatures below 2400°F the top curve (●) represents the Ta-8W-2Hf alloy after a low-temperature stress relief (1 hr at 2000°F); the next lower curve (△) is for a higher-temperature stress relief (3 hr at 2250°F); and the next curve (○) represents Ta-8W-2Hf in the recrystallized condition. The latter curve falls very near the data for Ta-10W alloy in the stress-relieved condition (3 hr at 2250°F). Above 2400°F there is little difference in the three curves for the Ta-8W-2Hf alloy or for the three alloys, except in the case of the lowest curve reported for Ta-10W alloy. These alloys are all characterized by excellent low-temperature ductility, excellent fabricability, and good weldability.

Summary of Property Data for the Various Alloys

Figure 9 summarizes the ultimate tensile strength, compensated for density, for the highest-strength Cb-base and Mo-base alloys and for the Ta-8W-2Hf alloy. Ta-8W-2Hf was selected for this plot because data for this alloy were available over a larger temperature range than for Ta-10W, although both alloys exhibit about equal tensile properties. Data for commercial unalloyed powder-metallurgy tungsten sheet are also included; no commercial tungsten sheet alloy is available. TZM shows clear superiority over all other commercial refractory metal sheet materials up to 3000°F. For about an 800°F range, up to approximately 3400°F, Ta-8W-2Hf and P-M W are about equal. Above 3500°F, P-M W is superior to the other materials.

Figure 10 summarizes short-time creep-rupture data compensated for density for selected alloys, all in a stress-relieved condition. The data are particularly interesting, since the tests were conducted using similar test conditions and equipment at the Marquardt Corporation. Directly comparable creep-rupture data for a Cb-alloy were not available. TZM is again clearly superior up to temperatures of 3000°F. Above 3500°F unalloyed tungsten sheet exhibits the highest creep strength of any commercially available sheet material.

The summary of property data presented in this paper had by necessity to be limited to a fairly brief treatment, covering primarily ultimate-tensile-strength and stress-rupture data. A more complete review of property data is available in a forthcoming book[22] which includes available yield strength, ultimate tensile strength, elongation data, ductile-brittle transition-temperature data, and thermal-property data for the commercial alloys covered in this paper.

Table 6 Parts, materials, and processes used in production forming of refractory metal sheet

Part Classification	Materials and Processes[a] Mo-0.5Ti-0.1Zr (TZM)	Cb-5Zr (D-14)	Cb-10Ti-5Zr (D-36)	Tungsten (unalloyed)
Singly Curved				
Straight Section	1,3	1	1	
Straight Flange	1,2	1,2	1,2	
Single Contour Curve	3	6		
Contour Flanged				
Stretch Flange	2,8	5,8	8	3,9
Shrink Flange				
Reverse Contour	7	7	7	
Curved Sections				
Uniform		4		9
Non-Uniform				
Compact Curved				
Deep Recessed				
a. Vertical Wall				
Cup				
Tubular				3,9
Box				
b. Sloping Wall				
Closed				9
Open				
Semi-Tubular				3
Shallow Recessed				
Double Curvature	3			3
Dish Shaped				
Beaded	3		2	
Corrugated	1	1		

[a] Forming Processes
1 Brake Form; 2 Press Form; 3 Hammer Form; 4 Stretch Form; 5 Rubber Form; 6 Roll Form; 7 Joggle; 8 Dimple; 9 Spinning

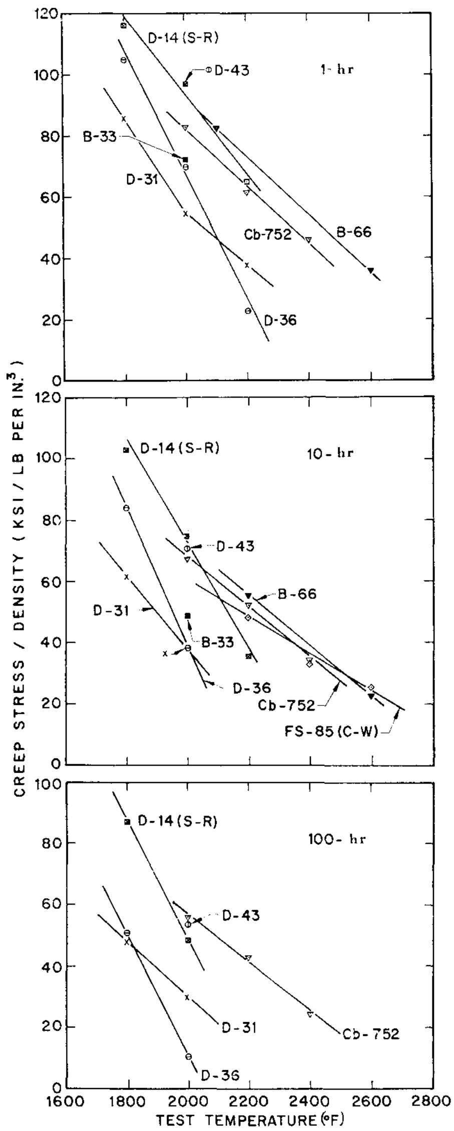

Fig. 5 Creep stress-density ratio for 1-, 10-, and 100-hr rupture life for various columbium alloys.

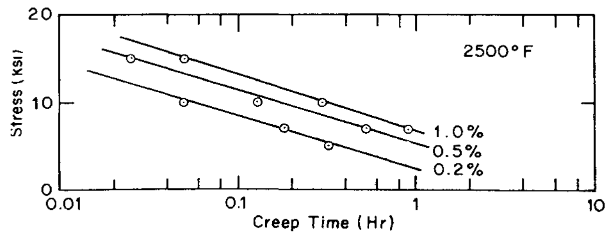

Fig. 6 Stress vs creep time for constant values of total strain at 2500°F for Cb-752 (Ref. 8).

Production Forming

Much of the so-called production or shop-forming of refractory metals would still be classed as a laboratory operation when compared with production forming of more conventional sheet-metal structures. Production rates are low, and only limited quantities of any particular item are manufactured. Each piece is individually handled, and close adjustment and control of all details of the forming processes are maintained.

The parts, materials, and processes used in preproduction and shop-forming of aerospace components are summarized in Table 6. Parts are classified on the basis of contour, as proposed by Sachs.[23] The majority of sheet-metal parts fit readily into this system of classification, and the system is applicable to all metals and alloys. Laboratory and pilot production studies have demonstrated that all of the basic parts listed in Table 6 can be produced from refractory metal and alloy sheet. Of the 19 different basic types of parts, at least 12 currently are being formed on a commercial basis.

Although 23 different compositions of refractory metal sheet materials are available commercially, only four are being used in quantity to manufacture components for aerospace applications: Mo-0.5Ti-0.08Zr (TZM), Cb-5Zr (D-14), Cb-10Ti-5Zr (D-36), and unalloyed tungsten. These materials have been selected on the basis of ability to meet service and reliability requirements, availability in required quality and quantity, and fabricability. In the case of molybdenum- and columbium-base alloys, coatability for oxidation protection and the reliability of coating systems also have been an important factor in materials selection. The parts formed from these materials are relatively small and simple in shape, compared with the more conventional aircraft sheet-metal parts.

Most of the parts being made from molybdenum- and columbium-base alloys fall into the classification of singly curved parts and are produced by simple bending and straight flanging operations. Many of the parts are small clips, channels, and angles which are formed by bending sheet and strip over a small radius along straight lines. The largest parts are flat, beaded, or corrugated flanged panels; single-contoured curves; and stretch-formed corrugated curves. Of these, the largest single part can be formed from a 12-in.-wide × 36-in.-long piece of sheet. Only the thinner-gage sheet materials, 0.010–0.040-in. thick, are used in making these components.

The parts being formed from tungsten sheet represent an entirely different area of technology. In general, they are shallow and deep recessed parts which are very difficult shapes to form. Several of these have compound or double curvatures and semicircular or circular flanges. All of the parts have curved surfaces and cannot be formed along straight lines. Most of the tungsten parts are small and can be formed from sheet up to 15-in. square. The use of shear spinning whereever possible has greatly reduced the need for large-size sheet in making these parts. Components are produced from heavy sheet and light plate with a thickness range of 0.060–0.5 in.

With the exception of blank preparation, all fabrication of the molybdenum-base alloy (TZM) is conducted at elevated temperatures to avoid brittle fracture. The ductile-to-brittle transition temperature of this material in tension and in bending is below room temperature, and formed parts of good quality can be made at room temperature. However, due to high and variable strain rates in shop-forming processes, elevated temperatures are used to decrease the probability of brittle fracture. Most operations are conducted at 100°–350°C using preheated dies and blanks. In general, successful forming of molybdenum requires the use of perfectly sound blanks. All blank preparation is done at room temperature, with chemical blanking being the preferred method. Difficulties with edge cracking and delamination have led to a general reluctance to use shearing, abrasion, or sawing for this critical operation. All forming of the two columbium-base alloys is conducted at room temperature. Blanks are prepared by shearing or punching without danger of cracking or delamination, although chemical blanking often is used to prepare irregular shapes.

The relative simplicity of parts formed from molybdenum and columbium alloys permits most operations to be conducted on vertical brake presses or drop hammers using steel dies. Straight flanges are made by press or hammer forming in steel dies and reverse contour (offset) flanged by joggling. In forming flanges on columbium alloys, rubber staging often

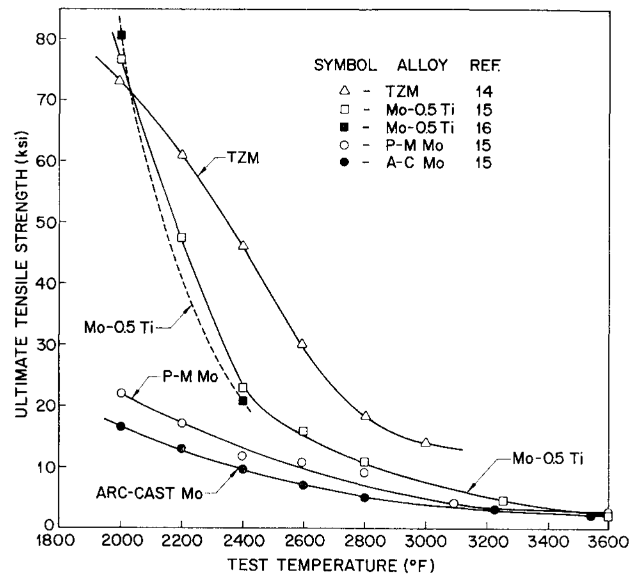

Fig. 7 Ultimate-tensile-strength vs test temperature for TZM, Mo-0.5 Ti, and for powder-metallurgy and arc-cast molybdenum, all in the stress-relieved condition.

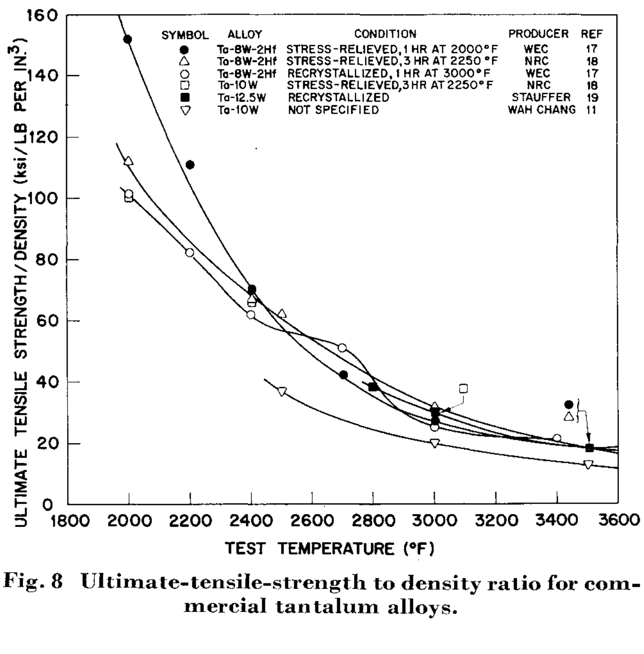

Fig. 8 Ultimate-tensile-strength to density ratio for commercial tantalum alloys.

is used. Rubber forming is used to produce a curved, flanged part from the D-14 alloy. A limited amount of stretch forming is used, primarily to make curved sections in columbium-base alloys. Stretch forming has not been successful in making compound curves with the TZM alloy and these parts are made by hammer forming.

With molybdenum and columbium parts where stretch flanging or forming is involved, a maximum of 10% stretch (elongation) is permitted in part designs. The most common type of stretch flange is the dimple, which is used widely to permit mechanical fastening of components. Conventional dimpling machines are used for this purpose. Holes for dimples in molybdenum are drilled before forming, and mechanical support and power feed are used to minimize edge cracking.

Spinning (shear and conventional) and drop-hammer forming are the principal methods for working sheet tungsten. All work is done with heated tools and blanks to avoid brittle fracture. Most forming is conducted at red heat with blank temperatures from 650° to 1100°C. Dies and parts are heated with oxyacetylene torches or quartz lamps, and all forming is done in air. In this temperature range, unalloyed tungsten works in a manner similar to that of mild steel at room temperature, and severe forming operations are possible. Complex-compound curved and shallow or deep recessed parts are formed readily by drop-hammer methods. In shear spinning, which is used to produce most symmetrical parts, material often is reduced 50% in thickness in one pass. As with molybdenum, success in forming requires the use of sound blanks. The presence of edge cracks or delaminations can result in failures, particularly on heating for working or on initial loading. Blanks usually are cut by electrical discharge machining or are hot-sheared. Abrasive or friction cutting work well with some sheet materials but result in cracks in others.

The development of sheet-metal forming practices has progressed simultaneously with the development of refractory metal and alloy sheet. This rather unfortunate combination of circumstances has created a number of transient problems in forming. As sheet-manufacturing processes and quality have improved, the response to forming and handling requirements has changed. The fabricator usually finds greater ease in forming present materials in comparison with materials that were available six months to a year ago.

Problems in production forming today are more likely to arise from exacting requirements for reliability than from any basic limitation of the material. The manufacturer is concerned primarily with the ability to produce useful parts on a repetitive basis with a minimum number of rejects. The ultimate failure in any formed part is a crack that destroys structural integrity. In present applications, even the most

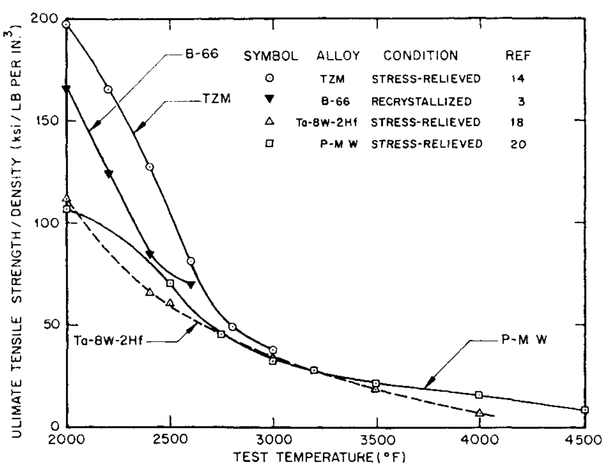

Fig. 9 Ultimate-tensile-strength to density ratio for selected refractory metal alloys and unalloyed tungsten between 2000° and 4500°F.

minor cracks cannot be tolerated, and all specifications call for materials that are free from surface or internal defects. Surface and edge cracks are particularly detrimental in parts that are to be coated for oxidation protection. They result in points of weakness in the coating that can lead to localized failure and rapid destruction of the part in service. In addition to cracking, the usefulness of a part may be destroyed by local thinning or necking in regions of tensile stress, or by buckling and wrinkling in regions of compressive stress. Another troublesome problem arises from failure to maintain dimensional tolerances as a result of springback and residual stress.

The least number of problems are encountered in working with the low-strength columbium-base alloys. This fact accounts for the preference for the use of columbium wherever possible and a general reluctance to use molybdenum and tungsten. The largest single problem encountered in working with molybdenum and tungsten is brittle fracture by cracking and delamination. The low-strength columbium-base alloys are resistant to failure of this type unless contaminated with carbon, oxygen, or nitrogen. Cracks and delaminations are two distinctly different defects in terms of mode and mechanism of failure. Cracks are brittle fractures aligned normal or at an inclined angle to the surface. They are caused by tensile stresses in the plane of the sheet and occur on stressing at temperatures below the ductile-to-brittle transition for the stress system and strain rate involved. Delamination is a form of brittle fracture in which cracks are aligned parallel to the surface (in the plane of the sheet) and separate the sheet into two or more layers. This results from a weakness in the plane of the sheet and occurs when material is stressed in the thickness direction. Sheet can delaminate over a wide range of temperatures, even above the transition temperature as defined by tensile or bend tests.

Cracking and delamination occur most frequently in cutting, shearing, punching, and drilling operations used to prepare blanks for forming. They usually occur at edges and corners and are difficult to detect visually. The presence of these defects in starting blanks results in failure by splitting or complete fracture when defective regions are stressed during forming. The defects also originate in forming, particularly in flanging, dimpling, and reverse bending. With tungsten, they may occur during initial loading in spinning or die forming. The problem has been alleviated to some extent by metallurgical control; however, metallurgical factors that reduce the tendency to delaminate increase the tendency to crack.

The approach to solving this problem has been largely mechanical; forming operations that are likely to cause failures are either avoided or carefully controlled. Chemical blanking and electrical-discharge machining are used in preference to sawing, shearing, or drilling in preparing blanks for forming. Where drilling must be done, the situation has been improved by power feed, adequate mechanical support, and proper machine settings. Reverse bending and excessive compression in the plane of the sheet are avoided whenever possible. All forming operations are conducted well above the normal tensile or bend transition temperature, and all details of the forming process are closely adjusted and controlled.

The problem of cracking and delamination is particularly critical in working with tungsten where the minimum forming temperature depends on grain size and shape, amount of prior cold work, and percent of recrystallization. Practices can be devised for forming successfully any grade of sheet, from fully recrystallized to fully cold worked; however, forming parameters may be quite different for the various grades. Brittle fracture problems arise when practices established for one grade of material are used to form a different grade or if the sheet material has a mixed grain structure and/or is partially recrystallized. Tungsten sheet is not supplied today in a standard uniform grade from source to source. Even from one source, metallurgical characteristics vary markedly with sheet thickness and often are not closely controlled for a given thickness of material.

Minor difficulties in localized deformation and wrinkling have been encountered in forming tungsten. These are not problems with molybdenum and columbium at present, since stretch is limited to 10% maximum and compression is avoided in most parts that are made. In tungsten, design and operation of the forming dies probably have as much to do with these problems as does any characteristic of the material. The problems generally are solved by providing proper die clearance, staging and contours, and mechanical support.

Dimensional stability is a minor problem encountered with all materials. This is usually the result of over- or under-forming, variable springback, and relief of residual stress. A large share of the problem is due to over- or under-forming as a result of inadequate tooling. The limited number of parts to be produced often does not justify the manufacture of the more expensive hardened tools and dies. The use of less durable tooling may result in dimensional variations, but these can usually be corrected. With molybdenum, however, reverse bending to correct an over-formed part can cause delaminations. Springback has been found to vary within sheets and from sheet to sheet, but in general this is not considered too serious a problem in parts made today. Dimensional changes on stress relief are found primarily with the columbium and molybdenum alloys formed at or near room temperature. In some cases, the stress-relief treatment is not complete, and further changes in dimensions occur during deposition of a pack-cementation type of coating. This problem is a major concern, since dimensions of coated parts often cannot be corrected without cracking the coating or the part.

Summary

Substantial progress has been made during the past two years in the development of refractory metal and alloy sheet suitable for aerospace applications. Today's designer has at his disposal 23 different alloys, commercially available as wide sheet, from 11 domestic producers. The material is available in sufficient size, quantity, and quality for most anticipated applications. Cost remains high, and considering the number of alloys that may compete for a given application, tradeoffs between properties and price on a density- or weight-compensated basis should be given more consideration.

The Mo-0.5Ti-0.08Zr (TZM) alloy exhibits clear superiority over all other commercial refractory metal alloys at temperatures to 3000°F on the basis of density-compensated tensile strength and creep-rupture behavior. Above 3000°F, the Ta-8W-2Hf (T-111) and powder-metallurgy tungsten are superior to all other commercial alloys. Problems with brittle fracture at ambient temperature have placed molybdenum and tungsten at a disadvantage, despite their superior strength properties. Columbium-base alloys are approaching the strength characteristics of TZM at temperatures to 2600°F and as a result of their excellent fabricability are receiving more attention. The largest variety of alloy compositions and widest range of properties are available in this class of alloys. A number of Cb alloys possess attractive high-strength properties in the temperature range 2000°–2600°F. The alloys which appear to be the most interesting in this respect are B-66, Cb-752, D-43, and FS-85. Sufficient data are not available to indicate the spread in properties which might be expected for different lots of any one of these alloys. In addition, one alloy may possess superior strength under one set of test conditions of temperature, stress, and time, whereas another alloy may be superior for a different set of conditions. Meaningful design data (guaranteed minimums) are lacking for nearly all of the refractory sheet alloys, and considerable effort is required fully to characterize commercial materials.

Virtually all types of sheet-metal parts have been or are being produced from these materials. Commercial production in quantity at present is limited to relatively few Mo- and Cb-base alloys and unalloyed tungsten. All types of sheet-metal forming processes are being used successfully with a minimum of difficulty. Columbium and tantalum alloys, by virtue of their low ductile-to-brittle transition temperature, offer the greatest ease of forming and present the least number of problems.

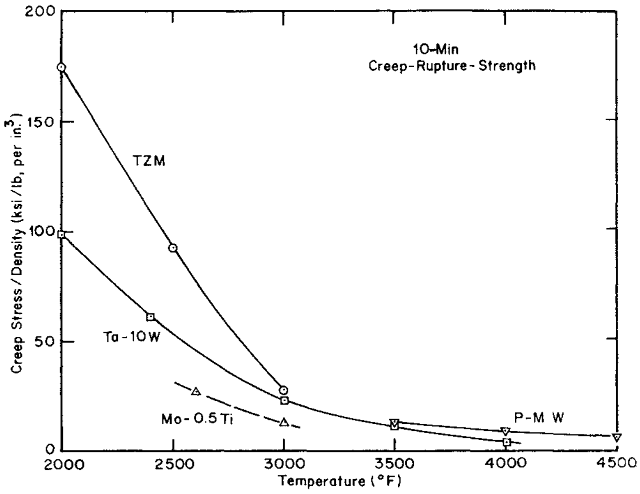

Fig. 10 10-min creep-rupture strength-to-density ratio for selected refractory metal alloys and unalloyed tungsten between 2000° and 4500°F (Ref. 20).

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