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METAL-METAL LAMINAR COMPOSITES FOR HIGH-TEMPERATURE APPLICATIONS

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HIGH-TEMPERATURE APPLICATIONS

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ABSTRACT

A study was conducted to obtain indications of the potentialities of laminar metal-metal composites for elevated temperature use. Most of the composites consisted of multiple layers or laminae of tungsten alternated with laminae of Nichrome V, a ductile, weaker but oxidation resistant alloy. Composites with 50 volume percent of each phase made from 0.0025 cm (0.001 in.), 0.0125 cm (0.005 in.) or 0.050 cm (0.020 in.) laminae, were tested in tension and stress rupture at temperatures of 871 and 1093° C (1600 and 2000° F) and in impact at 23 and 524° C (73 and 975° F). A tension and a short time stress-rupture test was conducted on specimens of 77 v/o W-Re-Hf-C/23 v/o Inconel Alloy 600 at 1093° C (2000° F).

The highest tensile strengths obtained for tungsten/Nichrome V specimens were 600 MN/m² (87 000 psi) at 1600° F and 448 MN/m² (65 000 psi) at 1093° C (2000° F). Highest 100-hour stress-rupture strengths for tungsten/Nichrome V at the same respective temperatures were 387 MN/m² (56 000 psi) and 121 MN/m² (17 500 psi). The W-Re-Hf-C/Inconel 600 composite had a tensile strength of 683 MN/m² (99 000 psi) at 1093° C (2000° F).

Maximum stress-rupture strength at 1093° C $(2000^{\circ}$ F) for the tungsten-Nichrome V laminar composites were 131 and 100 percent of rule-of-mixtures values for 1 and 100 hours, respectively. These values compared favorably with corresponding values for fiber reinforced composites reported in the literature. The strengths of the laminar composites generally equaled or exceeded the values predicted by the rule-of-mixtures. Since composite strengths were related to constituent strength, development of stronger sheet or foil materials should permit production of stronger composites.

INTRODUCTION

The potential of fiber composite materials has been demonstrated most graphically by the development of practical high strength, high modulus-to-weight ratio, fiber reinforced materials. Although principal developmental efforts have been for lightweight structural materials, some very promising composites with high stress-rupture strength at 1093° C (2000° F) and above have been produced from refractory metal fibers combined with superalloy matrices (1,2).

Laminar sheet or foil composites may also have potential for structural use and for use at high temperatures, but relatively little has been done to study such materials. Sheets or foils of metal are known to increase in strength as their thickness is decreased, analogously to the manner in which fibers increase in strength as their diameter is decreased by drawing. If thinner, strong sheets or foils could be fabricated into composites without significant losses in sheet or foil properties, the resultant composites might be expected to have greater strength and other more desirable qualities than bulk monolithic materials. Further, if layers of strong thin refractory metal foils could be combined with similarly thin layers of weaker, more ductile, and high temperature corrosion-resistant materials, composites with useful properties at high temperatures should result. Generally, such composites should be superior at high temperatures to superalloys in strength, modulus of elasticity, and strength or modulus-to-weight ratios. High strength laminar composites with near in-plane isotropy, useful for applications involving biaxial stress conditions, can be envisioned for turbine disks, low and high temperature pressure vessels, tubes and combusion chambers. Other applications might include structural panels, turbine and compressor blades for gas turbines, and hollow drive shafts, to name a few.

Previous work with laminar composites has been largely confined to clad materials or low temperature structural materials. Clad sheet metal has been produced for years. Copper clad stainless steels and corrosion resistant materials clad over noncorrosion resistant materials are examples. Efforts to study multilayered laminar composites have only recently been made (3,4,5). Impact studies (6) with mild steel sheets interlayered with soft solder, copper or silver were reported. Weak interfaces were shown to act either as crack arresters or crack dividers, but in each case, they acted to increase impact resistance relative to materials in bulk form. Studies of beryllium/ aluminum laminar composites and beryllium/titanium/aluminum composites have been reported (7). The results showed that lamination provided a means for obtaining improved notched impact resistance and resistance to cracking of laminar composites consisting largely of beryllium. Ballistic impact resistance was also improved relative to that of pure beryllium. Tensile properties of the latter composites were close to those expected from the rule-of-mixtures.

Although composites such as those mentioned should have potential as high modulus, high-strength structural materials, laminar composites

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have not been considered for their high temperature potential. Accordingly, this investigation was conducted to obtain indications of the potentialities of laminar metal-metal composite materials for high temperature use. The program was concerned almost entirely with tungsten/ Nichrome V. The tungsten used for the stronger of the two laminae comprising the composites has high-temperature strength well above that of most superalloys, while the Nichrome V was chosen to represent a ductile high temperature, corrosion-resistant matrix material. A limited number of laminated composites of a strong tungsten alloy in combination with Inconel 600 was also investigated.

The test temperatures used for the tensile and stress-rupture tests were chosen to cover; namely, use-temperatures of practical interest for gas turbine operation 871° C (1600° F) and 1093° C (2000° F). Disks and some turbine blades operate at, or close to, the lower of the above temperatures, while the higher temperature is a material target temperature for advanced gas turbine blades. Whereas almost no metallurgical reaction occurs between the tungsten and Nichrome V phases at the lower temperature, significant reactions occur at the higher temperature. Thus, the system chosen for these studies may be considered to be a model system in the sense that its behavior can be considered representative of nonreactive and reactive combinations of materials. Composites with alternating layers of tungsten and Nichrome V foils or sheet were vacuum hot pressed. Most of the latter composites contained laminae of equal thickness; the thicknesses of laminae used were 0.0025, 0.0125, and 0.050 cm (0.001, 0.005, and 0.020 in.). Tensile and stressrupture tests at 871° C (1600° F) and 1093° C (2000° F) were performed on the tungsten/Nichrome V composites, containing nominally 50 v/o tungsten. Impact tests were conducted at room temperature and 524° C (975° F) : the latter temperature being above the DBTT for tungsten.

MATERIALS APPARATUS AND PROCEDURE

Lamina Sheet and Foil Materials

The nominal composition and thicknesses of the sheet and foil stock utilized for the composites and sheet or foil specimens of this study are listed in Table I. Commercially made sheet and foil (lamina) were obtained as rectangles 10×15 cm (4×6 in.). The 15 cm (6 in.) length was in the direction of the major rolling operation. The tungsten alloy (W-Re-Hf-C) was rolled at the Lewis Research Center. Because of the difficulty in rolling this material, only about 15 cm (6 in.) of usable material (enough for two specimens) was obtained from a total of about 75 cm (30 in.) of strip. Inconel Alloy 600 was used with the W-Re-Hf-C alloy, since preliminary compatibility screening studies indicated that it was the least reactive of several alloys with the tungsten alloy.

Tensile and Stress-Rupture Specimens

Specimen configurations. - Sketches of the specimens used for ten-

TABLE I: COMPOSITION AND THICKNESS OF MATERIALS USED FOR SPECIMENS

Material	Nominal Composition	Thic	kness	Source
		cm	in.	
Tungsten Tungsten Tungsten Tungsten	99.9+ 99.9+ 99.9+ Unknown	0.0025 .0125 .050 .25	(0.001) (.005) (.020) (.10)	National Research Corp. General Electric Co. General Electric Co. Unknown
Nichrome V	80Ni,20Cr	.0025 .0125	(.001) (.005)	Driver Harris Driver Harris
Tungsten Alloy	W.4Re, 0.35Hf,0.02C	.0875	(.035)	NASA-Lewis Research Center
Inconel Alloy 600	7Fe,15Cr Bal. Ni	.0125	(.005)	International Nickel Co.

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sile and stress-rupture testing of sheet and foil and of laminar composites are presented in Fig. 1. The constitution of composite specimens is indicated in Table II. Methods of fabricating or consolidating composites and machining of sheet or foil and composite test specimens will be described subsequently.

Sheet or foil lamina specimens. - The test conditions are given in Tables III and IV along with results that will be described later. Most of the rectangular sheet stock (l2.7 cm \times 2.54 cm (5 \times 1 in.)) from which test specimens were ultimately obtained were cut from the as-received rectangular stock so that the long dimension of the specimens were oriented in the direction utilized to roll the sheet or foil stock. Some of these specimens were annealed as indicated in the table (871° C (l800° F) for four hours in vacuum) to simulate the thermal cycle used in the consolidation of the laminar composite specimens. Two 0.050 cm (0.020 in.) specimens were cut from the original stock so that their long direction was transverse to the rolling direction.

<u>Composite specimens.</u> - Tensile and stress-rupture composite specimens, Table II, were made by stacking alternating layers (lamina) of the strong phase, tungsten, with the weaker phase, Nichrome V, and vacuum hot pressing the stack to diffusion bond the layers into a continuum. The tungsten/Nichrome V composite contained laminae of equal size; composites of three thicknesses of laminae were studied, 0.0025, 0.0125, and 0.050 cm thick (0.001, 0.005, and 0.020 in.). The W-Re-Hf-C/Inconel 600 specimens were made similarly except that single layers of the tungsten alloy were sandwiched between layers of Inconel 600. In all tungsten/Nichrome V composites the external layers were Nichrome V.

A vacuum hot press was utilized to consolidate the composite materials. The hot press had temperature, pressure, and vacuum capabilities of 1600° C (3100° F), 55 MN/m² (8000 psi), and 0.133 N/m² (10⁻³ torr), or better, respectively. Hot pressing was first done on a single specimen at a time; stacks to be consolidated were vacuum hot pressed at 982° C (1800° F) for four hours at a pressure of 28 MN/m² (4000 psi). After the four-hour hold at temperature, the heater power was turned off but the specimen pressure and chamber vacuum were maintained; the unit was allowed to cool overnight. The hot pressing conditions were found to produce a good bond while avoiding visible interdiffusion resulting from consolidation. Later, a method to hot press a number of specimens simultaneously was devised. Thin rectangular sheets of mild steel having twist tabs were used to hold and align stacks of foil. The sequence followed for making one laminar composite specimen is illustrated in Figs. 2(a), 2(b), and 2(c). Up to six specimens were pressed at one time (fig. 2(d)). Each was wrapped in the steel tabs. Note that an aluminum oxide cloth was placed between the laminae contacting the steel twist-tabs. This was done to prevent bonding of the tabs to the test specimens. After hot pressing, the tabs were cut to remove the six individual specimens. All specimens were pressed between flat surfaces without the use of external restraints (e.g., dies).

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ABLE II:

Tensile and Stress-Rupture Specimens

Remarks			,			Sandwich specimen	Diffusional effect	study
Volume percent tungsten	or tungsten alloy	49	49	48	50	77	16	
:	kness in.	(100.0)	(100.)	(.005)	(020) (010)	(.005)		
Matrix constituent	Thic) cm	0.0025	.0025	.0125	.05 .025	.0125	 	
	No. of laminae	25	21	긔	ർ.മ പെര	N	:	1
ituent gsten	cness in.	(100.0)	(100.)	(.005)	(.020)	(.035)	(.005) ^c	(.0055) ^d
ing const en or tur alloy)	Thick cm	0.0025	.0025	.0125	.05	.0875	.0125	.0139
Reinforc (Tungst	No. of laminae	24	20	IO	N	Ч	Ч	
Constit- uents		Tungsten/ Nichrome V			\rightarrow	W-Re HfC/ Inconel 600	Tungsten arc	sprayed with Nichrome V
Spec. type		Ъа	ЪЪ	0	Ю	4	ហ	

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^aThe inner lamina was 0.05 cm (0.020 in.). ^bThe outer laminae were 0.025 cm (0.010 in.); in both cases (a) and (b), the matrix laminae were diffusion bonded 0.0125 cm (0.005 in.) Nichrome V foil. ^cOriginal dimension of tungsten. ^dAfter diffusion heat treatment.

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RESULTS	ос (тес
TENSILE III: TENSILE AND STRESS-RUPTURE	FOR SHEET AND FOIL SPECIMENS AT 871

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Lwife, hours		10.0 ⁸ 56.0 ⁸ 9.0 ⁸ 500.0 ⁸ , ^b	2.3 14.7 45.0 570.8 3.0	6.6 ^a 143.2 ^a 65.8 268.3 ^a 369.6
ress	10 ³ psi	(011) (011) (08) (08)	(100) (90) (75) (62.5) (95)	(158) (158) (158) (158) (158)
St	MN/m ²	759 690 621 552 	689 621 430 655	621 517 517 517 476
tensile Meth	lo ³ psi	(156) (156)		(127) (97) (97) (97)
Ultimate strer	MN/m ²	699		877 669
Condition		As-received	As-received Annealed for 4-hrs 982° C (1800 [°] F)	As-received Annealed for 4-hrs 982° C (1800° F) As-received As-received As-received As-received
	ness in.	(100.0) (1001) (1001) (1005) (1005) (1005)	(.005) (.005) (.005) (.005)	(.020) (.020) (.020) (.020) (.005) (.005)
or foil	Thick cm	0.0025 .0025 .0025 .0025 .0125 .0125	.0125 .0125 .0125 .0125 .0125	.050 .050 .050 .050 .0125 .0125
Sheet	Material	Tungsten Tungsten Tungsten Tungsten Tungsten Tungsten	Tungsten Tungsten Tungsten Tungsten Tungsten	Tungsten Tungsten Tungsten Tungsten Tungsten Tungsten Nichrome V Nichrome V

^aTested at commercial laboratory. ^bTest discontinued.

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OBTAINEI	00 F)
RESULTS	5° C (200
RESS-RUPTURE	CIMENS AT 1097
TENSILE AND ST	TAND FOIL SPE
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Material	Thick	aness	Condition	Ult ter	imate Isile		Stress-	rupture
	сm	in.		str	ength	Stres	2°20	Life,
				MN/m ²	.10 ³ psi	T - 7m/m	isd DO	sund
ngsten	0.0025	(0.001)	As-received	596	(98)	1	1	8 3 8 1 1
ngsten	.0025	(100.)		1 1 1	 ! 	414	(09)	5.6
ngsten	.0025	(100.)		[]]	1 1 1	414	(60)	Broke on loading
ngsten	.0025	(100.)		1 1 1	1	345	(20)	194.5
ngsten	.0025	(100.)		L I I	1 1 1 1	345	(20)	3.4
ngsten	.0025	(100.)		 	 	310	(45)	Broke on Loading
ngsten	.0025	(100.)		1 1 1	l 1 1	276	(40)	594.0
ngsten	.0125	(300.)	-	589	(82)	1		6 1 1 1
ngsten	.0125	(300.)		584	(82)	1		8 1 1 1
ngsten	.0125	(.005)		i I I	 	414	(09)	1.1
ngsten	.0125	(.005)		 	1 1 1 1	276	(40)	91.3
ngsten	.0125	(.005)		 	 	241	(32)	240.0
ngsten	.0125	(.005)		1 1 1	1 1 1	241	(32)	240.6
ngsten	.050	(020)		569	(82)	t t t		
ngsten	.050	(020.)	_	548	(80)	8 1		E \$ 1 1 1
ngsten	.050	(.020)		 	- 1 1 1 - 1	310	(42)	.16.2
ngsten	.050	(020.)		1 	 []]	276	(40)	29.2
ngsten	.050	(020)		 	1 2 1	241	(35)	149.0
ngsten	.050	(020)		1 1 1		207	(30)	149.1
ngsten	.050	(020.)		 	1 1 1 1	276	(40)	39.0 ^a
ngsten	.050	(020)	>		1 1 1	241	(35)	103.2 ^a
ngsten	.050	(020)	Annealed 4-hrs	1 T I	1 1 1	276	(40)	62.2
			982 ⁰ C (1800° F)					
ngsten	.050	(020.)	Annealed 4-hrs	r 1 1	1 8 1 1	241	(35)	19.5
				•				
chrome V	.0125	(300.)	As-received	18	(2)	1 1] 1	1 - 1 - 1 -	1 1 1 1
chrome V	.0125	(.005)	As-received	1 	8 1 1 1	TO	(2)	22.0
ransverse	orientat	ion.						

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Dimensions of composites, measured with a micrometer after pressing, were equal to the sum of the individual thicknesses of the lamina comprising the composite. Metallographic examinations indicated that the relative dimensions of individual laminae were not affected by the pressing operation. Thus, volume percentages of the constituents could be determined from the numbers of foils or sheets in the composite.

Delamination during hot pressing materials of this type was evident in some of the preliminary experiments because of the differences in thermal expansivities (8) of the two materials. The 0.0025 cm (0.001 in.) laminae specimens did not delaminate; the 0.0125 cm (0.005 in.) laminae specimens delaminated in some cases at corners, while the 0.05 cm (0.020 in.) specimens frequently exhibited delaminations at the corners of the specimens. In the two latter cases, only those specimens with good bonds in the central sections of the specimens, or those specimens that did not delaminate at all, were tested. During tests, the unbonded corners diffusion bonded.

Shorter specimens were used for the laminar composites than for foil and sheet because the hot press susceptor cavity (to be described later) used to fabricate the laminar specimens was only 7.62 cm (3 in.) in diameter. Initially, tests were made on composite specimens 0.95 cm (0.375 in.) wide. Some of the specimens were so strong, particularly the 0.0025 cm (0.001 in.) laminae composites, that it was necessary to reduce the number of laminae slightly and also the width of the specimens to 0.46 cm (0.1875 in.) to permit the use of the same test fixtures.

<u>Tungsten arc-sprayed with Nichrome V.</u> - A number of tungsten lamina already machined to permit testing in stress-rupture were vapor coated with Nichrome V using a metallizing gun. These specimens were given the same thermal cycle given to laminar composites. The intent was to determine the possible effect of Nichrome V diffusion penetration on tungsten lamina strength.

Impact Specimens

Tungsten/Nichrome V laminar modified Charpy impact specimens, Table V and Fig. 3(a), were stacked and consolidated in an identical manner to that used for the tensile and stress-rupture specimens. In one case tungsten lamina 0.0125 cm (0.005 in.) and in another case Nichrome V lamina 0.0125 cm (0.005 in.) were stacked after applying resin to their ends, Table V and Fig. 3(b). The stacks were clamped and the resin cured by heating to 93° C (200° F). Bulk tungsten specimens of the same dimensions and configurations as the laminar composites were machined from 0.625 cm (0.25 in.) bar stock,

The composite impact specimens used are termed "crack divider" specimens (6). The 0.25 cm (0.10 in.) width of the impact specimens was about 1/4 that of the normal Charpy specimen. Unsuccessful attempts were made to fabricate "crack arrester" (6) specimens to the dimensions

Bond			fusion	ina	ina	1 1 1 1 1 1 1 1	specimer
	щ		Dif	Resi	Rest	1	of
TMENS	minae	kness in.	(0°05)	(0.005)	1 	8 8 8	(0.5 in.)
CT TEST SPE(Matrix 1	Matrix le	Thic cm	0.0125	0.0125	8 1 1 1 1		1.25 cm
E EJ	П	No.	디	21	1 1	1 1	ral.
N OF IMPA	uni nae	kness in.	(0,005)	1 1 1 1 1 1	(0.005)	(0.10)	The cent
STITUTION . trong lami	Strong la	Thic	0.0125	L I I I E	0.0125	0.25	t ends.
CON	01	No.	10	8	21	Ļ	led a
TABLE V:	Constituent(s)		Tungsten/	Nichrome V	Tungsten	Laminae Tungsten bulk	base resin bond not bonded
	Spec.	ad Vo	Ч	N	Ю	4	a NyLon Was

of the crack divider specimens. In such specimens where the laminae were stacked perpendicular to the direction of the notch the specimens delaminated during grinding (8) (fig. 4).

<u>Machining of specimens.</u> - The laminar composite specimens used for tensile and for stress-rupture tests were individually ground to shape as were the diffusion bonded, resin bonded and bulk impact test specimens. The lamina sheet and foil specimens used for tensile and stressrupture tests were also ground to shape, several at a time. Lamina were clamped between steel plates and the entire unit ground. Pin holes were produced in the tensile and stress-rupture specimens using electrical discharge machining. All critical dimensions obtained by grinding were specified to be within ± 0.0038 cm (0.0015 in.). The notch radii of the impact specimens had a specified tolerance of ± 0.0063 cm (0.0025 in.). The ground surfaces of the laminar specimens were determined to be 80×10^{-6} cm (32 µinches) or better. The ground surfaces of the lamina were assumed to have the same finish as the ground surfaces of the laminar specimens. The broad surfaces of the lamina had as-rolled finishes.

<u>Testing.</u> - Tensile tests of composites and lamina sheet and foil were made using a constant strain rate, screw driven tensile machine equipped with an X-Y chart recorder and a vacuum chamber. Values of strain from the recorder were used to estimate the elongation of laminar tensile specimens. Elongation measurements for sheet and foil specimens are not reported because pin holes of specimens distorted during the tests. Specimens were tested in a vacuum atmosphere of 0.0133 N/m^2 (10^{-4} torr) or better. A cross head speed (strain rate) of 0.050 cm/min (0.02 in./min) was used for the tensile tests. Both tensile and stress-rupture specimens were heated slowly to temperature over a period of three-quarters to one hour. Most of the stress-rupture tests were run at the Lewis Research Center and in machines designed and built at the Laboratory. A small number of stress-rupture tests were conducted by a commercial testing laboratory using commercial test units. The stress-rupture machines could attain a vacuum of 0.0133 N/m^2 (10^{-4} torr) or better.

Room temperature, 23° C (73° F) and elevated temperature 524° C (975° F), Charpy impact tests were conducted in air. Specimens tested at elevated temperature were heated in a furnace and quickly transferred to the impact tester. The nominal temperature at the start of the test was 524° C (975° F).

RESULTS

Tensile and Stress-Rupture Results

<u>Tungsten and Nichrome V lamina.</u> - Tensile and stress-rupture strengths of tungsten lamina tested at 871° C (1600° F) are given in Table III and those for 1093° C (2000° F) are given in Table IV. The tensile and stress-rupture data are plotted in Fig. 5 for tests conducted at 871° C (1600° F). No distinction is made in these plots

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between specimens tested commercially versus in-house laboratory tests. The 871° C (1600° F) stress-rupture strengths of the 0.0025 cm (0.001 in.) tungsten lamina were slightly greater than those of the 0.0125 cm and 0.050 cm (0.005 and 0.020 in.) tungsten lamina; the latter two having about the same stress-rupture strengths. A few specimens which were given a heat treatment of four hours at 981° C (1800° F) to simulate the laminate composite consolidation procedure have lower tensile strengths at 871° C (1600° F) compared to the as-received specimens.

Only two tests, a tensile test, and a stress-rupture test were made of 0.0125 cm (0.005 in.) Nichrome V material (Table III). The line drawn between these data points (fig. 5) is approximately parallel to the lower strength line of the scatter band of properties published for 80Ni-20Cr (9). It is assumed that the properties of other thicknesses of Nichrome V lamina; namely, the 0.0025 cm (0.001 in.) and 0.050 cm (0.020 in.) thick Nichrome \bar{V} would be approximately the same or within the scatter band of published data for 80Ni-20Cr.

Plots for tungsten and Nichrome V lamina tested at 1093° C (2000° F) are shown in Fig. 6. There are slight increases in stress-rupture strengths as lamina thickness decreases. Curves drawn through the rupture data extrapolate closely to the data points obtained in tensile tests which are plotted as 0.1-hour stress-rupture points in the figure. One specimen of the 0.05 cm (0.020 in.) tungsten lamina given the thermal treatment used for consolidation of the composites had low strength relative to most of the data for as-received material. Another had strength within the scatter of the as-received 0.050 cm (0.020 in.) material properties. Two lamina of the 0.050 cm (0.020 in.) tungsten tested transverse to their rolling direction have the same strength as the lamina tested parallel to their rolling direction indicating isotropy for the tungsten. Data are also presented for 0.0125 cm (0.005 in.) thick Nichrome V, one point being for tensile and the other for stress rupture. These material properties fall approximately in the middle of the scatter band calculated from Larson-Miller plots of cate data (9) for 80Ni-20Cr. It is assumed that data for the 0.0025 cm to 0.050 cm (0.001 to 0.020 in.) materials will fall in the scatter band shown in Fig. 6. It is important to note that the plots for the tungsten lamina for both 871° and 1093° C (1600° and 2000° F tests area straight lines over the time ranges studied. The significance of this will be discussed subsequently.

<u>Tungsten/Nichrome V composites.</u> - Tensile and stress-rupture strengths for composites tested at 871° C (1600° F) and containing different thicknesses of laminae are shown in Table VI and Fig. 7. Similarly, Table VII and Fig. 8 give strengths obtained at 1093° C (2000° F). Rule-of-mixtures strength values (ROM), to be discussed subsequently, are included in Figs. 7 and 8. The 871° C (1600° F) experimental data plot as straight lines (fig. 7). The curves for 1093° C (2000° F) break for the 0.0025 and 0.0125 cm (0.001 and 0.005 in.) laminae specimens at times less than 10 hours while the curve for the 0.050 cm (0.020 in.) laminae composites is straight (fig. 8). To compare the shapes of curves and magnitudes of the differences in rupTABLE VI: TENSILE AND STRESS-RUPTURE RESULTS OBTAINED FOR TUNGSTEN/ NICHROME V LAMINAR COMPOSITES TESTED AT 871° C (1600° F); NOMINALLY 50 v/o TUNGSTEN, AS FABRICATED CONDITION

	Ð	life hours		39.0	429.0	1 1 1 1 1	38.7	211.5	328.4	/278.2	>1075.0ª	34.7 ^b	1921.8 ^b	187.2 ^b	
	ss-ruptur	ress 10 ³ psi	 	(09)	(20)	 	(20)	(20)	(45)	(45)	(31)	(42)	. (30)	(40)	
	Stres	MN/m ²	+ 	414	345	 	345	345	310	310	215	310	207	276	
	e strength	elongation percent		1 1	1 . 1 1	4	1 1 1		1	1	- 		1		
	ate tensil	tress 10 ³ psi	(87)	1	1	(17)						 			
•	Ultime	MN/m ²	600		ł	531			 	1	!		2 		
	nae mess	in.	(100.0)	(100.)	(100.)	(.005)	(. 005)	(.005)	(005)	(.005)	(.005)	(020)	(020)	(020)	
	Lami thick	CH	0.0025	.0025	.0025	.0125	.0125	.0125	.0125	.0125	.0125	.050	.050	.050	

^aTest discontinued. ^bLaminae found to be separated and longitudinally cracked after cool down from test.

TABLE VII: TENSILE AND STRESS-RUPTURE RESULTS OBTAINED FOR TUNGSTEN/ NICHROME V AND W-Re-Hf-C/INCONEL 600 LAMINAR COMPOSITES AND NICHROME V SPRAYED TUNGSTEN TESTED AT 1093° C (2000° F)

Lau thi	minae	Ultim	ate tensil	e strength	St St	ress-rupt	ure Life,		
cm	in.	MN/m ^Ž	10 ³ psi	percent	MN/m ²	10 ³ psi	hours		
cm 0.0025 .0025 .0025 .0025 .0025 .0025 .0025 .0025	<pre>in. (0.001) (.001) (.001) (.001) (.001) (.001) (.001) (.001) (.001)</pre>	448 	(65) 	2 	MN/m ² 345 276 241 241 138 103 69	(50) (40) (35) (35) (20) (15) (10)	nours 1.0 2.2 4.8 2.5 23.5 23.8 148		
.0125 .0125 .0125 .0125 .0125 .0125 .0125	(.005) (.005) (.005) (.005) (.005) (.005) (.005)	345 	(50)	2.5	276 258 215 207 207 207	(40) (38) (31.2) (30) (30) (30)	0.5 1.1 3.7 6.4 1.7 3.7		
.0125 .0125 .050 .050 .050 .050 .050 .050	(.005) (.025) (.020) (.020) (.020) (.020) (.020) (.020)	250	(39) 	4 	138 103 207 172 137 138 103	(20) (15) (30) (25) (20) (20) (15)	16.4 46.6 21.5 ^a 31.6 ^a 70.4 ^a 111.5 ^a		
		77v/o W	-Re-Hf-C/2	3v/o Incone	1 600				
0.088/0 (0.035	125 cm /0.005 in	683 n.)	(99) ^a Arc Spraye	 d Tungsten ^b		(80)	0.8 ^a		
0.0125	(0.005) (.005)	352	(51)		276	(40)	0.3		

Tungsten/Nichrome V (Nominally 50 v/o Tungsten)

^aLaminae found to be separated and longitudinally cracked after cool down from test.

207

138

(30)

(20)

4.2

25.8

^bCoated with 0.00063 cm (0.00025 in.) Nichrome V

_ _ _

.0125

.0125

(.005)

(.005)

ture properties, the data of Figs. 7 and 8 are replotted in Fig. 9. It is evident that the laminar composite strength at 871° C (1600° F) varies inversely with the laminae thickness. The same is true at 1093° C (2000° F) for times less than about 10 hours; above this time, strength varies directly with laminae thickness.

Approximate elongations for the tensile specimens are also given in Tables VI and VII. The elongation values obtained from the 871° C (1600° F) tests appear slightly greater than those for the 1093° C (2000° F) tests for given sizes of laminae. Also the elongation increased at each test temperature, as laminae thickness increased.

<u>W-Re-Hf-C/Inconel Alloy 600.</u> - The two data points obtained for this material are also given in Table VII, one being a tensile value of 683 MN/m^2 (99 000 psi) and the other a short-time stress-rupture point strength of 552 MN/m^2 (80 000 psi) for 0.8 hours.

<u>Tungsten/arc-sprayed with Nichrome V.</u> - Tensile and stress-rupture data obtained at 1093° C (2000° F) for tungsten arc-sprayed with Nichrome V (Table VII) are superimposed on the curve for tungsten/Nichrome V composites consisting of 0.025 cm (0.005 in.) laminae, Fig. 10; the latter curve having previously been plotted in Fig. 8(b). Note that the data for the arc sprayed material correspond closely to the curve for the tungsten/Nichrome V laminae composites.

Impact Test Results

The results of the impact tests are presented in Table VIII. The room temperature impact test results for the tungsten Nichrome V composite specimens average 1.7 J (1.3 ft-lbs). By increasing the test temperature to 524° C (975° F), a temperature above the probable ductile-to-brittle transition temperature of the tungsten, the impact resistance of the composites was increased to 9.5 J (7 ft-lbs). Impact tests on bulk Nichrome V were not run since materials such as this, as well as superalloys, are known to have very high impact strengths and in the type of tests utilized would probably have bent.

In the attempt to determine the impact strength of tungsten, resin bonded tungsten foil specimens (i.e., those bonded with resin at the ends but not in the test section) were impacted and yielded strengths ranging from 2.7 to 6.1 J (2 and 4.5 ft-lbs) at 23° C (73° F). These values were greater than that obtained at 23° C (73° F) for the composite. Bulk tungsten on the other hand when tested at room temperature had a strength of 1.4 J (1 ft-1b) which was less than that of the composite. Even when bulk tungsten was tested at the higher temperature of 524° C (975° F) its impact strength was only 1.4 J (1 ft-lb) which was far less than that of the composite tested at the same temperature. The Nichrome V specimens, consisting of 0.0125 cm (0.005 in.) thick layers of Nichrome V bonded together at the ends with a resin and unbonded in the test sections of the specimen, exhibited impact strengths of 2 J (1.5 ft-lbs) to 3.5 J (2.5 ft-lbs). In the impacting process, these specimens were bent to the side by the pendulum, and thus the

TABLE	VIII:	IMP	ACT	RESUI	TS :	FOR	TUNGSI	TEN,	/NICH	IROME	V	COMPOSITE	s,
I	LAMINAE	OF	TUNC	STEN	AND	NIC	HROME	·V,	AND	BULK	ΤU	JNGSTEN	

Spec.	Constituents	Tempe	rature	Impact	strèngth		
type		oC	oF	J	ft -l b		
l	Tungsten/Nichrome V	23	(73)	1.4	(1.0)		
]	23	(73)	2.0	(1.5)		
		524	(975)	9.5	(7.0)		
	\checkmark	524	(975)	9.5	(7.0)		
2	Nichrome V laminae	23	(73)	2.0	$(1.5)^{a}$		
		23	(73)	2.0	(1.5) ^a		
		23	(73)	3.5	(2.5) ^a		
	\checkmark	23	(73)	2.7	(2.0) ^a		
. 3	Tungsten laminae	23	(73)	4.1	(3.0)		
	_	23	(73)	4.8	(3.5)		
		23	(73)	2.7	(2.0)		
	\checkmark	23	(73)	6.1	(4.5)		
4	Tungsten, bulk	23	(73)	1.4	(1.0)		
		23	(73)	1.4	(1.0)		
		524	(975)	1.4	(1.0)		
	\checkmark	524	(975)	1.4	(1.0)		

^aSpecimens were bent and deflected laterally.

values are not fully representative of the impact properties of the material. Presumably, this material 0.25 cm (0.1 in.) wide generally would not break or fracture in impact but would bend. It should be recalled that all impact tests made were conducted with crack divider type specimens. More will be said later of the significance of the impact test results.

Metallographic Results

<u>Microstructures of stress-rupture specimens.</u> - The microstructures of the tungsten/Nichrome V composites are shown in Figs. 11 and 12. In all composite specimens tested, the Nichrome V recrystallized, even at 871° C (1600° F), with the size of the recrystallized Nichrome V grains depending to some extent on the test temperature and duration, and on the thickness of the lamina. The tungsten in the specimens did not appear to recrystallize at temperatures of 871° C (1600° F) under any circumstances. Nor did tungsten recrystallize appreciably for short times at 1093° C (2000° F). Finally, the tungsten lamina within the composite failed in many cases in a ductile manner necking down severely at the fracture edge.

The specimens which fractured at 871° C (1600° F) (fig. 11), exhibited splitting of the tungsten laminae and in some cases void formations in the Nichrome V, particularly for long times of test. The tungsten exhibited considerable "neck down" ductility in most cases. Where elongation of the tungsten occurred in test, intergranular tears were produced in the Nichrome_V Similar structures; but with greater cracks, tears, and voids were observed in specimens tested at 1093° C (2000° F) (fig. 12).

<u>Macrostructures of impact specimens.</u> - Figures 13 to 16 are photographs of tested impact specimens. The photographs of the fractured specimens were taken viewing the fractured surface. Figure 13(a) is a tungsten/Nichrome V specimen fractured at room temperature which did not delaminate appreciably. This indicates that the bond between the laminae was very good. The impact specimens tested at 528° C (975° F) showed excellent ductility by exhibiting a flare-out at the struck side of the specimen (fig. 13(b)). In addition, the bond was excellent since no delamination was apparent.

The macrograph of the impacted Nichrome V material, resin bonded at the end and unbonded in the test section is shown in Fig. 14. The specimen has been very severely deformed transverse to the direction of impact; this would indicate very good ductility. The tungsten laminae specimens bonded together at the ends by resin and unbonded in the test section fractured completely and appeared brittle (fig. 15). Finally, in Fig. 16, bulk tungsten specimens fractured at room temperature and at 524° C (975° F) are shown. The fracture surfaces indicated a brittle behavior.

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DISCUSSION

This investigation was conducted to obtain an indication of the potential of laminar composites for high temperature use. The full potential of laminar composites relative to other types of materials can be partially established by comparing the strengths and specific strengths of the differing materials. In addition, two other types of comparisons were felt to be warranted; one involved comparing laminar composite strengths to values calculated utilizing rule-of-mixtures (ROM) equations and the other a comparison of strengths obtained based on a percentage of ROM values with those of fiber composites. The latter type of comparison tends to normalize the effects of reinforcement strengths, volume percentages of the stronger phases within the composites, and other material variables.

Comparison of Laminar Composite Strengths to Those of Fiber Composites and Superalloys

Tungsten/Nichrome V. - The best properties obtained in this investigation relative to competitive superalloys and to fiber superalloy composites, were obtained at 1093° C (2000° F). Figure 17 shows 1093° C (2000° F) tensile and 100-hour stress-rupture strengths of the strongest laminar composites compared with strengths of the strongest superalloys (10) and fiber/superalloy (1.2) composites revealed in the literature. At 1093° C (2000° F), the 100-hour stress-rupture strength of the tungsten/Nichrome V laminar composites, 121 MN/m² (17 500 psi) exceeds the values for one of the strongest superalloys, e.g., 89.5 MN/m^2 (13 000 psi) for NASA TRW VIA (10). These strengths may be compared with that of a typical high strength commercially used blade alloy B-1900, namely, 62 MN/m² (9000 psi). The 100-hour 1093° C (2000° F) specific stress-rupture value for the laminar composite is 1018 m (45 000 in.) compared with 762 m (30 000 in.) for B 1900 and 1005 m (39 000 in.) for NASA TRW VIA (fig. 18). The laminar composite strengths compared favorably to strengths of some fiber/superalloy composites (11,12) but were not as great as that of the tungsten -2 v/o ThO₂ fiber/ superalloy composites (2), Fig. 17. Laminar composite strengths were great enough however so that they would have about a 26° C (79° F) use temperature advantage (on an absolute strength basis) relative to conventional superalloys. On a specific-strength basis, the laminar composites were not as strong as the strongest of superalloys, Fig. 18.

In general, the 871° C (1600° F) tensile/and stress-rupture strengths obtained (table VI) compare well with those of fiber/superalloy composites made by others (12) but not with those of several of the stronger (e.g., 10) superalloys. Actually, in the 871° C (1600° F) temperature region, neither the laminar composites of this study nor the refractory metal fiber composites described in the literature have exhibited tensile and stress-rupture properties that exceed the strengths of the best superalloys. Also, specific strengths of both types of composites produced to date are lower than those of superalloys. With the development of stronger or lighter-weight sheets and foils, it may be possible to exceed superalloy specific strengths at this lower temperature.

<u>W-Re-Hf-C/Inconel Alloy 600.</u> - The strength of this composite was high relative to that of the tungsten/Nichrome V composites. However, it should be noted that the volume fraction of the strong phase was considerably greater than for the tungston/Nichrome V composites. The specimens were actually sandwich specimens (only one strong layer between two weaker layers). The strengths obtained do however indicate that the stronger laminar composites could be made if W-Re-Hf-C were used rather than tungsten in the composite. The o.l-hour stressrupture point (actually a tensile test result) was approximately 683 MN/m² (99 000 psi) at 1093° C (2000° F); a specific tensile value of 4191 m (165 000 in.). The latter value compares very favorably with the value of 3300 m (130 000 in.) for 37 v/o tungsten/Hastelloy X and 36 v/o TZM/Hastelloy X fiber composites (12) and with the superalloy NASA TRW VIA which has a specific tensile strength at 1093° C (2000° F) of 4013 m (158 000 in.)(10).

If the W-Re-Hf-C alloy could be produced in strong lamina form, construction of composites far stronger than those of the present investigation would be possible. In bulk form, W-Re-Hf-C alloy had a 2000° F (1093° C) tensile strength as great as 828 MN/m² (120 000 psi) (13). This alloy has been drawn into wire with unusually high 1093° C (2000° F) tensile strengths 2163 MN/m² (314 000 psi) and 100-hour stress-rupture strengths 1380 MN/m² (200 000 psi) at 1093° C (2000° F)(14).

Tungsten/Nichrome V Results in Relation to Rule-of-Mixtures Values

The ROM equation is known to satisfactorily represent fiber composite behavior for conditions of constant creep rate or constant time to rupture (15). While the equation has been most useful in predicting fiber composite strengths from strengths of the fibers and matrices, little or no work has been presented in the literature, to the authors knowledge, to show whether sheet or foil laminar composite strengths may be similarly related to laminae strengths. This study and the ensuing discussion will show that the tungsten/Nichrome V laminar composites with nominally 50 v/o tungsten have exhibited ROM or better than ROM stress-rupture strengths at 871° C (1600° F) and 1093° C (2000° F). No comparisons of composite tensile strengths with ROM calculated values will be presented since it is felt that too few tensile tests were made to permit an adequate analysis.

Stress-rupture curves based on ROM calculations of composite strengths were plotted in Figs. 7 and 8 along with the plots of the experimental data. Calculated composite strengths were obtained from the stress-rupture curves obtained for the tungsten and Nichrome sheet or foil materials (figs. 5 and 6). The Nichrome V strengths were obtained from the straight line plots shown in these figures that are contained within the data scatter bands from the literature. It was assumed that the vacuum environment in which the foils and sheet were tested was innocous. All experimental curves shown in Fig. 7 for the 871° C $(1600^{\circ}$ F) tests are above the calculated ROM curves, although in the case of the 0.050 cm (0.020 in.) laminae; the difference between the data and ROM curves was not very great. This result is evidence of synergism. Figure 9 shows that the strengths of these composites increased as the laminae thickness decreased. No metallographic evidence of damage to the tungsten, such as recrystallization, was observed in the specimens tested at 871° C (1600° F).

At 1093° C $(2000^{\circ}$ F) the results were somewhat different. For times less than 10 hours, the laminar composite strengths were greater than ROM values for the 0.0025 and 0.0125 cm (0.001 and 0.005 in.) laminae specimens. For times greater than 10 hours, strengths were less than ROM values. The thicker laminae composites, those with 0.050 cm (0.020 in.) laminae, had strengths equivalent to ROM values and greater than those of thinner laminae specimens (figs. 8 and 9).

Breaks in the stress-rupture curves of the thinner laminae specimens above 10 hours were believed to be the result of recrystallization of the tungsten sheets or foils. The recrystallization was promoted by the diffusion penetration of the Nichrome V elements into the tungsten. The thicker tungsten laminae in the 0.050 cm (0.020 in.) laminae composites were not damaged by diffusion or recrystallization to the same degree as the tungsten in the thinner laminae composites.

From the preceding discussion, it should be realized that the laminar composites tested had strengths at least equivalent, and often better, than ROM calculated strengths except for those cases where undue metallurgical damage was incurred. These overall results may be interpreted more broadly and suggest that laminar composites (those with close to 50 v/o of the stronger phase) would have at least ROM strengths if deleterious reactions could be avoided.

It could be argued that in-situ strengths of the constituents of the laminar composites, particularly those of the stronger tungsten phase, should be used to calculate the composite ROM strengths. It is beyond the scope of this paper to attempt to obtain such strengths, and in fact, it is difficult to prove that one can ever duplicate, in a constituent, the exact conditions found in a composite. In a system such as tungsten/Nichrome V, where the constituents have a reactivity potential, the problem can be far worse than if the materials were mutually insoluable, as was true in Refs. 15 and 16 for fiber composites. As part of this program two limited attempts were made to vary the condition of the tungsten lamina to simulate their condition within the composite. The first involved heat treating the tungsten utilizing the thermal cycle (982° C or 1800° F for 4 hours) used to consolidate the composites. Some heat treated lamina appeared to have lower stressrupture strengths than those of the tested as-received tungsten sheet or foils. In the second experiment, tungsten foil, 0.0125 cm (0.005 in.) was arc sprayed with a thin coat of Nichrome V (0.0038 cm or 0.0015 in.), subsequently heat treated to simulate the hot pressing conditions, and tested in stress-rupture at 1093° C (2000° F). The properties obtained (fig. 10) were almost identical to those of the laminar composites of

Fig. 8. In fact, there is even a break in the curve at slightly less than 10 hours similar to that for the composite. The spraying treatment combined with the subsequent anneal at 982° C (1800° F) caused the tungsten to recrystallize severely, as shown in Fig. 19. Further, the coated tungsten lamina had strengths about 1/4 to 1/2 that of the asreceived tungsten; compare Fig. 6 and 10. Thus, tests at 1093° C (2000° F) revealed that the arc-sprayed Nichrome severely damaged the tungsten. While it might be expected that the tungsten laminae within a composite would be visibly altered by diffusion bonding tungsten and Nichrome V laminae, the bonding process used did not cause noticeable damage to the tungsten, i.e., no metallographic evidence was found of recrystallization resulting from the diffusion bonding treatment. However, the Nichrome V in the laminar composites did cause a recrystallization of the tungsten in the stress-rupture specimens tested at 1093° C (2000° F) for long time periods. The extensive recrystallization in the arc sprayed tungsten specimens was believed to be triggered by the spray process itself rather than the diffusion process.

The preceding experiments indicated that there was a tendency for the diffusion bonding thermal treatment alone and for the diffusion of Nichrome V into the tungsten to damage the tungsten. The ROM values calculated from as-received tungsten tested as a separate material would then, if anything, be high. Thus comparisons of actual data for the laminar composites should give a conservative indication of the value of laminar composite strengths in relation to the strengths of the constituents. In addition, the synergism noted earlier should tend to be somewhat greater than indicated by the plots of Figs. 7 and 8. Further, the authors believe that the synergism results from mechanical restraint of the matrix, rather than metallurgical enhancement of the strength of the tungsten or strengthening of the matrix by alloying with the tungsten.

<u>Comparison of Normalized 1093^o C (2000^o F) Stress Rupture</u> <u>Strengths of Tungsten/Nichrome V Laminar Composites</u> <u>and Fiber Reinforced Superalloys</u>

Plots of normalized (present ROM values) 1- and 100-hour stressrupture strengths of the different composites of this investigation and of the fiber composites of Refs. 1, 2, 11 and 12 are given in Fig. 20. For 1-hour life, two types of laminar composites; those with 0.0025 cm and 0.0125 cm (0.001 and 0.005 in.) laminae, exhibited strengths of 130 and 117 percent of ROM values, evidence of appreciable synergism. The decrease in percent of ROM values with increased laminae thickness as indicated in the figure may be attributed to the lower strength of the thicker tungsten and to a lower restraint effect on the matrix by the tungsten.

The fiber composites also exhibited synergism, composite strengths for 1-hour ranging from 130 to 80 percent of the ROM values. Such synergism was not described in the references cited. Values plotted in Fig. 20 were calculated by the authors of this paper from the data contained in the references. At a life of 100 hours, both laminar and fiber composites had somewhat lower percentages of ROM strengths than for 1-hour; neither exhibiting synergism. The laminar composites exhibited approximately 40 to 100 percent ROM with the percent ROM increasing with laminae thickness. This increase may be attributed to the greater resistance of the thicker laminae to metallurgical degradation from diffusion with time. The fiber composites had percents of ROM values ranging from about 60 to 100.

In summary, the normalized values for the laminar composites compare favorably with those of some of the strongest fiber reinforced composites, although, as has been noted, some of the fiber composites represented by the bar graphs have greater absolute strengths than the laminar composites.

The preceding analysis indicates that the strengthening constituents (the strong laminae) in laminar composites can be utilized as effectively as can fibers in fiber reinforced composites; at least for about 50 v/o of the strong constituent. There is no obvious reason why the laminar reinforcements cannot also be utilized with similar effectiveness for composites with greater or lesser percentages of the strong phase. It may also be concluded that laminar composite strength can be increased by the use of stronger reinforcing laminae, analogous to the manner in which fiber reinforced composites are strengthened by the use of stronger fibers.

Impact Resistance of Composites

The tungsten in the tungsten/Nichrome V composites (table VIII) was more brittle at room temperature than at high temperature. This behavior was expected since most tungsten sheet undergo a ductile to brittle transition at temperatures between 150 and 370° C (302° to 698° F). The impact strength of the composites at 524° C (975° F) was 9.5 J (7 ft-lbs) compared with an average value of 1.7 J (1.3 ft-lbs) obtained at room temperature. Bulk tungsten had a 1.4 J (1 ft-lb) impact strength at both room and higher temperature. Thus the composite had superior impact resistance to that of bulk tungsten at high temperature but not at the low temperature. Probably, if the laminar composites were tested perpendicular to the planes of the foils in the composites (crack arrestor types of specimens) rather than perpendicular to the edges of the foils in the specimens (crack divider specimens), the impact strengths at room temperature would have been somewhat greater.

The impact strengths of the resin bonded tungsten specimens (2.7 to 6.1 J (2 to 4.5 ft-lbs)), were greater than that of the tungsten/ Nichrome V composites and bulk tungsten both of which had 1.4 J (1 ft-lb) impact strength. The resin bonded Nichrome V specimens bent and thus impact measurements were of little value, the material may be considered ductile, however.

The impact tests of the composites did reveal that the bonds between the tungsten and the Nichrome V were very good. Figure 13(a)shows that specimens impacted at room temperature had only slight evidence of delamination cracks while the specimens impacted at 524° C (975° F) flared out without delamination in the zone in which the hammer struck the specimen. This suggests that the interfaces between laminae were not embrittled by the diffusion bonding process used. Impact tests were not run perpendicular to the stacking of the laminae for reasons that have previously been described.

POTENTIAL OF LAMINAR COMPOSITES

To be considered for use as engineering materials, laminar composites would have to offer advantages relative to conventional materials. Ultimately, they should compete with fiber composites for some applications. The previously presented comparisons of laminar composite strengths to those of fiber composites (based on percent of ROM values) suggested that laminar composites have potential similar to that of fiber composites.

At the present time, metallic foils and sheet do not have strengths equivalent to those of the better fibers. Actually, very fine lamina are very difficult to produce and to test. As a consequence, intrinsic properties of fine foils may not be appreciated. It is felt that with further development efforts, thin films and foils may be made with far greater strengths.

Even with presently available sheet or foils, laminar composites should offer potential advantages relative to some fiber composites and to conventional alloys for those applications where biaxial stress states would exist. A type of laminar composite of great interest would be one having equally high strength in all planar directions. Such composites could be produced from cross-rolled sheet or foil. To produce near-in plane isotropic fiber composites, it is necessary to cross ply unidirectional fiber composite lamina. Such "pseudoisotropic" (in plane) composites have less strength than unidirectional composites of the same materials containing the same v/o fibers and tested in the direction of the fibers. Where matrices of such composites are considerably lower in strength than the fibers, the in-plane properties may be between 10 to 40 percent of the maximum unidirectional properties (17). Laminar composites comprised of planar isotropic sheets or foils would not have the orientation problem of fiber composites. In fact, laminar composites with a weaker reinforcing phase than that of fibers in a cross-plied fiber composite might very well have greater planar "isotropic" strength than would the fiber composites.

One of the principal envisoned uses for near in-plane isotropic laminar composites is for disks for various stages of gas turbines. In fact, for any circularly-shaped component where principal loads are planar (i.e., biaxial), laminar composites may be particularly useful. Other specific applications might include high temperature turbine blades, vanes, combustion chambers, high pressure, high temperature preburner chambers and chambers for advanced rocket engines, reentry vehicle edges, thrust reversers and nuclear or other furnace components such as hearths. Possible low-temperature applications include structural stiffeners, tubular beams (the laminae would be concentric tubes), gusset plates, channels, I-beams, panels without stiffeners, cryogenic tanks, high pressure vessels, pipes and bulkheads.

In many cases, laminar composites may be utilized in an "asconsolidated" condition. Subsequent to consolidation, many combinations of metal-metal laminar composites should be rollable, forgeable, bendable, and, in fact, may possibly be deep drawn or shaped by numerous existing metal working practices. Riveted connections should be simple to make, although other joining techniques (e.g., diffusion bonding and spot welding) could also be used.

SUMMARY OF RESULTS

This investigation was conducted to obtain indications of the potentialities of laminar metalOmetal composite materials for high temperature use. The investigation, concerned primarily with 50 v/o tungsten/50 v/o Nichrome V and secondarily with a 77 v/o tungsten alloy/ Inconel 600 composites, yielded the following results:

1. The highest tensile strengths were obtained for the tungsten/ Nichrome V specimens with 0.0025 cm (0.001 in.) thick laminae. Absolute and specific strengths were 600 MN/m^2 (87 000 psi) or 4350 m (171 000 in.) at 871° C (1600° F) and 448 MN/m^2 (65 000 psi) or 3380 m (130 000 in.) at 1093° C (2000° F).

2. The tungsten/Nichrome V composites with 0.025 cm (0.001 in.) laminae exhibited maximum 100-hour absolute and specific stress-rupture strengths of 387 MN/m^2 (56 000 psi) and 2900 m (112 000 in.) at 871° C (1600° F). Composites with 0.050 cm (0.020-in.) laminae had the maximum 100-hour stress-rupture strength and specific strength of 121 MN/m^2 (17 500 psi) and 915 m (36 000 in.) at 1093° C (2000° F).

3. Tungsten/Nichrome V composites manifested increasing tensile and stress-rupture strength with decreasing thickness of laminae at 871° C (1600° F). At 1093° C (2000° F), the same trend was evident for tensile strengths and short time rupture strengths (less than 10 hours). For longer times at 1093° C (2000° F) the trend was reversed, the thicker the composite laminae, the greater the composite strength.

4. The tensile and stress-rupture results for tungsten/Nichrome V laminar specimens generally equaled, or exceeded, rule-of-mixtures estimated strengths. Thus, synergism occurred for a broad range of conditions. Less than rule-of-mixtures strengths were obtained for laminar composites tested at 1093° C (2000° F) for times beyond about 10 hours; the poorer strengths were attributable to recrystallization of the tungsten. The best stress-rupture strength obtained for laminar composites at 1093° C (2000° F) for a time of one hour was 130 percent of rule-of-mixtures for a 0.0025 cm (0.001 in.) laminae composite; and for 100 hours it was 100 percent for 0.050 cm (0.020 in.) laminae composites. These values compared very favorably, and under some conditions exceeded corresponding values for some of the best tungsten fiber

composites reported in the literature.

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5. The W-Re-Hf-C/Inconel 600 composite had a tensile strength at 1093° C (2000° F) of about 680 MN/m² (99 000 psi) and an 0.8-hour stress-rupture strength of 552 MN/m² (80 000 psi).

6. Impact strengths obtained for modified Charpy tungsten/Nichrome V laminar specimens made with 0.0125 cm (0.005 in.) laminae were 2 J (1.5 ft-lbs) at 23° C (75° F) and 9.5 J (7 ft-lbs) at 524° C (975° F), this difference reflecting increased ductility of tungsten laminae above the probable DBTT. Macroscopic examination of impacted specimens revealed that a good bond between tungsten and Nichrome V existed.

7. Metallographic studies of tungsten/Nichrome V laminar composites showed no metallurgical degradation of the tungsten at 871° C (1600° F). At 1093° C (2000° F) the tungsten in the 0.00250 and 0.0125 cm (0.001 and 0.005 in.) laminae composites parly recrystallized at times less than 10 hours and fully recrystallized at times greater than 10 hours. The 0.050 cm (0.020 in.) tungsten laminae did not fully recrystallize for a time period up to 148 hours. The tungsten laminae tended to fracture in a ductile manner while the Nichrome V laminae tended to fail intergranularly.

CONCLUDING REMARKS

Evidence has been obtained in this investigation that indicates that laminar metal-metal composites have a similar potential for strengthening to that of metal fiber/metal matrix composites. Composites had rule-of-mixture, or synergistic properties. Synergism was believed due to mechanical restraint of the weaker by the stronger of the laminae. The authors believe that the results of this study may be interpreted more broadly to indicate that laminar composites of various material combinations may have potential for lower temperature use as well as high temperature applications. Far stronger composites than those of this investigation could be produced if sheet and foil materials were developed for specific use in composites. The situation is analogous to that which existed for fiber composites several years ago when those working in the field used available materials which although strong, weren't designed specifically for composite applications.

Because laminar composites have several intrinsic characteristics, they may be desirable for many applications. For example, they probably can be made to have near-in plane isotropy without sacrificing maximum strength or modulus. This should enable laminar composites to withstand high transient stresses arising from impact, fatigue, etc., while providing a high safety factor. Many combinations of laminated materials can be envisioned that would be readily fabricable into parts or components of structures.

REFERENCES

- 1. D. W. Petrasek, R. A. Signorelli, and J. W. Weeton: NASA TN D-4787, 1968.
- 2. D. W. Petrasek, and R. A. Signorelli: NASA TN D-5575, 1970.
- 3. A. Lawley, H. L. Gaiher, and S. Shuster: Franklin Institute, Rep. F-A2366, 1964.
- 4. R. A. Covert and E. Ravinowicz: Allkoyd Electronics Corp., ASD-TR 62-67, June 1962.
- 5. J. Melill, L. Ecker, H. J. Greenspan, and R. Lorenz: North American Aviation, Inc., IR-9122 (III), AD-805408, Dec. 1966.
- 6. J. D. Embury, N. J. Petch, A. E. Wraith, and E. S. Wright: <u>Trans.</u> <u>AIME</u>, 1967, vol. 239, p. 114.
- 7. R. L. Jones and P. W. Cooke: Battelle Memorial Inst., DMIC Memo 243, 1969, p. 28.
- 8. C. A. Hoffman: NASA TN D-5926, 1970.
- 9. J. V Gluck and J. W. Freeman: Michigan Univ., ASD-TR-61-339, Sept. 1961.
- 10. H. E. Collins: TRW, Inc., Rep. TRW-ER-7162, NASA CR-54507, June 1967.
- 11. A. V. Dean: J. Inst. Metals, 1967, vol. 95, p. 79.
- 12. Baskey, R. H.: Fiber-Reinforced Metallic Composite Materials. Clevite Corp., AFML TR-67-196, AD-825364, Sept. 1967.
- 13. W. D. Klopp, P. L. Raffo, and W. R. Witzke: <u>J. Metals</u>, 1971, vol. 23 (6), p. 27, June 1971.
- 14. D. W. Petrasek: High-Temperature Properties of High Strength Refractory Alloy Wires, and Consideration for Application to Metallic Composites. Proposed.
- 15. D. L. McDanels, R. A. Signorelli, and J. W. Weeton: NASA TN D-4173, 1967.
- L. McDanels, R. W. Jech, and J. W. Weeton: NASA TN D-1881, 1963.
 C. C. Chamis: NASA TN D-6696, 1972.

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Figure 1. - Tensile and stress-rupture specimens.



Figure 2. - Method of assembling laminae stacks for hot pressing.

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Figure 4. - Delamination resulting from grinding thin crack arrestor Charpy specimen from large block of hot pressed tungsten/ Nichrome V.





- 0.0025 CM (0.001 IN.) TUNGSTEN-AS RECEIVED
- D 0.0125 CM (0.005 IN.) TUNGSTEN-AS RECEIVED
- △ 0.05 CM (0.020 IN.) TUNGSTEN-AS RECEIVED
- ▲ 0.05 CM (0.020 IN.) TUNGSTEN-ANNEALED 4 HR AT 982° C (1800° F)-VAC.
- △ 0.05 CM (0.020 IN.) TUNGSTEN TESTED TRANSVERSE TO ROLLING DIRECTION



Figure 6. - Tensile and stress rupture strength of tungsten and Nichrome V sheet and foil lamina specimens at 1093⁰ C (2000⁰F) in vacuum. Tensile results are plotted as 0.1 hr points.

















Figure 11. - Microstructures of tungsten/Nichrome V laminar composite specimens after testing in stress-rupture at 871° C (1600° F). M = matrix; R = reinforcement.

CS-63356

(c) 0.050 CM (0.020 IN.) SPECIMEN AFTER 1921 HOURS; X100. UNETCHED.



(b) 0.0125 CM (0.005 IN.) AFTER 328.7 HOURS; X100. UNETCHED.



Figure 12. - Microstructures of tungsten/Nichrome V laminar composite specimens after testing at 1093° C (2000° F). Etched. M = matrix; R = reinforcement.

CS-63355

(c) 0.050 CM (0.020 IN.) SPECIMEN AFTER 21.5 HOURS; X50.





(a) 0.0025 CM (0.001 IN.) SPECIMEN, TENSILE TESTED; X100.

(b) 0.0125 CM (0.005 IN.) SPECIMEN AFTER 3.7 HOURS; X50.

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Figure 15. - Tungsten 0.0125 cm (0.005 in.) laminae impact specimen, resin bonded at ends, after impact testing at room temperature.



Figure 16. - Bulk tungsten impact specimens after testing. Views are perpendicular to fracture surfaces.

TENSILE STRENGTH 100 HR STRESS-RUPTURE STRENGTH 77 v/o W-Re-Hf-C/INCONEL 600 . 0875 CM (. 035 IN.) TUNGSTEN ALLOY 2 70 v/o W-2ThO2/SUPERALLOY .035 CM (. 015 IN.) FIBERS (REF. 37 v/o TUNGSTEN'HASTELLOY X (REF. 12) 50 v/o TUNGSTEN/NICHROME V . 050 CM (. 020 IN.) LAMINAE NASA - TRW VI A (REF. 10) 150x10³ 1000 800 STRENGTH, MN/m² STRENGTH, PSI 100 600 400 50 200 0 LAMINAR COMPOSITES FIBER COMPOSITES SUPERALLOYS CS-63364 (OF THIS STUDY) (LITERATURE) (LITERATURE)





Figure 18. - Specific tensile and stress-rupture strengths of laminar composites, fiber composites, and superalloys at 1093^o C (2000^o F). (a) Extrapolated from stress-rupture data.



Figure 19. - Tungsten foil arc sprayed with Nichrome V powder and given thermal cycle used to consolidate laminates. Stress-rupture tested for 4.7 hours at 1093° C (2000° F). Etched; X250.



Figure 20. - Comparison of 1093° C (2000° F) laminar composite stress-rupture strengths obtained in this study with strengths of fiber composites, the best reported in the literature, on the basis of percent of rule-of-mixture values. Abbreviations or chemical symbols are used in this figure; W = tungsten; SA = superalloy, NiCr V = Nichrome V, ThO₂ = thoria.

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