ASD TR 7-924 (II)

ASD INTERIM REPORT 7-924 (II)
May 1962

FIBER REINFORCEMENT OF METALLIC AND NONMETALLIC COMPOSITES

198100

R H Baskey

CLEVITE CORPORATION
Mechanical Research Division

Contract AF 33(657)-7139
ASD Project 7-924
17 January 1962 to 17 April 1962

Initial parameter studies have indicated that powder metal bars of stainless steel type 316 were reinforced by 14 volume percent 10 mil diameter continuous tungsten wires. The room temperature tensile strength of the stainless steel was increased from 40,000 psi to 48,000 psi by adding tungsten wires. Nichrome V (~~80 Cr - 20 Ni~~) matrices were not reinforced by either tungsten or molybdenum wires. (80 Ni - 20 Cr)

MANUFACTURING METHODS BRANCH
METHODS AND MATERIALS DIVISION
Aeronautical Systems Division
Air Force Systems Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

ABSTRACT - SUMMARY

Interim Progress Report

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Parameter studies are in progress to determine the effect of fiber diameter, fiber length, fiber spacing, fiber direction and fiber-to-matrix bond strength.

The fibers and powdered metal have been consolidated by hot pressing, hydrostatic pressing or cold pressing followed by sintering.

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FOREWORD

This interim technical report covers the period performed under contract AF 33(657)-7139 from 17 January 1962 to April 17, 1962. It is published for technical information only and does not necessarily represent the recommendations, conclusions or approval of the Air Force.

This contract with Clevite Corporation, Mechanical Research Division, Cleveland, Ohio was initiated with ASD Manufacturing Methods, Project 7-924. It is administered under the direction of Mr. Lova Polley, (ASRCTB) Aeronautical Systems Division, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio.

Mr. Raymond Baskey of Clevite's Mechanical Research Division is the project engineer. Others who cooperated in the research and in the preparation of the report were: Mr. Arthur D. Schwope, Director; and Mr. Gail F. Davies, Supervisor.

The primary objective of the Air Force Manufacturing Methods Program is to increase producibility, and improve the quality and efficiency of fabrication of aircraft, missiles, and components thereof. This report is being disseminated in order that methods and/or equipment developed may be used throughout industry, thereby reducing costs and giving "MORE AIR FORCE PER DOLLAR".

Your comments are solicited on the potential utilization of the information contained herein as applied to your present or future production programs. Suggestions concerning additional Manufacturing Methods development required on this or other subjects will be appreciated.

****

PUBLICATION REVIEW

Approved by: Gail F. Davies
Gail F. Davies, Head
Aerospace Research Section

Approved by: A. D. Schwope
A. D. Schwope, Director
Mechanical Research Division

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Interim Report No. 2
Contract AF 33(657)-7139

INTRODUCTION

The objective of this program is to establish parameters for the selection and application of fibers to the reinforcement of metal matrices and demonstrate that this can be achieved through the fabrication of sheet and forged products.

One of the earliest applications of reinforcing metals was that of Whitehurst[1]. Here, aluminum was reinforced at elevated temperatures by fiberglas. Other investigators have reported on the use of ceramic or metal filaments to reinforce other materials.

Tinklepaugh[2, 3] and associates have reported on a program to reinforce mullite-alumina ceramic and alumina with molybdenum wires. Baskin (4) reported on thoria reinforced by molybdenum or niobium fibers. Here the thermal shock properties were improved.

Parikh and Fisher[5] have reported on the felting process developed by Armour and programs to study the fundamentals needed to reinforce materials.

Jech and Weber[6] found that molybdenum fibers improved the elevated temperatures tensile properties of titanium. However, the stress-rupture properties were unsatisfactory because the molybdenum fibers oxidized during the test period with a resultant loss of strength as compared to the tensile properties at a specific temperature.

McDanels, Jech, and Weeton[7] have reported on a study to reinforce copper with tungsten wires. They have achieved excellent reinforcement at room temperature. Their main purpose is to study the fundamentals involved in reinforcing materials.

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(1) References are at end of text.

Several investigators [8, 9] have developed theories for the reinforcement of metal matrices with wires or whiskers. Many of these theories are based on the work of Outwater[10]. However, there are insufficient data available on metal fiber-metal matrices systems to confirm the validity of the theories or equations. Mac hlin[11] has stated that the method of consolidating fibers and matrices must be improved to provide a satisfactory structural composite material. The best results and data on reinforcing materials is that obtained in the fiberglas-plastic systems. One example is the work of Bell[12] where the effects of fiber orientation and length are related to specific physical properties.

Reinforcing ceramic or metallic materials with high-strength, high-temperature wires, filaments, or whiskers appears to be one of the best methods for improving the strength-to-density ratio of materials.

The basic assumption for most composites containing fibers (or whiskers) is that they, rather than the metallic phase, or matrix, carry the major portion of the tensile load. One of the common examples of strengthening is the use of steel mesh, wire, or rods in reinforced concrete. Here the steel provides tensile strength which the concrete itselt does not possess; the concrete functions only in compression.

This program is divided into three phases. They are:

- Phase I - State-of-the-art survey on fiber metallurgy and compilation of bibliography
- Phase II - Parameter study and process development
- Phase III - Pilot Production

Phase I was completed and reported as ASD Interim Report 7-924(I). The report[13] was divided into two parts. Part one of this survey reviewed the current state-of-the-art on strengthening theories, methods of producing fibers or whiskers, methods of reinforcing composites with fibers or whiskers, and whisker formation. Part two was a bibliography of fiber metallurgy and consisted of abstracts of each of the 293 articles or patents covered by the survey.

Phase II is now in progress. It consists of (1) selection of the best compatibility between the two components (i.e., fiber and matrix), (2) evaluation of the parameters needed to obtain the optimum reinforcing, i.e., fiber diameter, fiber length, fiber orientation, inter-fiber spacing, fiber volume, etc., and (3) experiments to determine the best methods of consolidating the fibers and matrix. Physical property data will be obtained concurrent with the processing experiments.

DISCUSSION

PROCEDURE

Three methods have been used to consolidate wires with metal powders. These are hot pressing, hydrostatic pressing, and cold pressing followed by sintering. Each of these methods is described briefly below:

1. Hot Pressing

   Five-mil diameter tungsten wire was cut to 1/8 inch lengths and blended with -140 mesh Rene' 41 powder or -140 mesh Nichrome V powder. This mixture was then hot pressed in a graphite die at temperatures of 1950 F, 2050 F, and 2150 F at a pressure of 4000 psi in a vacuum or hydrogen-argon atmosphere. The finished compact size was a 2 inch diameter by 3/8 inch thickness. Hot pressing under these conditions produced specimens with densities of 95 percent theoretical density. A metallographic examination of the microstructure indicated the fiber spacing and the interface between fiber and matrix.

2. Hydrostatic Pressing

   The hydrostatic pressing method was investigated for consolidating wire and metal powder because a wide variety of shapes can be formed by this method. The green compacts must be sintered in the same manner as cold pressed powder.

   Compacts were produced by using either discontinuous or continuous wires with either Nichrome V or Rene' 41 powder. They were pressed at

60, 000 psi and sintered to a 91 percent theoretical density. A metallographic examination indicated that this method of consolidating wire and powder was satisfactory for the materials used.

3. Cold Pressing

The cold pressing method is being used at the present time to press tensile bars for parameter studies. The preliminary results indicate that the cold pressing technique will produce compacts comparable to the hot pressed or hydrostatic methods.

Test specimens were cold pressed in a steel die to the shape of a standard powder metal tensile bar. This bar is 3-1/2 inches long by 0.230 inch wide at the reduced section by a thickness of 0.250 inch. The powder materials were cold pressed to furnish a green specimen with a theoretical density of approximately 85 percent. Next, the green bars were sintered to attain a theoretical density of approximately 90 percent.

Three types of tensile bars were prepared for comparison. These are:

1. 100 percent metal powder.
2. Metal powder plus random distribution of fibers.
3. Metal powder plus continuous and discontinuous fibers oriented parallel to the applied stress.

RESULTS

Ceramic or metallic whiskers offer the maximum strength potential but they are not available commercially.

Ceramic fibers are promising for high temperature applications (i.e. silica fibers). They are resistant to oxidation and have a low density. However, these fibers are susceptible to abrasion. In addition, they exhibit poor bonding with the metal matrix. Data are incomplete on the strength of these fibers at elevated temperatures.

Metal fibers offer advantages in reinforcing materials for high temperature applications. However, metal fibers are susceptible to oxidation and loss of strength due to recrystallization.

Tungsten and molybdenum metal fibers were selected for the initial experimental work. They possess high strength at elevated temperatures. Several of the more promising ceramic fibers will be investigated later. Table 1 indicates the short time tensile strength of 0.003 inch diameter tungsten wire at elevated temperatures. Several of the tungsten alloy wires (i.e. tungsten-thoria or tungsten-rhenium) have higher recrystallization temperatures than pure tungsten wire. Figure 1 represents[14] the stress rupture life of commercially available superalloys. In general, these materials are not strong enough for use as the reinforcing agent.

Nichrome V (80 Ni - 20 Cr) was selected as the matrix material for the initial experiments. Other matrix materials being used for the initial investigation include Rene' 41, stainless steel, iron alloys, cobalt, and copper. Nichrome V has several advantages as a matrix material. These are:

a. It is a simple binary alloy system, being one which does not exhibit any precipitation hardening or strengthening mechanisms. Thus, any strength improvement offered by the incorporation of metal fibers may be said to be the result of the fiber addition alone.

b. This material has some excellent high-temperature properties. Although lacking in hot strength, it has adequate oxidation resistance up to 2200 F, and may be easily hot worked by conventional means.

Figure 2(a, b, c) illustrate some of the fiber patterns used on the parameter investigation. Figure 2a represents continuous fibers while figure 2b and 2c are discontinuous fibers placed in two different types of patterns.

The discontinuous fibers were 10 mil diameter tungsten or molybdenum which had been cut to 1/8 inch lengths. They were laid out in straight rows so that the ends were about 0.035 inch apart. The lateral and vertical spacing from

TABLE I

Tensile Data at Elevated Temperature - Unalloyed Tungsten Wire

Testing C	Temperature F	Tensile Strength psi
20	68	430,000
200	392	350,000
400	752	320,000
600	1112	240,000
800	1472	200,000
1000	1832	100,000
1500	2732	50,000
2000	3632	20,000
2500	4532	10,000
2800	5072	5,000

Source: Tajme, R., Japan, J. Phys. 4 (23A)(1926)

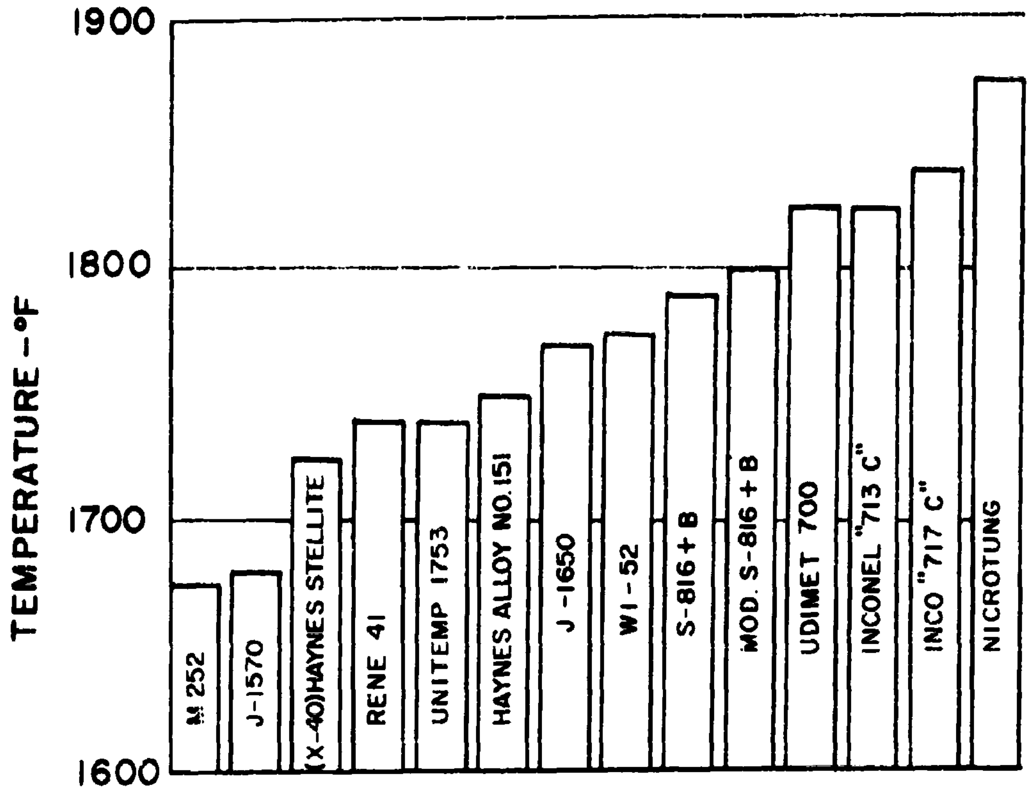


FIG. 1: OPERATING TEMPERATURE FOR 100-HOUR RUPTURE TIME AND 15,000 PSI STRESS LEVEL FOR VARIOUS SUPERALLOYS. (REF. 15)

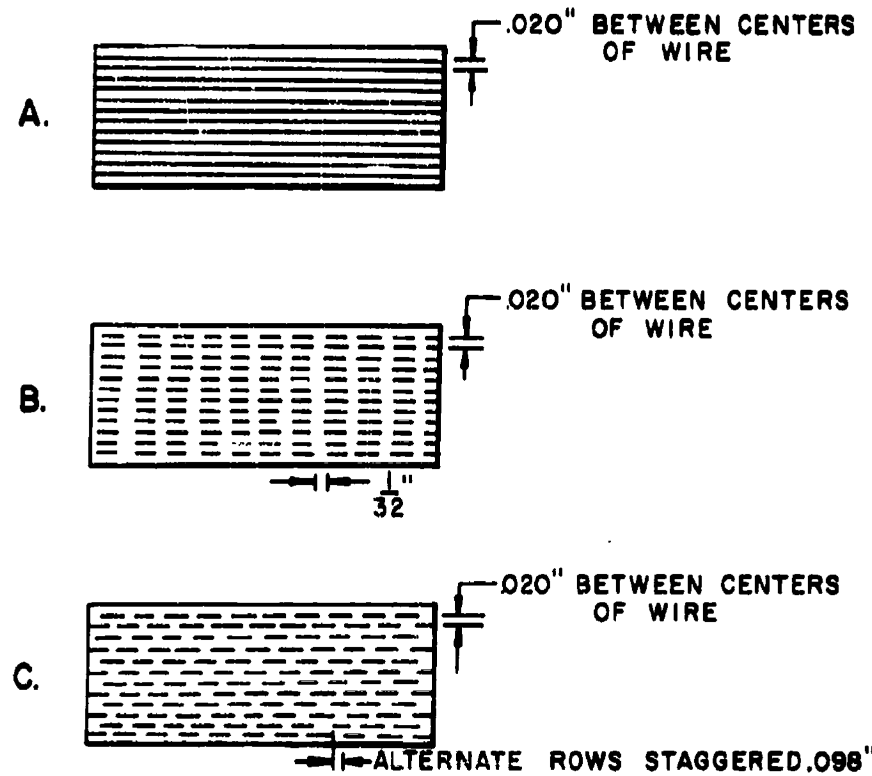


FIG. 2 : FIBER PATTERNS USED ON INITIAL PARAMETER STUDY. CONTINUOUS AND DISCONTINUOUS PATTERNS.

fiber diameter to fiber diameter was approximately 0.020 inch. Metal powder was added to separate each layer of fibers. The main emphasis while surveying a number of combinations is to produce tensile bars in which the continuous fibers are aligned in the direction of applied stress. This type of tensile bar will exhibit the maximum reinforcement when good bonding is achieved between the matrix and fibers. The tensile bars with continuous fibers are being compared to specimens which contain no fibers; specimens which contain discontinuous fibers oriented in a random pattern; and specimens with discontinuous fibers aligned parallel to the applied stress.

Several combinations of materials were cold pressed and sintered. They were:

(1) Tungsten fibers and nichrome powder.
(2) Tungsten fibers and stainless steel powder (316 type).
(3) Tungsten fibers and copper powder.
(4) Molybdenum fibers and nichrome powder.
(5) Molybdenum fibers and stainless steel powder.

Table II lists the physical property data obtained on tensile specimens prepared from the above materials. Figure 3 summarizes the average data obtained for specimens of 100 percent powder metal compared to the specimens with continuous or discontinuous fibers.

The maximum degree of strengthening was obtained by using 12.6 v/o continuous tungsten fibers in a copper matrix. An example of the cross section of this specimen after tensile testing is shown in Figure 4. Good results were obtained from this system because the tungsten wets the copper and these materials are mutually insoluble. This copper-tungsten system is not acceptable for high temperature applications. The reason for pressing the three copper-tungsten fiber tensile bar specimens was to demonstrate the feasibility of obtaining reinforcement by a cold pressing operation where a good bond exists between the copper matrix and tungsten fiber.

A theory of composite strengthening[15] states that the composite property (S) is equivalent to the sum of the matrix property (M) and the fiber property (F), or:

TABLE II

Room Temperature Physical Properties of Composites. Pure Metals and Metals Containing Discontinuous or Continuous Fibers

Spec. No.	Type Fiber	Fiber Dia.	Matrix Material	Vol. % Fibers	Fiber Orient.	Cold Pressed at psi	Sintering Conditions	Ultimate Tensile Strength psi	Yield Strength psi	Elong %	Elastic Modulus x10^6
49	None	-	NiCr-140	-	-	60,000	1 hr. 2250F 93%A, 7%H_2	36,900	-	7.5	
50	None	-	NiCr-140	-	-	60,000	1 hr. 2250F 93%A, 7%H_2	36,400		5.5	
54	None	-	NiCr-140	-	-	60,000	1 hr. 2300F Vac 93% A	30,500	19,900	-	13.6
47	NF-W	.010	NiCr-140	10.0	-	60,000	1 hr. 2250F 7%H_2	27,500	-	2.0	-
53	NF-W	.010	NiCr-140	9.7	-	60,000	1 hr. 2300F Vac.	23,700	-	-	-
57	None,	-	NiCr-140	-	-	120,000	1 hr. 2100F NH_3	40,000	32,100	5.0	19.
58	None	-	NiCr-140	-	-	120,000	1 hr. 2100F NH_3	40,000	31,800	5.0	21.5
59	None	-	NiCr-140	-	-	120,000	1 hr. 2300F Vac.	60,100	28,500	20.0	22.3
63	None	-	NiCr-140	-	-	120,000	1 hr. 2300F Vac	56,500	30,000	5.0	22.3
64	None	-	NiCr-140	-	-	120,000	1 hr. 2300F H_2 1 hr. 2300F Vac	54,400	29,400	-	21.0
65	None	-	NiCr-140	-	-	120,000	1 hr. 2300F H_2 1 hr. 2300 F Vac	50,400	29,000	-	24.9
66	None	-	NiCr-140	-	-	120,000	2 hr. 2300F Vac	60,400	27,000	22.0	23.6
67	None	-	NiCr-140	-	-	120,000	20 hr. 2300F Vac	73,600	28,700	35.0	23.1
68	None	-	NiCr-140	-	-	120,000	20 hr 2300F Vac	74,400	28,000	39.0	26.5
69	None	-	NiCr-140	-	-	120,000	20 hr. 2300F Vac	70,800	26,600	34.0	24.0
60	218-W	.010	NiCr-140	8.6	Random	120,000	20 hr. 2300F H_2 Vac		-	-	-
61	218-W	.010	NiCr-140	8.7	Random	120,000	1 hr. 2300F Vac	30,300	-	-	-
62	218-W	.005	NiCr-140	8.6	Random	120,000	2 hr. 2300F Vac	43,000	-	7.5	-
81	218-W	.010	NiCr-140	8.6	Random	120,000	1 hr. 2300F H_2 1 hr. 2300F Vac	36,700	25,100	5.0	20.5
83	218-W	.010	NiCr-140	8.6	Random	120,000	1 hr. 2300F Vac	32,500	24,500	5.0	19.5
85	NF-W	.010	NiCr-140	11.4	See Fig. 2b, 2c	120,000	2 hr. 2300F Vac	51,600	31,000	12.5	22.8
86	NF-W	.010	NiCr-140	15.8	Full length	120,000	2 hr. 2300F Vac	48,600	42,000	10.0	23.2
103	Mo	.010	NiCr-140	17.0	Full length	120,000	2 hr. 2300F Vac	51,100	36,200	7.0	22.7
104	Mo	.010	NiCr-140	16.2	Full length	120,000	1 hr. 2300F Vac	57,400	29,000	10.0	12.2
88	None	-	Cu 150	-	-	60,000	1 hr. 1850 F H_2	26,200	4,400	35	14.2
89	None	-	Cu 150		-	60,000	1 hr. 1850F H_2	26,500	4,620	325	10.0
90	NF-W	.101	Cu 150	12.6	Full length	60,000	1 hr. 1850F H_2	55,700	44,500		8.1
93	None	-	316-SS 100	-	-	100,000	1 hr. 2200F Vac	40,000	23,000	15.0	13.7
95	NF-W	.010	316-SS 100	14.4	Full length	100,000	1 hr. 2200F Vac	48,000	-	-	-
105	NF-W	.010	316-SS 100	17.2	Full length	100,000	1 hr. 2200F Vac	28,800	36,500	-	10.6

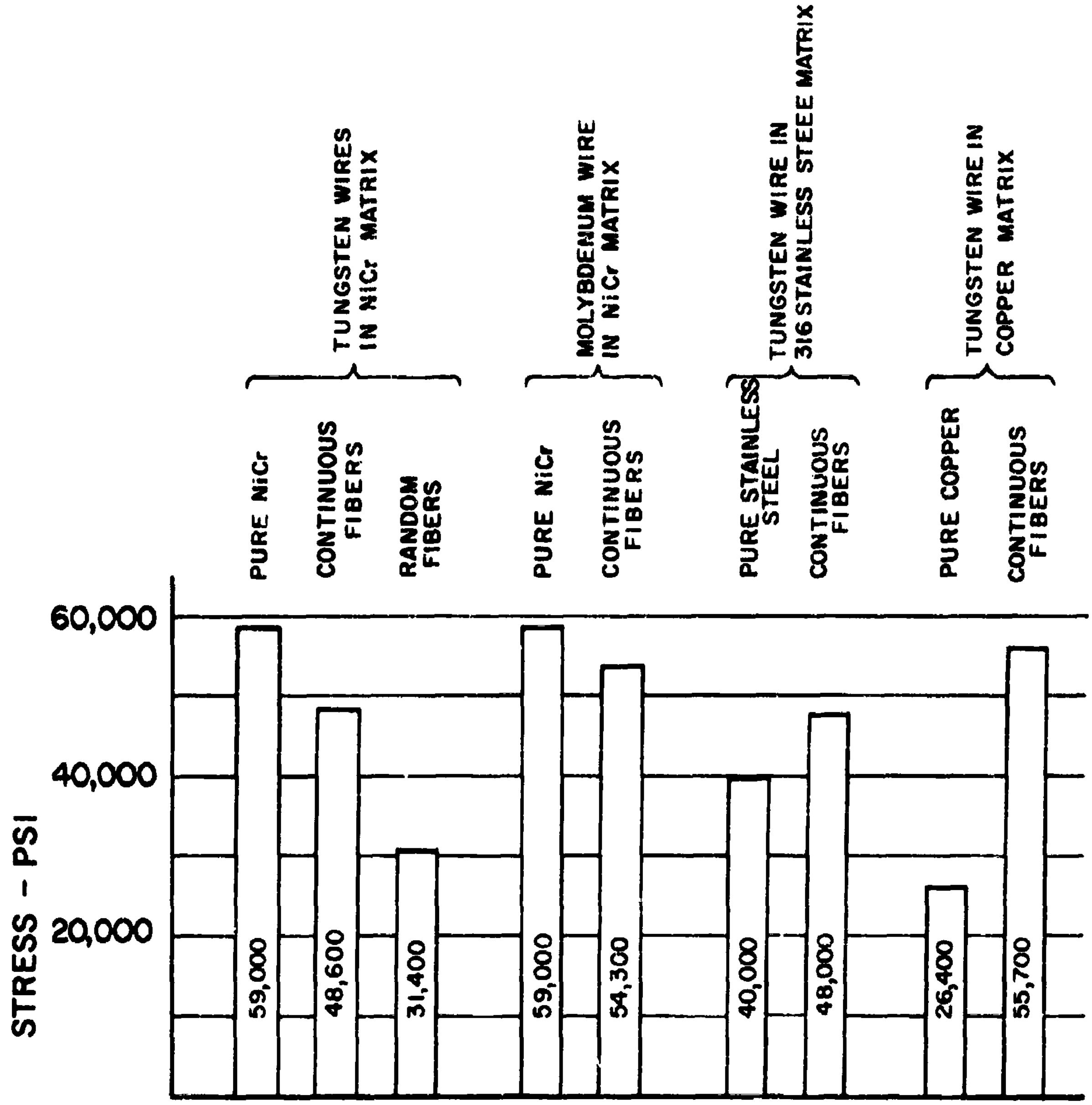
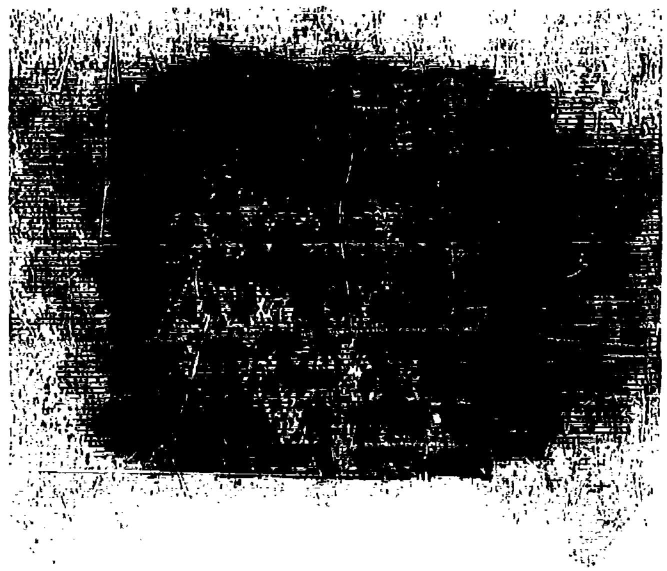


FIG. 3: COMPARISON OF PURE METAL AND COMPOSITES CONTAINING CONTINUOUS OR DISCONTINUOUS FIBER ORIENTATION.



9180 8X

FIGURE 4: FRACTURED CROSS SECTION OF COLD PRESSED AND SINTERED COPPER TENSILE BAR REINFORCED THROUGHOUT ITS FULL LENGTH WITH .010" DIAMETER NF TUNGSTEN WIRE.

$$S = Fx + M(1-x)$$

where x = fiber volume

This theory is based on three assumptions:

1. Hookes Law is valid; i.e. the composite is homogeneous and isotropic.
2. The fibers are uniformly bonded to the matrix such that the fibers and matrix strain together.
3. The fibers are unidirectional and have a uniform cross section.

An example of the above equation with the tungsten fiber-copper matrix is as follows:

volume percent of tungsten fibers (x) = .126
strength of copper matrix specimen no. 90 M = 26,350 psi
strength of 10 mil diameter tungsten fibers F = 273,000 psi
(annealed at 1875 F - 1 hour)

$$S = (273,000)(.126) + 26,350(.874)$$
$$= (57,500 \text{ psi ultimate tensile strength at room temperature}$$

The measured ultimate tensile strength of specimen 90 was 55,700 psi.

This sample calculation is an example of the strengthening obtained by fibers in an ideal combination of materials. It appears that each constituent in the continuous fiber specimen carried a tensile load proportional to the strength and volume percentage of the constituent.

The other specimens tested to date do not conform to this equation. For instance, neither the tungsten or molybdenum continuous fibers provided any strengthening to the Nichrome matrix. However, the 316 type stainless steel matrix was strengthened by tungsten fibers. The pure matrix had a strength of 40,000 psi. When 14.4 v/o 10 mil diameter tungsten fibers were added to the stainless steel matrix, its tensile strength increased to 48,000 psi. One possible reason why these materials did not obey the above equation was the formation of a brittle intermetallic between the matrix and the fiber.

One method is being investigated to determine if the strength of the nichrome or stainless steel can be improved. It consists of coating the fibers before they are mixed with the powder. Results are not yet available.

The tensile bars with a random fiber distribution were weaker than the bars composed of pure metal. The angle between the fiber and the applied stress is critical because it governs the amount of strengthening that can be obtained. Fiber patterns will be prepared in the tensile bar specimens to study the effect of the angle between the stress direction and fiber orientation on the composite strength.

Only a limited amount of data are available from the tensile bars which contain discontinuous fibers aligned parallel to the stress. (c.f. Figure 2b, 2c)

Additional types of fibers and matrices are also in process but the results are incomplete. Tests are also underway to determine the physical properties of the tungsten or molybdenum wires after annealing at the same temperatures and times involved in the sintering process.

CONCLUSIONS

1. Continuous tungsten fibers (12.6 v/o) reinforced a copper matrix so that its tensile strength at room temperature was more than twice that of a pure copper specimen. Both specimens were cold pressed and sintered in the same manner.

2. Continuous tungsten fibers (14.4 v/o) strengthened a type 316 stainless steel matrix at room temperature. The pure stainless steel had a tensile strength of 40,000 while the fiber reinforced stainless steel was 48,000 psi.

3. The tungsten and molybdenum continuous fibers did not strengthen a Nichrome matrix.

4. Random distribution of fibers in a matrix lowered the composite strength.

5. Powder metals and fibers can be consolidated by hot pressing, cold pressing, followed by sintering or hydrostatic pressing.

FUTURE WORK

1. Continue evaluation of parameters of metal fiber-reinforced metal systems.
2. Continue evaluation of powder metallurgy method of consolidating fiber with matrix.
3. Investigate rolling properties of composites containing fibers.
4. Investigate high temperature strength of selected composite systems.

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