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Progress Report Number 16 CONTRACT NUMBER NAS8-11508 Control Nr. CPB 02-1234064

Covering Period of October 1,1964 to November 1,1964

DEVELOPMENT OF ULTRAHIGH STRENGTH, LOW DENSITY, ALUMINUM PLATE COMPOSITES

November 5, 1964

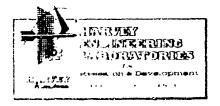
For George C. Marshall Space Flight Center National Aeronautics & Space Administration Huntsville, Alabama

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## ABSTRACT

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The objective of this project is to develop a high strength, low density, composite plate of aluminum alloy reinforced with steel wires for cryogenic and structural applications in space and launch vehicles.

The effort to produce composite aluminum plate and develop methods for joining same, has been divided into two areas: the even distribution of the steel wire in the aluminum matrix and the bonding of aluminum to aluminum and aluminum to steel.

Progress in the development of experimental methods for producing modular units of the composite plate has been made. Tensile strengths of 137,000 psi and 173,000 psi have been attained in 5456 and 2024-T62 alloy, respectively, containing approximately 25% NS355, 0.010" steel wire by volume. A sample of 2219 alloy with 25% 0.010" NS355 wire attained a tensile strength of 158,000 psi, and preliminary joining camples and a 3/4" thick sample were produced.

Design parameters for hot extruding 0.020" 2219 alloy have been established and tooling has been designed for generating hot rolling parameters.

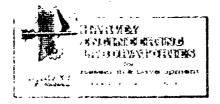


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## I. ACCOMPLISHMENTS PREVIOUSLY REPORTED

#### A. Literature and Industrial Survey

The contractor reviewed literature reported in current technical abstracts published by the U.S. Government and in publication of various technical societies. The results were published in HA-1955 dated August 28, 1963 and revised and brought up to date in HA-2059 dated August 15, 1964 and HA No. 2077 dated September 10, 1964.

## B. Wire Distribution

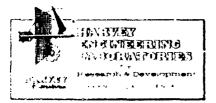
1. <u>Extrusion</u> - Basic tool concepts were developed for the extrusion of composite wire consisting of 0.030" 0.D. and 0.045" 0.D. aluminum extrusion containing 0.010" diameter steel wire core. After a number of modifications 0.030" and 0.045" extrusions containing 0.010" steel wire were produced at a temperature of 400°F and extrusion ratios of 88/1 and 36/1 respectively, using 1100 alloy aluminum. Tests of the extrusion indicated mechanical bond only between the steel and aluminum. The results indicated that improvements could be expected by increasing the temperature of operation.

The ability of NS355 were to withstand elevated temperature without loss in room temperature properties was determined. The parameters obtained will be used in the high temperature extrusion equipment design. It was found that the properties of the wire were not affected if the wire passed through a one inch column of aluminum at a rate exceeding 5 ft./min. at  $1200^{\circ}F$ .

## 2. Rolling

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a. It was found that 0.010" steel wire could be rolled, in the direction of the long aris of the wire, into aluminum sheet without breaking the wire provided the movement of the aluminum did not exceed the elongation of the wire. This method has proved advantageous by cold working the age hardening alloy 2%, after solution heat treatment and prior to aging.



b. Strands of 0.010" NS355 wire were rolled into 5052 and 2219 sheet using a 5" diameter roll. Percent reductions were increased from 5 to 55%. The wire was embedded in the aluminum between 10 and 50% reduction; above 50% the wire fractured. The calculated roll pressure at time of fracture was approximately the same as experienced when cold pressing with a 1/2" die. The tests indicate that wire can be distributed in the aluminum matrix by the rolling process.

## 3. Pressing

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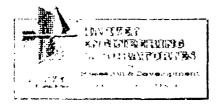
a. <u>Cold</u> - Cold pressing areas of 36 square inches at 100 tons/sq.in. resulted in poor bond and wire fracture. Reducing the area pressed to 1 sq. in.  $(\frac{1}{2}$ " x 2") and the pressure to 50 tons/sq.in. successfully distributed the wire without breaking in thin aluminum sheets (0.013"-0.026"). However, the bond between the steel and aluminum was insufficient for uniform fracture.

When a cold pressing 0.004" wire using the same tooling used for the 0.010" wire, wire breakage resulted. This difficulty was overcome by reducing the width of the pressing punch from 1/2" to 1/4" and rounding off the edges.

b. Hot - Hot pressing at temperatures of 850 to  $950^{\circ}F$  using reduced pressure (3 tons/sq.in.) has resulted in samples which exhibit a uniform fracture and a strength equal to or better than 95% of the theoretical.

The hot press tooling was reworked to accommodate 3/4" samples. One 3/4" sample was produced using 2219 alloy.

4. <u>Cold Drawing</u> - An effort was made to improve on incremental cold pressing by drawing the aluminum and steel wire through a tapered die. Difficulties encountered were wire breakage and galling of the die. These difficulties could be overcome; however, pressing and rolling appeared more expeditious and efforts were temporarily discontinued.



## C. Bonding

1. <u>Aluminum to aluminum</u> - Five approaches have been followed to overcome the difficulties in bonding aluminum caused by aluminum oxide on the surface of the aluminum.

- a. Movement of the surface
- b. Covering the surface so it will not oxidize
- c. Cleaning and bonding by the explosive method
- d. Ultrasonic welding
- e. Diffusion bonding at high temperature

Movement of the aluminum surface and explosive bonding have produced the most satisfactory results to date and work is continuing along these lines.

Evaluation tests were made with aluminum strips coated with various combinations of indium, tin, zinc silver and copper in an effort to get better bonding at temperatures below  $400^{\circ}$ F. These coatings did improve the low temperature bonding over uncoated strips which exhibited no bond; however, peel tests indicated that not sufficient strength was achieved, considering the other complications, to warrant continuation at this time.

2. <u>Aluminum to Steel Wire</u> - Tests were made on factors affecting the aspect ratio using 0.010" NS355 wire and 2024-0 aluminum. Three factors were found to improve the aspect ratio: Scratch brushing in a direction transverse to wire direction, heat treating the 2024 to T4, and reducing the spacing between the wires.

3. Assembly of Composite Strip

a. <u>Cold</u> - Assembly of composites at temperatures below  $400^{\circ}$ F have resulted in mechanical bonds only with the exception of explosive bonding.

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b. Hot - Assembly of composites by hot pressing in the  $850^{\circ}$  950 F range has resulted in tensile test specimens which fracture uniformly. However, metallurgical examination gave no indication of alloying.

#### D. Joining

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Modules consisting of two pieces of .006"5% silicon clad 3003 aluminum and one layer of 0.010" NS355 wire one inch wide by eight inches long were hot pressed at  $950^{\circ}F$ . The modules were then cut and the pieces assembled and hot pressed to made a test sample three modules thick with three overlapping joints. The over lap was 1/2" thus making the total joint length 1" in the center of the test bar. (see sketch below)

Three samples were made; (1) A control sample with no joints; (2) A three joint sample with no over lap at each joint; (3) A three joint sample with 1/8" overlap at each joint. Test results of the three samples are listed below:

	Tensile Strength	% Efficient
	Psi	
1.	138.000	100%
2.	88.700	64.5%
3.	107.000	77.5%

The results indicate that good load transfer around a joint is possible; i.c. In sample No. 2 expected efficiency would be 66.67% on the basis that there is only one joint in any given pross section.



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## E. Mechanics Testing of Samples

The samples tested to date have lead to the observations listed below:

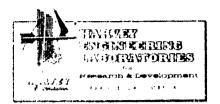
1. The ultimate tensile strength achieved when the aspect ratio is 27/1 is determined by the tensile strength of the wire and the proof strength of the aluminum matrix at the limit of elongation of the wire.

2. If there is no bond between the wire and the matrix the strength of the wire only is realized.

3. The ultimate tensile strength of the composite is increased when the strength of the matrix is increased by heat treatment and working provided the wire is not adversely affected by the operation.

4. The yield strength of a 2024 sample containing 19.2% NS355, 0.010" steel wire was increased from approximately 70,000 psi to 120,000 psi by cold rolling (from 0.115" to 0.107") after solution heat creatment (within 1 hour); i.e., the 70,000 psi Y.S. sample was T-6 and the 120,000 psi Y.S. sample was T-3. Since the increase in yield strength is greater than is to be expected from a change from T-6 zo T-3 condition, it is felt the addition yield strength is coming from an autofrettage effect.

5. The type of stress strain curve obtained is sepandent on residual internal stresses remaining in the sample then it is tested, i.e. if the aluminum is in tension (reacting) from thermal expansion differences) the curve exactifies a definite change in slope when the yield strength of the aluminum is exceeded. If the aluminum is in compression then a smooth stress strain curve indicating a modulus of 15 x  $10^6$  psi results.



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6. Properties of NS355, 0.010" wire were determined after various exposure times to elevated temperatures Ly testing at room temperature and  $-320^{\circ}F$ :

a. The wire was not significantly affected by exposure at 950°F for up to 20 hours.

b. Above 950°F the properties of the wire, when tested at toom temperature, are dependent on time; i.e., the shorter the time the higher the temperature of exposure without affecting the room temperature properties.

c. In liquid nitrogen an average increase, in tensile strength, of approximately 24% was realized over average room temperature properties.

7. Carbon steel rocket wire was tested at room temperature and at -320°F. When tested in liquid nitrogen 0.010" wire increased in strength by approximately 14% and the 0.004" wire by approximately 12%.

8. Using the hot pressing procedure developed for fabricating tensile test specimens, the aspect ratio of 27/1 was established for 0.010" NS355 wire in 2024 aluminum.

9. Explosively bonded 5052 sheet exhibited an increase in tensile strength from 26,300 psi to 35,190 psi and a loss in elongation from 20% to 5%.

10. The highest strengths obtained to date are 176,520 psi with 2024 aluminum and 26% Q010"wire and 158,500 psi with 2219 aluminum and 25% Q010"wire.



## II. ACCOMPLISHMENTS SINCE LAST REPORT

## A. Literature and Industrial Survey

Literature reviewed on composite materials fabrication processes and materials properties are reported in the annotated bibliography in Appendix I.

## B. Wire Distribution

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1. Extrusion-Hot - Basic parameters for extrusion of 0.020" 2219 aluminum alloy were established. It was found that 2219 could be extruded into 0.020" diameter wire at a pressure of 102 tons/sq.in. at 950 F at an extrusion ratio of 625/1.

Using the extrusion formulas  $P = K \ln \frac{A}{a}$  and P = d - cT where

- P = extrusion pressure in tons/sq.in.
- K = constant (resistance to deformation)
- A = Cross section area of container
- a = Cross section area of extrusion
- d = Constant
- c = Constant
- T = Extrusion temperature in <sup>o</sup>c

and assuming the ratio of  $\zeta = 0.00175$  it is estimated the extrusion pressure can be reduced by 20% if the extrusion ratio is lowered from 625/1 to 156/1 or by 65% if the temperature is increased to  $1020^{\circ}$ F.

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## 2. Rolling - Cold:

Multiple 0.010" NS355 wires(0.017" o.c.) were rolled into 0.026" 2219 alloy without wire fracture; however, the wires did not bond to the aluminum. It is expected this difficulty can be overcome by increasing the temperature Tooling was designed to obtain parameters for hot rolling.

#### C. Bonding

Lithium metal was evaluated as a low temperature diffusion aid. Samples of 2219 were cleaned and coated with Lithium in an argon atmosphere and then pressed at 400°F for 2 to 48 hours at pressures from 10 to 40 tons/sq.in. The results were not considered sufficiently encouraging to warrant further effort.

## D. Joining

Four joining samples, composed of modules of 0.009" NS355 wire and 0.006" 3003 aluminum, were produced as previously reported and tested as listed below:

a)	Five modules lay-up ; 1/2" between joints (Total joint length 2")	77,200 <b>psi</b>
ь)	Same as (a) with pads over joint	90,700 psi
c)	Same as (a) with two joints doubled back (Total joint length 1")	98 <b>,300 psi</b>
d)	Same as (a) but 1/4" wire overlap at each joint	117,400 <b>pai</b>

Laboratory work on the control samples was not completed during this period; consequently data on efficiency of the above samples will be in the next report.



## E. Test Samples

1. The metallurgical department of National Standard Company (at Niles, Michigan) was consulted as to the possibilit of obtaining increased strength in their NS355 type wire. Their suggestion was a stress relief at  $750^{\circ}F$ . Variation of ultimate tensile strength with stress relief temperature for 0.006" wire is listed below:

Room Temperature	470. Ksi
Stress Relief	
700°F 1 hr.	500, Ksi
750°F 1 hr.	500. Ksi
800°F 1 hr.	500. Ksi
900°F 1 hr.	485. Ksi
1000 <sup>°</sup> F 1 hr.	470. Ksi
1100°F 1 hr.	382. Ksi

2. The three shipments of NS355 wire on hand were given a stress relief of 1 hour at  $750^{\circ}F$  and compared with as received properties as well as other exposure times and temperature as listed below:

a.	Diameter	0.004″	0.009"	0.010"
	As Received	493.Ksi	480. Ksi	504. Ksi
	1 hr. 750 <sup>0</sup> F.	490.Ksi	504. Ksi	494. Ksi

b. NS355 0.009"Wire

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		eived	480.	Ksi
20	Min	950 <sup>0</sup> F	485.	Ksi
40	Min	990°F	480.	Ksi
90	Min	990°F	460.	Ksi

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c. NS355 0.010" Wire

As	Rece	eived	504.	Ksi
30	Min	910 <b>°</b> F	488.	Ksi
30	Min	930 <b>0</b> F	488.	Ksi
		950 <b>°</b> F	484.	Ksi
30	Min	970 <sup>0</sup> F	478.	Xsi
30	Min	990 <b>°F</b>	475.	Ksi
30	Min	1110 <b>°</b> F	466.	Ksi

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The tentative conclusion is that, if the as-received NS355 wire is at its maximum strength, it will suffer some loss in strength on further exposure to temperatures in the range of  $800^{\circ}$ F to  $1000^{\circ}$ F, and if the wire contains some residual stresses an increase in strength may be realized by heating in the range of  $700^{\circ}$ F to  $900^{\circ}$ F. We have requested that National Standard investigate the effect of varying time, temperature on the stress relief treatment and to determine consistancy of response to stress relief of several lots.

2. Considering the variation in wire strength with temperature in the heat treat range for 2219, it was decided to survey this area to determine the processing temperature which would produce the best overall properties for the composite. Hardness tests on 2219 sheet 0.026" thick with varying solution heat treatment and aging time are listed.

	Rockwell 1/16" Ball Age 325°F	15 Kg	
S.H.T. Temp.	20 hrs.	24 hrs.	28 hrs.
800°F	67	69 (28.3 K#1)*	69
900°F	75.5	79.5 (45.4 Ksi)*	80
1000 <sup>9</sup> F	83,5	84.5 (54.5 Ksi)*	84.5

\* Tensile strength at 2.0% elongation.



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On the basis of 25% by volume wire it is evident that with 2219 any increase in strength in the wire developed using a lower solution heat treat temperature will be more than off set by loss in properties of the aluminum.

3. A test sample was cut and tested from the 3/4" sample produced last month. The sample with 23.2% wire pulled 143.4 Ksi.

If the wire is figured at 460. Ksi (strength after exposure to  $990^{\circ}$ F) the calculated strength at 25% wire would be 150.5 Ksi.

F. Hours Expended

During the 16th month period of effort, 432.6 hours were expended with the following approximate breakdown:

Engineering	61%
Shop	33%
Reporting	4%
Other	2%

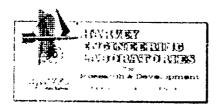


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# III. ANTICIPATED WORK FOR NEXT REPORTING PERIOD

Continue with development work on rolling 0.010" and 0.004" diameter wire into strip modules and joint and bonding samples. Start work on wire distribution system for full size plate.



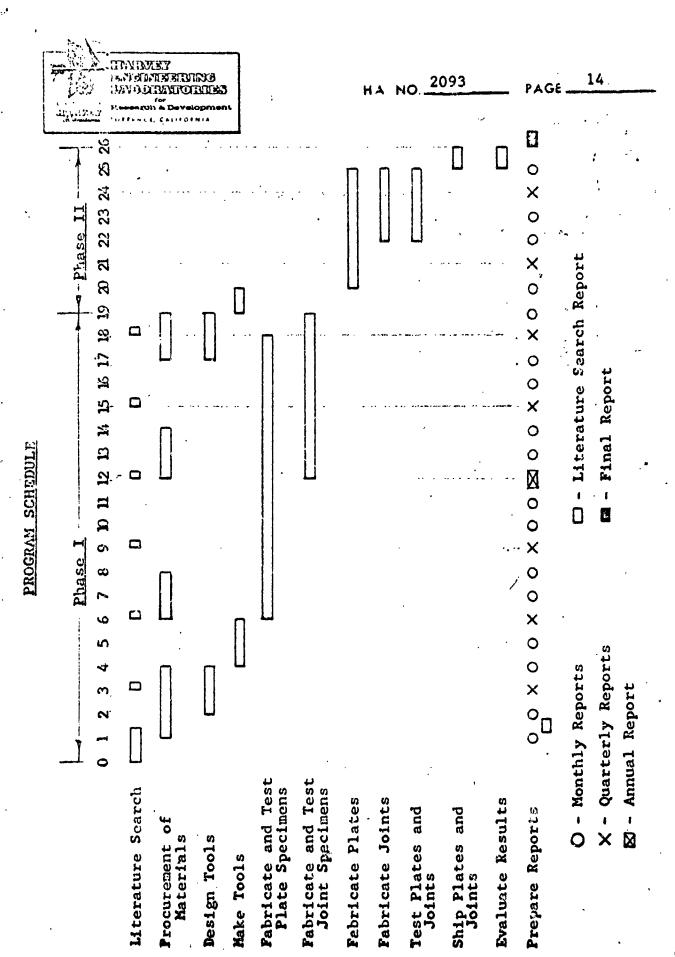
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# IV. SCHEDULE CHANGES AND PROBLEMS ENCOUNTERED

No changes in the program schedule are anticipated at this time.





## APPENDIX 1

#### ANNOTATED BIBLIOGRAPHY

## COMPOSITE MATERIALS

<u>Preparation and Properties of Fiber-Reinforced</u> <u>Structural Material</u>: Battelle, DMIC, Columbus, Ohio, DMIC Memorandum 176, August 22, 1963.

Covers a review of methods of preparation and properties of various composite types including: metal-metal (by powder metallurgical methods and by infiltration of molten matrix), metals reinforced by ceramic fibers, ceramics reinforced by metal fibers, and other nonmetallics reinforced by metal fibers.

<u>Summary of the Eighth Meeting of the Refractory</u> <u>Composites Working Group</u>: Battelle, DMIC, Columbus, Ohio, DMIC Memorandum 192, April 20, 1964.

Contains abstracts of papers presented by attendees at Fort Worth in January 1964 covering: (1) Materials Technology, (2) Process Technology, (3) Specific Hardware, (4) Evaluation Techniques pertaining to application of refractory composites for use at temperatures above 2500°F.

Structural Behavior of Composite Materials: Ford Motor Company, Aeronutronic Division, Contract NAS7-215, NASA, Washington, D. C. Progress Report No. 6, August 1963 (2nd Quarterly).

Covers theoretical prediction of the mechanical constitutive equation of laminated anisotropic plates with special emphasis on cross-ply and angle-ply composites.



Some Studies on the Nature of Deformation in Composite Materials: Massachusetts Institute of Technology, Cambridge, Mass., AIAA Launch & Space Vehicle Shell Structures Conference, Palm Springs, California. Report No. 2924-63, April 1963.

Covers studies of (1) the thin joint in tension, (2) the thin joint in shear, (3) the filamentary composite and (4) the particulate composite. Experimental data were analyzed from the standpoint of increasing understanding to enable more successful composite design. Composites include a silver brazed steel joint, steel wire reinforced silver matrix, and rounded tungsten particles in a copper matrix.

Effect of Couple-Stresses on Force Transfer Between Embedded Microfibers: U.S. Army, Watervliet Arsenal, Watervliet, New York. Report WVT-RR-6407, June 1964.

This report is a continuation of the Weitsman problem. The investigation is concerned with the nature of the stress field which develops in the presence of couple-stresses in composite material formed of microfibers embedded in a filler substance. The case considered when one row consists of an infinitely long microfiber, while the second row is composed of two semi-infinite microfibers.

Principles Affecting High Strength to Density Composites with Fibers or Flakes: A.F. Materials Laboratories, Wright-Patterson Air Force Base. Report No. ML-TDR-64-85, May 1964.

Contains a summary of theoretical background as applied to the mechanics involved in fiber reinforced composites.

Boron Fiber Reinforced Structural Composites: ASD, Wright-Patterson Air Force Base, Report No. 2926-63, April 1963.

Contains a resume of work on production of boron fibers by vapor deposition and fabrication of composites of boron fibers in plastic by coating to form tapes which are then bonded together.



## FABRICATION PROCESSES

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Development of Ultrasonic Welding with Emphasis on <u>Producing Hermetic Seals</u>: Aeroprojects, Inc., West Chester, Pa. Contract DA-36-034-0RD-3254 RP, Pittman Dunn Laboratory., Frankford Arsenal, Philadelphia, Pa. Report No. RR-61-99, September 1963.

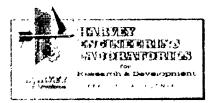
The work was conducted in two phases: I - Investigate the feasibility of hermetically sealing certain configurations and II - Investigate continuous seam welding of materials significant to missile fabrication. Results show that hermetically sealing and continuous seam welding by ultrasonics is feasible; e.g., successful welds were made in 0.040 and 0.064 inch 2014-T6 aluminum alloy at 1 ft./min. with a 5 KW welding unit.

<u>Press Welding of Rolled Sections of Aluminum-Magnesium</u> <u>Alloys</u>: Institute of Electric Engineers of Academy of Science, U.S.S.R., February 1962.

Describes investigation of press welding of Al-Mg by heating above critical point  $Ac_3$  and joining by applying pressure. Properties of joints were equal to those of the base except for impact strength which was 50% lower. This is explained by differences in the grain flow patterns and inherent anisotrophy of the Al-Mg alloys.

Study of the Feasibility of Applying Ultrasonic Energy to the Rolling Process for Sheet Metals: Westinghouse Electric Corporation, Pittsburgh, Pa. Contract NOW 64-0294-J, BuWeps, First Bimonthly Report, April 1964.

Plans are made for study of the effect of ultrasonic vibrations on rolling of aluminum  $(1\frac{1}{2}" \times 3")$  in a 2-4 high 8" x 8" Stanat mill by application of the energy (1) to the workpiece and (2) to the rolls.



## Effect of Surface Preparation and Elevated Temperature Storage on Adhesive Bonds to Aluminum: U. S. Army, Picatinny Arsenal, Dover, New Jersey

Covers a study of two room-temp-curing epoxy-type adhesives with two chemical methods of cleaning the aluminum, on long time storage at temperatures of  $-54^{\circ}$ ,  $23^{\circ}$ , and  $71^{\circ}$  C.

## MATERIALS PROPERTIES

<u>Strengthening Mechanisms in Wire Products</u>: Crucible Steel Co. of America, Pittsburgh, Pa. Contract AF 33(615)-1684, AF Material Lab, USAF, Wright-Patterson Air Force Base, Ohio. First Quarterly Report, September 1964.

The primary purpose of this project is to explore the additivity of different strangthening mechanisms available in high-strength metallic materials in fine wire form. Representatives of high strength carbon and alloy steels, stainless steel, and superalloys are included in the program. In the initial phase, attention has been focused on the new high strength hardenable stainless steel AFC-77A. Significant strengthening has been obtained by combinations of tempering and cold working steps. Tensile strengths at the 500,000 psi level were attained by several different, relatively simple routes.

<u>Ultra-Fine High Temperature. High Strength Metallic</u> <u>Fibers</u>: Hoskins Manufacturing Co., Detroit, Michigan. Contract AF 33(616)-8366, M&P, ASD, Wright-Patterson Air Force Base, Ohio. Report No. ASD-TDR-62-727-Part 1 August 1962.

Eight superalloys were processed to ultra-fine fibers of approximately .001 inch diameter and evaluated for drawability, room temperature tensile strength and elongation, and effect of reduction of area on tensile properties. Elgiloy and Hastelloy B alloys processed more readily with less wear than the other alloys.



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Prestressed Monolithic and Segmented Brittle Structures: I.I.T. Research Institute, Chicago, Illinois. Contract NASr-65(04) NASA, Washington, D. C. NASA Centractor Report NASA CR-113, October 1964.

Covers studies of stresses in segmented columns and monolithic structures. The strength of prestressed monolithic brittle beams is formulated in terms of reliability, and a specific example is treated in which the prestress leads to a twenty-five fold increase in strength when compared to a conventional beam of equal weight and reliability.