

## PROCESSING OF SUPERALLOY MULTIFILAMENT YARN

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ABSTRACT

Eight superalloys of A-286, Elgiloy, Hastelloy B, M-252, Rene 41, Udimet 500, Udimet 700 and Waspaloy were processed to ultra fine fibers of .001 inch diameter or less and their room temperature physical properties evaluated. All alloys except A-286 had ultimate strengths in the range of 160,000 to 220,000 psi as solution heat treated. Alloy A-286 had a tensile strength of 100,000 psi.

Multifilament yarns composed of seven (7), nineteen (19) and thirty-seven (37) filaments of Elgiloy and Rene 41 were sheathed with a Chromel-C alloy ribbon and processed to .0027 inch diameter. Tensile strengths were determined for the sheathed yarns at 1600°F., 1800°F., and 2000°F. in air and argon. The oxidation resistant Chromel-C considerably improved the 1800°F. and 2000°F. tensile strength of the Rene 41 alloy, but the effect on Elgiloy was slight.

INTRODUCTION

The research project on processing of various superalloys to fine wire and then fabrication into multifilament yarn for further reduction was sponsored by the Aeronautical Systems Division under USAF Contract No. AF 33(616)-8366 and monitored by the Fibrous Materials Branch of the Nonmetallic Materials Laboratory, Directorate of Materials and Processes. The metallic filaments and yarns are needed for fabrication into heat and shock resistant metallic cloths so necessary for aerospace applications. The present organic fibers cannot withstand the frictional heat and high shock loads encountered in outer space and re-entry applications. The alloys selected for this research study were A-286, Elgiloy, Hastelloy B, M-252, Rene 41, Udimet 500, Udimet 700 and Waspaloy. The selection was based on availability of material, physical properties compatible with end requirements, and previous wire drawing experience that would insure some degree of success in processing to fibers. The nominal compositions are noted in Table 1.

TABLE 1

Nominal Chemical Compositions of the Superalloys

<u>Alloy</u>	<u>Chemical Composition - %</u>								
	<u>Mn</u>	<u>Si</u>	<u>Cr</u>	<u>Ni</u>	<u>Co</u>	<u>Mo</u>	<u>Ti</u>	<u>Al</u>	<u>Fe</u>
A-286	1.35	0.5	15	26	-	1.25	2.0	-	Bal.
Elgiloy	2.0	-	20	15	Bal.	7.0	-	-	16
Hastelloy B	1.0	1.0	1	Bal.	2.5	28	-	-	6
M-252	0.5	0.5	19	Bal.	10	10	2.5	1.0	2
Rene 41	-	-	19	Bal.	11	10	3.1	1.5	3
Udimet 500	-	-	19	Bal.	19	4	3.0	2.9	4
Udimet 700	-	-	15	Bal.	19	5	3.5	4.5	4
Waspaloy	-	-	19	Bal.	14	4	3.0	1.3	1

In order to utilize metallic fibers in woven structures, the material must have flexibility, or low bending rigidity. Metallic filaments, as a class, tend to be 10 to 20 times stiffer than organic fibers of equal tensile strength.<sup>(1)</sup> Metal filaments would have to be drawn less than 1/4 the diameter of organic filaments for equal rigidity and strength. The metal fibers must have uniform fiber strength and high ultimate elongation for optimum fabric properties.<sup>(2)</sup> These property requirements also apply if the filaments are processed into yarn prior to weaving into fabric form. Non-uniformity in strength and elongation will result in uneven loading across the yarn cross-section during the weaving operation causing occasional filament failure in extreme cases and/or variable permeability in the woven fabric in others. In this research study individual wire fibers were to be bunched as a yarn and the multifilaments drawn through dies as a means of reducing the diameter of the yarn which would result in smaller filament diameters. The minimum diameter of commercially available superalloy wire is 0.5 mil. The development of a successful process for drawing multifilament yarn would not only provide a more flexible fibrous product, but should effect economies in the production of ultra-fine fibers.

The problems encountered in processing the multifilament yarn to smaller diameters are influenced considerably by the physical properties of the individual wires or filaments. Very little is known of the physical properties of any of the superalloys in wire form. Each of the selected alloy compositions had to be processed to ultra-fine wires in order to determine the optimum wire drawing procedures and conditioning operations. General wire drawing experience indicates that most alloys will process easiest when in the most ductile condition.

Annealing conditions that produced the most ductile wire had to be investigated and determined. Many alloys cannot be processed to fine wire because of segregation, non-metallic inclusions, impurities and other metallurgical factors which are difficult to control in the melting or subsequent processing stages.

The superalloys, by nature of their complex metallurgical systems, are exceptionally sensitive to processing conditions. All the alloys studied were vacuum melted except the electric furnace air melted Elgiloy and Hastelloy B alloys. These alloys were successfully processed as air melted so further melting techniques were not investigated.

In addition to the development of drawing processes for multifilament yarn, the final product was to be tested at room temperature and higher. The high temperature tests were conducted at 1600°F, 1800°F, and 2000°F. in both air and argon atmospheres. Ideal geometric configurations of yarn with 7, 19 and 37 filaments were processed in the program. All the alloys were drawn to fine wire but to date only Elgiloy and Rene 41 were processed and tested as yarns.

## PROCEDURES AND RESULTS

### A. FIBER PROCESSING

#### 1. WIRE DRAWING

Since the superalloys are a class which has little commercial usage as fine wires and are so difficult to process, there is limited data available. Past wire drawing experience at Hoskins on related alloys, and limited experience with this particular alloy group indicated that most wire problems were encountered at processing sizes less than .005 inch diameter. It was at this size and smaller that a detailed history was kept of each alloy during the various stages of processing. In order to compare the fine wire physical properties of the alloys, processing variables were kept at a minimum as conditions permitted.

The general process procedure followed for all alloys was to continuous anneal in a dissociated ammonia atmosphere at optimum temperatures the .005 inch or .004 inch diameter of as received wire, draw in diamond dies to .002 inch diameter, continuous anneal, redraw to .001 inch diameter, continuous anneal and redraw if possible to a contemplated final filament size of .0005 inch diameter and re-anneal. This procedure was followed quite closely with few exceptions. A few of the alloys could not be drawn to .0005 inch diameter so the final comparative size was changed to .0006 inch diameter as a more realistic goal.

All the wire was drawn in a tub type fine wire drawing machine. A lubricant of 40% water soluble Draw Cool X oil plus 20% Johnson's Wax with the remainder water was used. The wire was drawn one B & S\* gage reduction per pass to .001 inch diameter and approximately one-half B & S gage reductions to .0005 or .0006 inch diameter. Drawability problems and excess die wear were promoted by contaminated wire surfaces. Thin oxide films, as a result of annealing, were very abrasive to diamond dies. Insufficient cleaning off of lubricants prior to annealing would leave a residue after the anneal. Scratches, slivers, burrs, etc. all contributed to die wear and wire breakage.

(\*Brown and Sharp)

The fine wire was continuous annealed in a 30 inch long electrically heated furnace at approximately 140 feet per minute and at a range of temperatures compatible with each alloy that would result in the most ductile wire.

All the alloys were processed to approximately .001 inch diameter with Hastelloy B, Rene 41 and Waspaloy going to .0006 inch diameter. Die wear during the drawing operations was excessive for all alloys except Elgiloy and Hastelloy B. The alloys which processed the easiest were the air melted Elgiloy and Hastelloy B metals. Comparative studies between air and vacuum melted super-alloys generally favor the vacuum melted alloys as having superior working properties due to less inclusions and gasiness. In this instance the expected results were reversed. The answer can possibly be traced to the composition variables. All the alloys, but Elgiloy and Hastelloy B, have major additions of either titanium or aluminum or both. These two elements are known to combine with each other or other trace elements in the alloy to form complex intermetallic compounds. These compounds are beneficial in improving the high temperature strength of the alloys, but are detrimental to hot and cold forming. The aluminum and titanium also form complex oxides which are abrasive and contribute considerably to die wear.

The first lot of Elgiloy to be melted and processed would not readily draw to fine wire, while a second lot was relatively trouble free. The two heats were processed alike and microscopic and metallographic examination could detect no apparent differences. Past experience has indicated that there is no sure way of determining the drawability of a heat of superalloy or other conventional alloy except by actual practice. Any metallic or non-metallic inclusions become proportionately larger to wire size as the wire is drawn smaller and smaller. Surface imperfections are also magnified. Slivers, burrs, pits, etc. that were incidental at .010 or .005 inch diameter wire could be quite a factor at .001 or .0005 inch diameter.

The A-286 alloy was the most difficult to process as indicated by excessive wire breakage in drawing. It is an iron base alloy and had the lowest tensile strength and ductility of all the alloys. The U-500 and U-700 alloys had relatively high percentages of aluminum and titanium which contributed to drawing problems. Die wear was considered excessive if wire drawn through a .001 inch diameter die increased in size .0001 inch or greater for 0.10 lb. or less of drawn wire.

## 2. PHYSICAL PROPERTIES

The effect of annealing temperatures on the tensile strength, yield strength, and elongation on all the alloys at .002 inch diameter are illustrated in Figure 1. Figures 2 and 3 illustrate the effect of cold reduction on the tensile strengths and yield strengths of the alloys when processed from .002 inch diameter annealed wire.

## B. MULTIFILAMENT YARN PROCESSING

### 1. YARN DRAWING

All yarn drawing was accomplished by pulling it through a single die

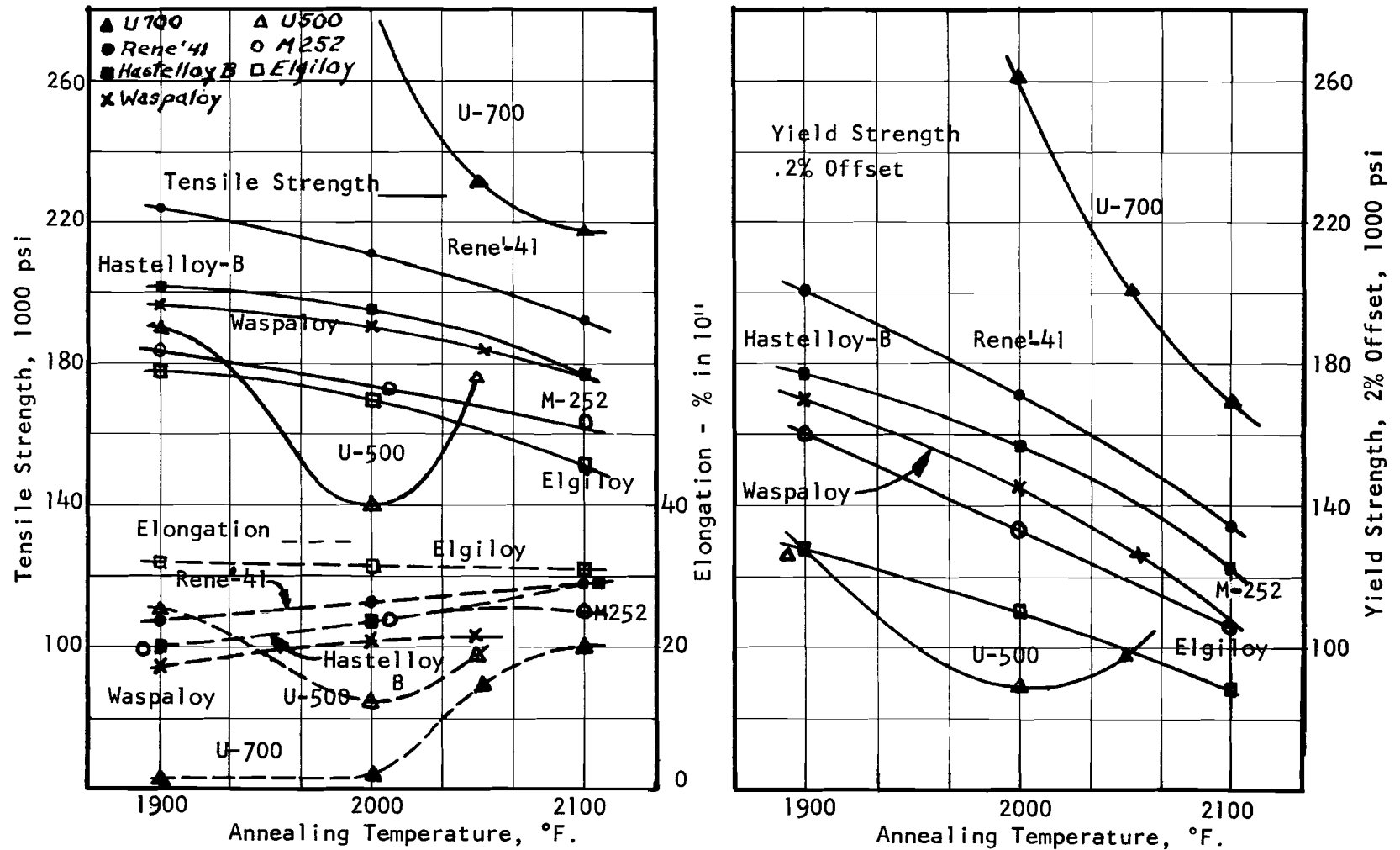


FIGURE 1. Effect of Annealing Temperatures on .002" Dia. Fibers.

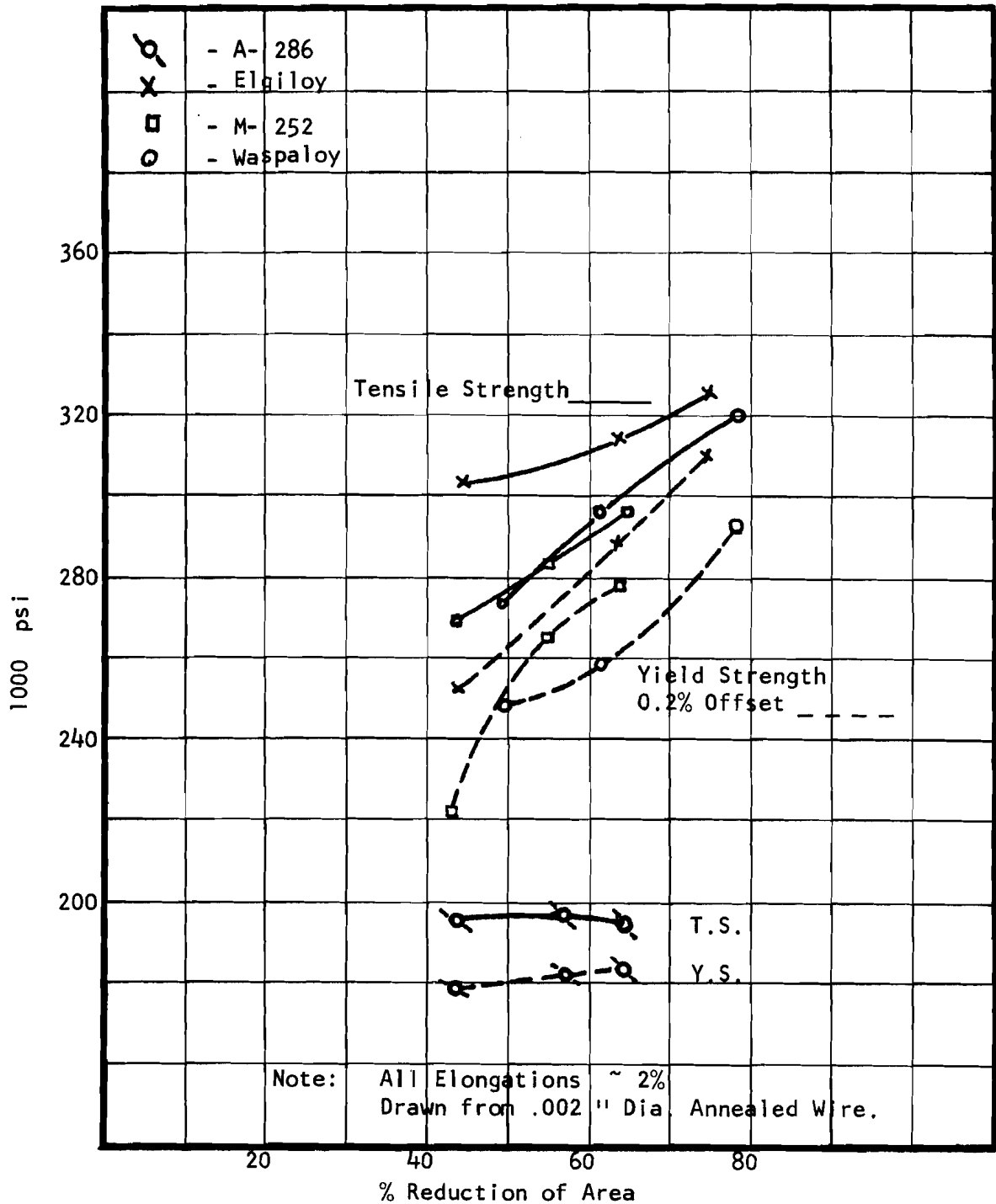


FIGURE 2 Effect of Cold Reduction on Properties of A-286, Elgiloy, M-252 and Waspaloy.

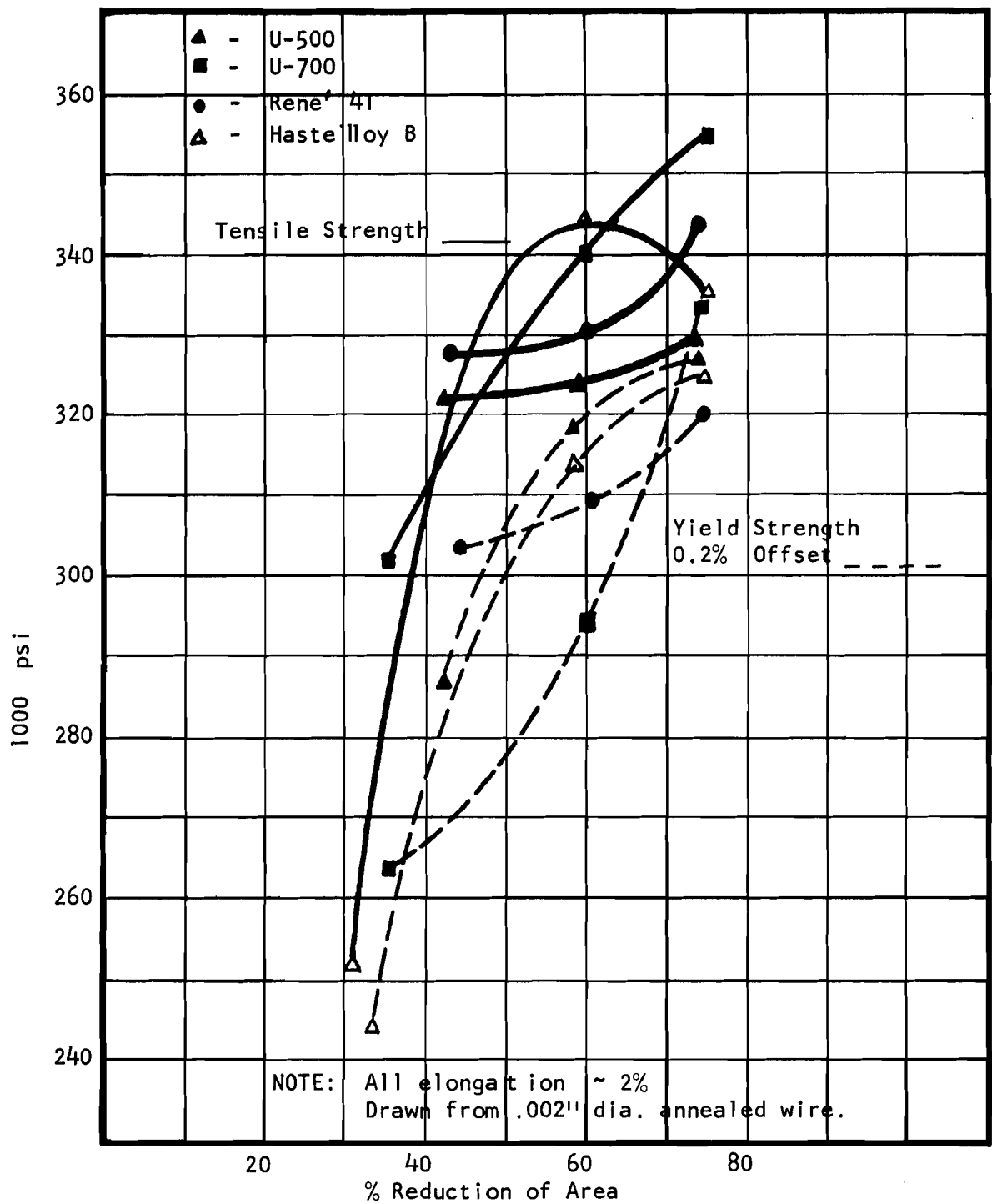


FIGURE 3 Effect of Cold Reduction on Properties of U-500, U-700, Hastelloy-B and Rene' 41.

one-half B. & S. gage reduction per pass either by hand or with a slow speed motorized take-up at approximately 20 fpm. Initial attempts to draw twisted yarn with high viscosity grease as a lubricant failed due to the lubricant breakdown. A number of different twists per inch were tried in an attempt to determine the optimum number for drawing, but it appeared that zero twist produced the best results. Evidently the twists presented an uneven surface at the periphery of the yarn and as it was drawn through the die, the tangential pressure to the axis of the filament caused the filaments to bunch up and break. The number of turns per inch also decreased as the yarn was reduced in diameter due to the increased length as the cross-section became smaller. Conceivably, if the yarn was reduced in cross section sufficiently, the final result would be a no-twist end product.

The most successful method for reducing the yarn diameter was to encapsulate it in a metal sheath and draw the composite. A method was devised for the continuous encapsulation of multifilaments of yarn using Chromel-C (60 Ni - 16 Cr - 24 Fe ) ribbon. Refer to Figure 4. Small quantities of 7, 19, and 37 strands of .002 inch diameter wire of Elgiloy and Rene 41 were encapsulated and the composite drawn to .0027 inch diameter for physical property tests. Elgiloy and Rene 41 were selected for the limited comparative tests because of previous extensive investigation of these alloys by other organizations on related projects. The first lot of encapsulated yarn disclosed welding of the filaments. Thereafter, subsequent to bunching and encapsulation, all filaments were annealed in a slightly oxidizing atmosphere. The thin oxide layer formed a barrier to diffusion welding. A cross-section of 37 filaments of Elgiloy in a sheath drawn to .0027 inch O.D. is illustrated in Figure 5.

## 2. PHYSICAL PROPERTIES

All physical property tests on the multifilament yarn were conducted on the Chromel-C sheathed fibers. The effect of the sheath on the properties could not be readily determined, but it must have had some bearing on the test results. Considering the .0027 inch diameter sheathed yarn, the approximate actual total superalloy fiber cross-sectional area as compared to total composite area of each configuration was as follows:

7 strands	- 37%
19 strands	- 52%
37 strands	- 78%

All data tabulated and recorded is on the composite. The room temperature tensile strength and elongation properties of the different yarn configurations annealed at temperatures ranging from 1800°F. to 2100°F. for Elgiloy and Rene 41 are illustrated in Figure 6. Elgiloy appears to have better strength and ductility than Rene 41. Figure 7 indicates the effect of cold reduction on the yarn properties. The Elgiloy results of Figure 7 are in the expected sequence for the 7, 19 and 37 strand configurations when considering the effective cross-section area of the superalloy fibers. The greater ductility of the Elgiloy may account for the higher strength as some of the less ductile Rene 41 fibers may be fractured under the sheath. Fiber breakage may account for the lower tensile strength with increased reduction of area and this would be especially true of the 37 strand yarn as the fibers are smaller and in the order of .0003 inch diameter.



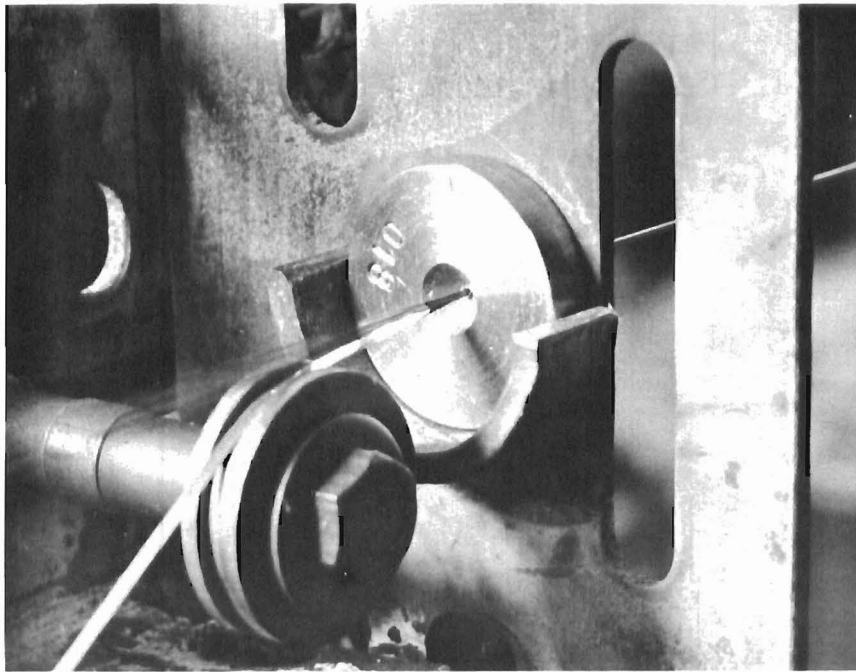


FIGURE 4 Forming Ribbon Sheath Around Multifilaments

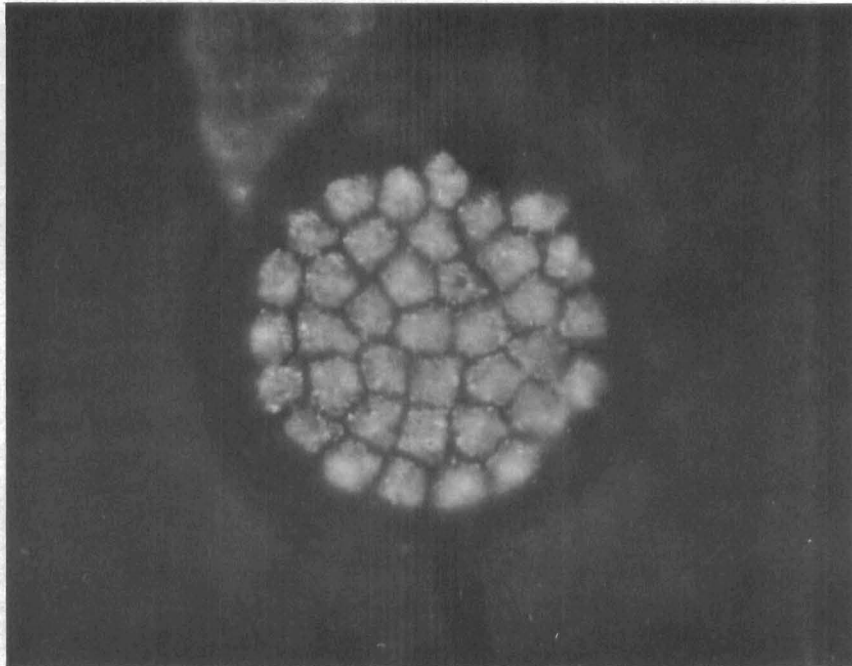


FIGURE 5 Thirty-seven Strand Elgiloy in Chromel-C Ribbon Sheath, .0027" Outside Diameter, 900X.

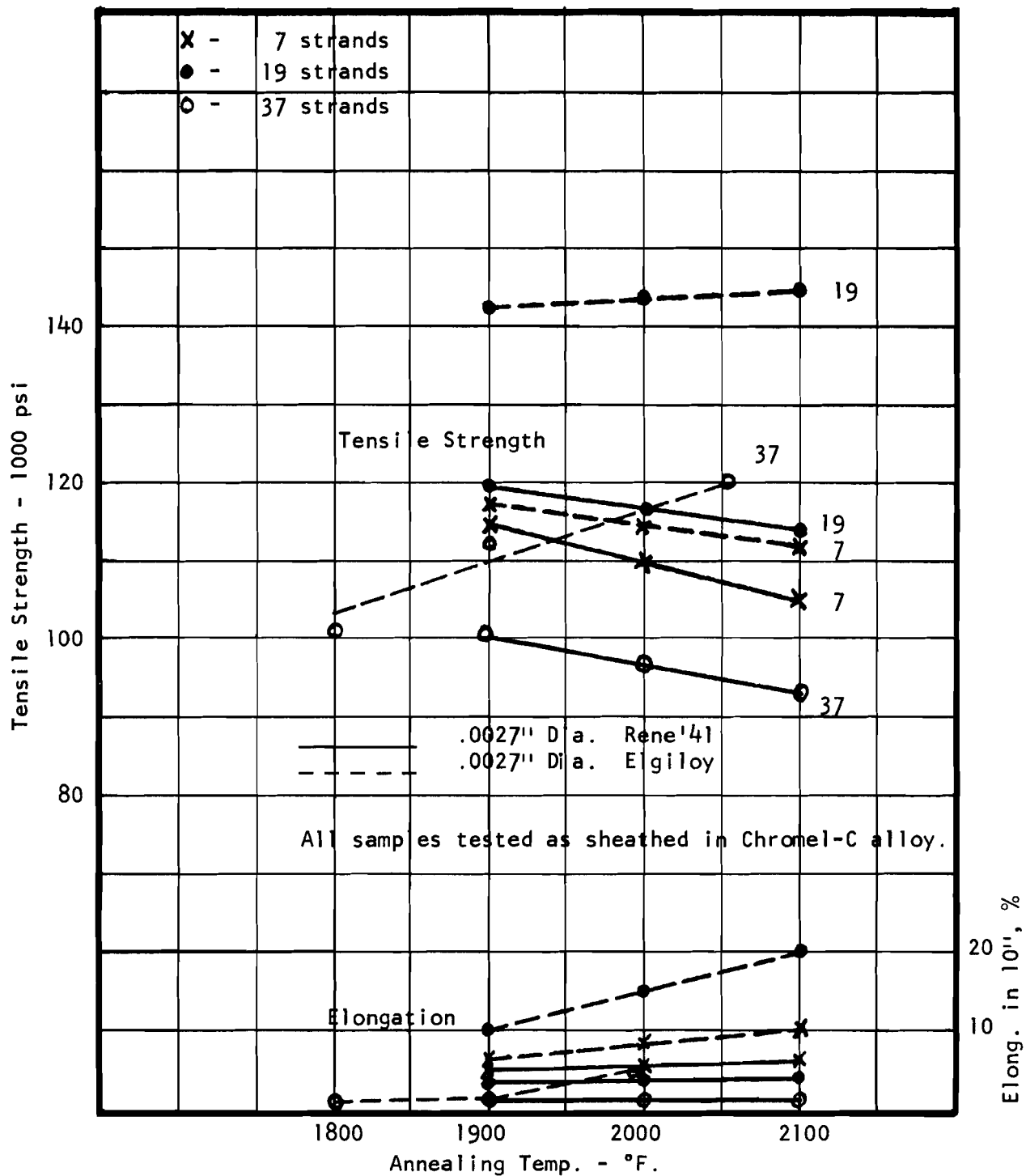


FIGURE 6 Effect of Annealing Temperature on Properties of Elgiloy and Rene'41 Multifilament Yarn.

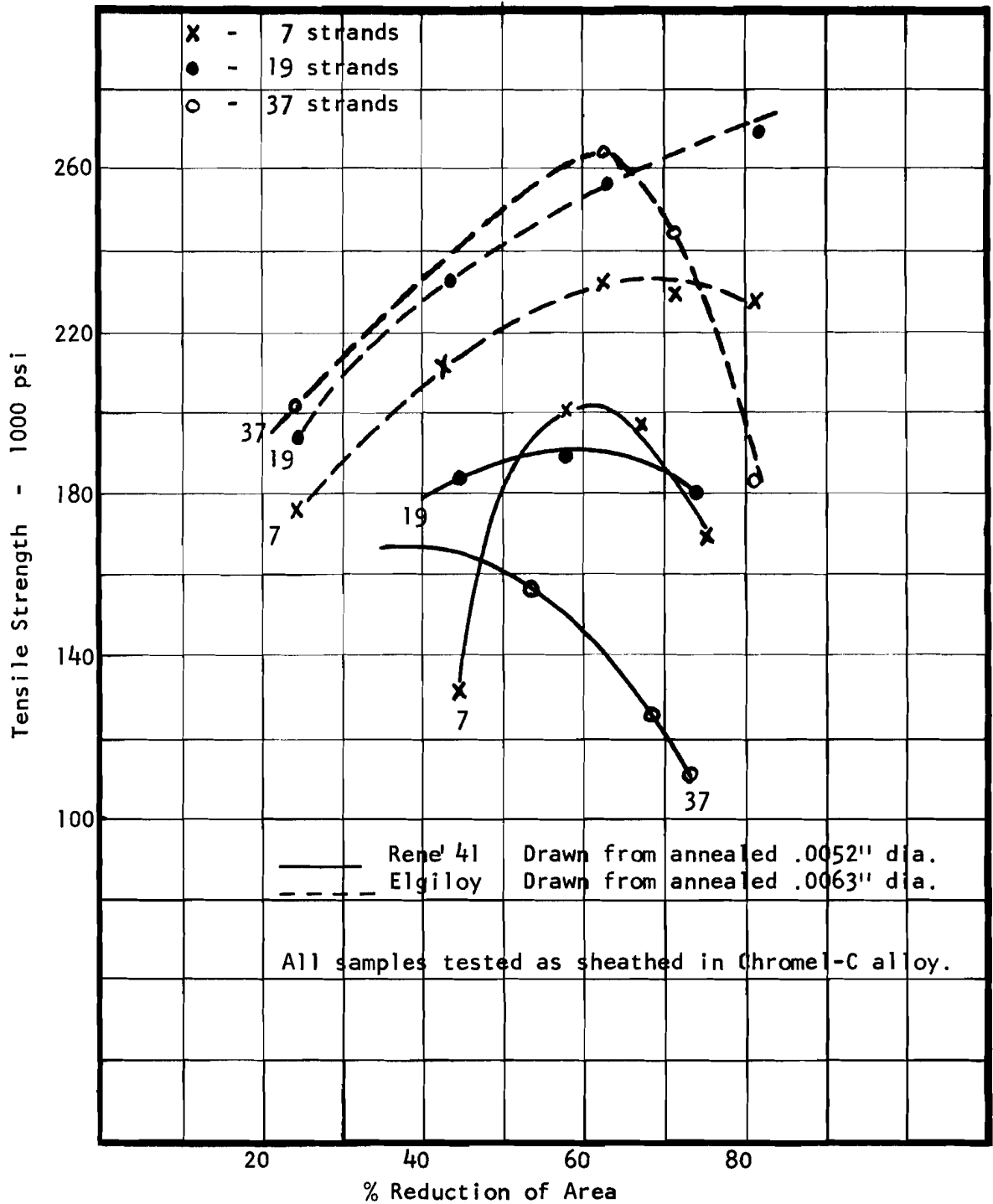


FIGURE 7 Effect of Cold Reduction on Tensile Strength of Elgiloy and Rene 41 Multifilament Yarns.

The high temperature tensile strength and elongation tests were made at temperatures of 1600°F., 1800°F. and 2000°F. in both air and argon atmospheres. The yarn diameters were all .0027 inch and were from the same lots as for the room temperature tests. The load measuring device was a Saxl Tension Meter calibrated for accuracy with gram weights. The strain was 0.5 inch per minute with an effective gage length of 3 inches and a maximum temperature gradient of  $\pm 20^\circ\text{F}$  at all test temperatures. The yarn was exposed to the test atmosphere for intervals of 1, 5 and 10 minutes prior to applying the load. Figure 8 illustrates typical high temperature tensile strengths of the yarn when held at the test temperature 5 minutes prior to load application. Results on a single solid wire of .0027 inch diameter are also illustrated for comparison. Tests on all configurations held at temperature 1, 5 and 10 minutes resulted in a slight increase in tensile strengths with increased holding time.

### CONCLUSIONS

The superalloys of Elgiloy and Hastelloy B were relatively easy to process to ultra-fine fibers and had the least die wear of all alloys. U-700 and Rene 41 respectively, had the highest room temperature tensile strength in the solution heat treated condition. Most of the alloys could be solution heat treated over a relatively wide range with only a moderate effect on physical properties. U-500 was temperature sensitive and had a sharp decrease in ductility when annealed between 1900°F. and 2050°F. The iron base alloy A-286 had very low tensile properties.

The encapsulation method of drawing fibers to finer sizes appears to be the most feasible but methods have to be found for removing the sheathed material in order to better evaluate the fiber properties. Increasing the number of filaments in yarn from 7 to 37 improved the high temperature properties to some degree, but because the effect of the sheathed construction was difficult to evaluate, the results were somewhat inconclusive. The Chromel-C sheath material did definitely improve the higher temperature properties of Rene 41 by protecting the fibers from oxidation. The results were not so apparent with Elgiloy.

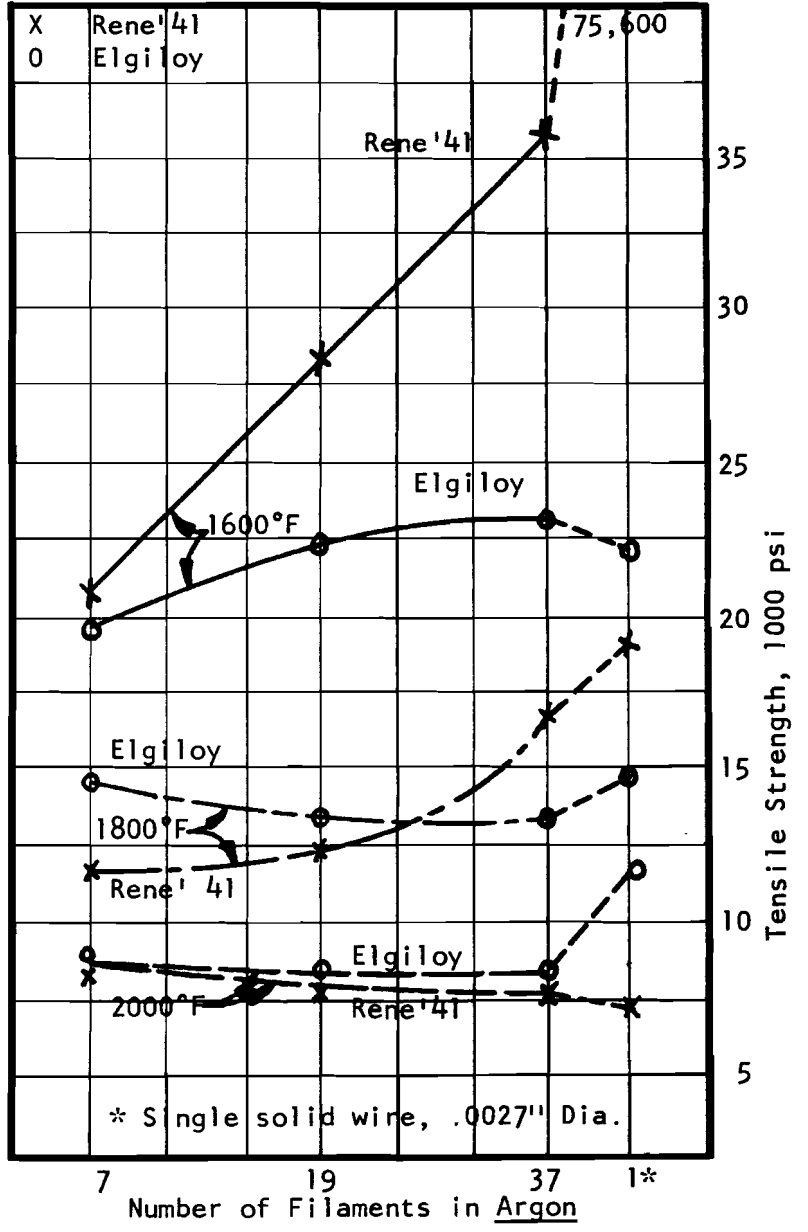
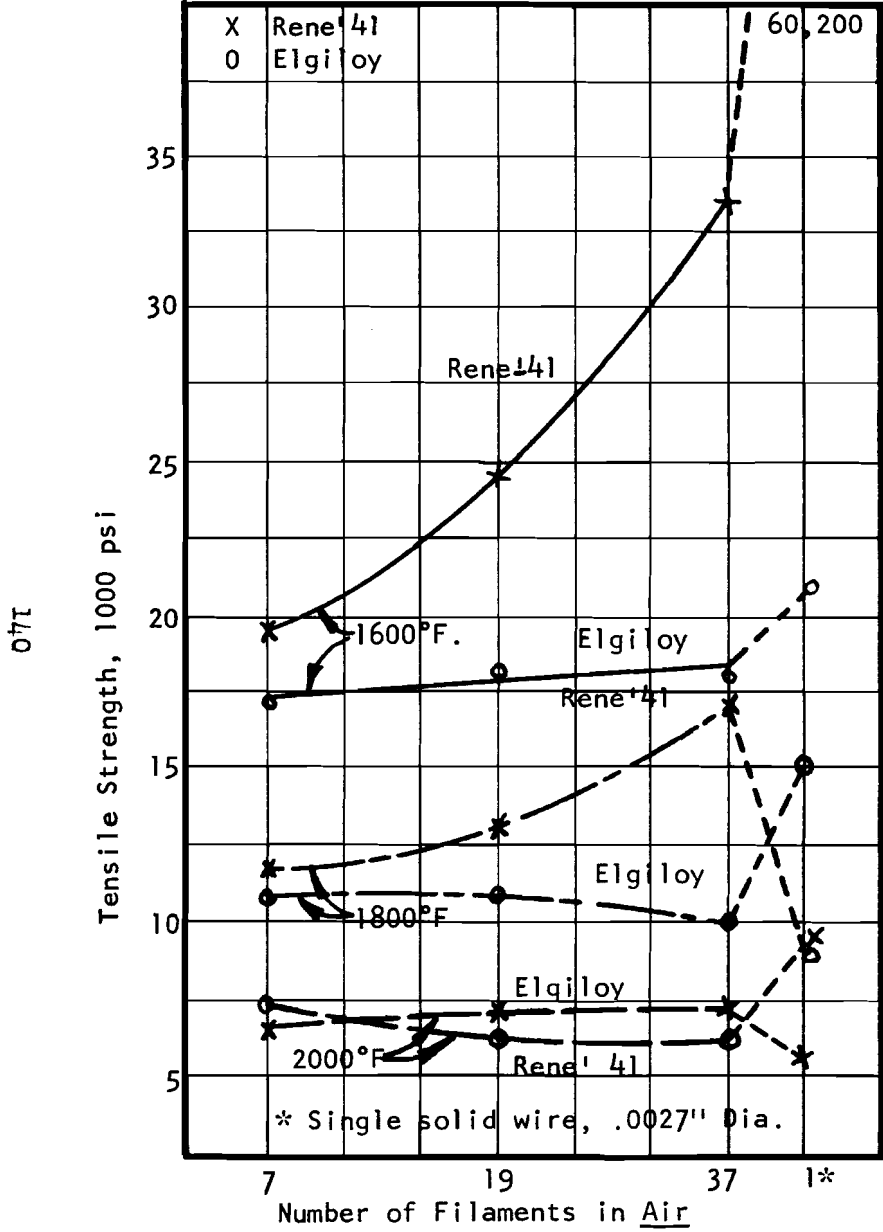


FIGURE 8. High Temperature Tensile Strength of Elgiloy and Rene' 41 Multifilament Yarn.

## REFERENCES

1. Coplan, M. J., Freeston, Jr., W. D. and Powers, Jr., D. H., "Flexible Low Porosity, Woven Metallic Materials", ASD TR 61-677, November, 1961.
2. Ross, J. H., "Prospects for Use of Fibrous Metals", Metal Progress, April 1962, p. 93.

