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ARMOUR RESEARCH FOUNDATION OF ILLINOIS INSTITUTE OF TECHNOLOGY




# DEVELOPMENT AND EVALUATION OF HIGH-TEMPERATURE TUNGSTEN ALLOYS

F. C. Holtz
R. J. Van Thyne

Summary Report
Contract AT(33-3)-4
Task I



AEC RESEARCH AND DEVELOPMENT REPORT

ARMOUR RESEARCH FOUNDATION
of
ILLINOIS INSTITUTE OF TECHNOLOGY
Technology Center
Chicago 16, Illinois

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## ABSTRACT

Tungsten-rich alloys, developed for use at temperatures up to 2000F, exhibit ductility, fabricability and joinability not found in commercially-available materials. An envelope type of microstructure was produced in compositions containing at least 90 wt% tungsten by liquid-phase sintering of cold-pressed powders in hydrogen. At room temperature the alloys could be rolled extensively, and tensile elongations up to 25% were noted. Strength properties of a W-Ni-Fe base were improved by small quaternary additions. The ultimate tensile strength of a 90W-4.8Ni-3.2Fe-2Ru alloy was 46,700 psi at 2000F, compared to 30,000 - 35,000 psi for unalloyed tungsten or W-Ni-Fe; the 100-hour stress-rupture strength at 1600F was 15,000 psi. Excellent joints were produced by spot welding and localized induction heating. The oxidation resistance of unprotected 90 wt% tungsten compositions was not significantly affected by alloying.

## TABLE OF CONTENTS

## LIST OF ILLUSTRATIONS

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# DEVELOPMENT AND EVALUATION OF HIGH-TEMPERATURE TUNGSTEN ALLOYS

## I. INTRODUCTION

This report summarizes the work performed during the interval October 1, 1958 to September 30, 1959 under Contract No. AT(33-3)-4, Task I, entitled "Development and Evaluation of High-Temperature Tungsten Alloys." The studies reported herein are a continuation of investigations initially conducted under Contract AF 33(616)-5218-Task No. 72023, for Wright Air Development Center, from May, 1957, through September, 1958.[1]

The primary objectives of this program are concerned with the development of high-tungsten alloys which are readily fabricable and are capable of operating at temperatures up to 2000F. Oxidation protection has been considered to be of secondary importance because of other current research efforts in this field. Unalloyed tungsten possesses adequate elevated-temperature strength but is not readily fabricated into massive and complex shapes. Commercial "Heavy Metal" alloys of the W-Ni-Cu type are not readily workable and exhibit a rapid loss of strength at temperatures below 2000F. These materials consist of slightly alloyed tungsten grains surrounded by a copper-nickel matrix which contains some dissolved tungsten. The powder metallurgy techniques used in the fabrication of these high-tungsten alloys are adaptable to the production of large parts utilizing commercially available equipment. Consequently, initial tungsten alloy development work was concerned with powder metallurgy methods, and the low-melting copper-nickel matrix of W-Ni-Cu alloys was replaced with nickel-iron and other higher-melting matrix phases.

Results of preliminary studies of the W-Ni-Fe system showed that compositions containing up to 97 wt% tungsten were ductile at room-temperature. Alloys at the 95wt% tungsten level could be cold-rolled to reductions of 60 per cent in thickness without edge cracking. Tensile strength values at 2000F for W-Ni-Fe materials ranged from 24,000 psi at 90 wt% tungsten to 34,000 psi at 97 wt% tungsten. Stress-rupture tests, however, showed a 100-hour life at a stress level of only 2500 psi at 2000F. Slight improvements in elevated temperature strength properties were produced by small quaternary additions of Cr, Mo, Nb and Ta.

During the current reporting interval, efforts were made to improve the high-temperature properties by further alloying in the W-Ni-Fe system, and by the use of other high-tungsten alloy systems. The factors which contributed to the excellent ductility in W-Ni-Fe materials were assessed and applied to other ternary and quaternary tungsten-base compositions. Consideration was given to various powder metallurgy fabrication techniques which could be adapted to the production of large and complex shapes. Joining methods were investigated, and limited efforts were devoted to oxidation protection. Thermal stability of the alloys under development was an important consideration. All materials were sintered in reducing atmospheres at temperatures slightly above the melting point of the matrix phase. Commercial, high-purity metal powders were used, and fabrication procedures utilized conventional processing equipment. Compositions were evaluated at room temperature and at temperatures up to 2000F by transverse-rupture and short-time tensile tests. The more promising alloys were evaluated on the basis of elevated-temperature, stress-rupture life, oxidation resistance, and thermal stability. Over one hundred compositions were studied; the data reported herein were obtained from test samples which were cold-pressed and hydrogen-sintered. Limited data from previous work have been included for comparison with current materials. Test results for the more promising compositions are discussed under sections covering the various alloy systems. Complete property data for all materials investigated are presented in the Appendix of this report.

## II. EXPERIMENTAL PROCEDURES

### A. Materials

Most of the alloys investigated under this program contained at least 90 wt% tungsten. High-purity commercial grade tungsten powders having average particle sizes ranging from 1 micron to over 4 microns were used. These hydrogen-reduced powders had the following range of impurities (wt%).

Fe, 0.001 to 0.007
Mo, 0.002 to 0.013
O, 0.03 to 0.10

Nonvolatile materials, 0.01 maximum.

Alloying additions to tungsten were made with high-purity commercial grade metal powders. Additions of Co, Cr, Fe, Mo, Ni, Pd, Ru, and Th were made in the form of elemental powders, minus 325 mesh, having purities in the range of 99.8 to 99.98 wt%. Some of the more reactive elements were added in the form of pre-alloyed powders in order to reduce oxidation during sintering and to provide lower melting points. The elements Cr, Ti, and Zr were added as commercially available, minus 325 mesh Ni-Cr, Ni-Ti, and Ni-Zr powders prepared by metal hydride processes. Aluminum and vanadium were added as non-consumable-electrode arc-melted master alloys which were comminuted through 325 mesh. Carbon additions were made in the form of lampblack or as tungsten carbide powders of about 5 microns average particle size.

### B. Preparation of Alloys

The materials produced under this program were prepared by powder metallurgy techniques. Commercial, high-purity metal powders were dry-blended by rolling in glass jars; no lubricants were used. Test specimens from which property data were obtained were compacted in steel dies at pressures in the range of 10 to 30 tsi. Sintering was carried out in a molybdenum-wound, alundum-tube furnace using a purified hydrogen atmosphere. The samples were packed in granular aluminum oxide within a molybdenum sintering boat; an optical pyrometer was used to measure the temperature of molybdenum targets placed in the sintering boat.

Sintering temperatures ranged from 2640F to 2910F for 30 minutes to 2 hours; heating and cooling rates of about 180F per hour were used at temperatures above 2140F.

Some compositions contained elements whose oxides are not reduced by hydrogen; these included Al, Th, Ti, V, and Zr. Although the hydrogen sintering atmosphere was passed through a catalytic converter and an activated alumina dryer, small quantities of oxygen were present in the furnace. The small particle size of the reactive materials also contributed to oxidation during sintering of materials containing the above elements. Sintering in vacuum or in an inert atmosphere may have resulted in improved properties of these alloys. However, the time available for such heat-treatments was limited, and a great majority of the alloys studied were sintered satisfactorily in hydrogen.

Several processing techniques other than cold-pressing followed by hydrogen sintering were used. These methods included hot-pressing in graphite dies and the sintering of loosely compacted and slip-cast powders. More detailed descriptions of these techniques are included in subsequent sections of this report.

## C. Metallographic Techniques

Specimens for metallographic examination were cut from sintered compacts and mounted in phenolic (Bakelite) resin. Rough grinding was accomplished on silicon carbide papers from 120 to 600 grit. The first polishing operation used a 9 micron diamond abrasive in kerosene on an A. Buehler "Metcloth" wheel. A second polishing operation utilized 1 micron diamond paste on a "Microcloth" wheel. Final polishing was accomplished on a Microcloth wheel using Linde B abrasive (0.3 to 0.8 micron alumina).

Unetched samples were found to be satisfactory for determining grain size, porosity, and volume and distribution of the matrix phase. The tungsten-rich grains were etched with a solution consisting of equal volumes of ammonium hydroxide, 3% hydrogen peroxide, and water; etching was accomplished by immersion or swabbing at room temperature. The matrix phase, generally containing substantial quantities of nickel, was etched with

equal parts of nitric acid, hydrofluoric acid, and water. Another etchant employed for some matrix phase structures was composed of 20 parts $HNO_3$ 20 parts HF, and 60 parts of glycerine. Both matrix-phase etchants were used at room temperature.

D. Test Procedures

Initial screening of sintered compacts utilized a transverse-rupture test at room temperature. Specimens measuring approximately 1/8 x 3/16 x 1 3/4 in. were supported on parallel cylindrical pins having a span of 1 1/4 in. A load was centrally applied and transverse-rupture strength values were calculated according to the standard formula for rectangular beam specimens:

$$S = \frac{3PL}{2wt^2}$$

where:

S = transverse-rupture strength, psi
P = applied load, lb.
L = length between supports, in.
w = width of sample, in.
t = thickness of sample, in.

These tests were conducted on a Hounsfield "Tensometer" and the stress-strain curves obtained were also used for yield-point determinations; the above formula was used. Some materials exhibited large deflections under a load, and transverse-rupture strength values for these ductile materials are only approximate. Room-temperature tensile tests were conducted on the more promising alloys; the test specimens measured approximately 1/8 in. x 3/16 in. with a 1 inch gage length. Good correlation was obtained between transverse-rupture strength values and ultimate tensile strength results for the same materials; the tensile strength values were about one half of the transverse-rupture strength values.

Other room-temperature properties investigated included hardness, bend ductility, and workability. Vickers 10kg hardness measurements were made on polished metallographic specimens. Microhardness readings, using 25 to 100 gram loads, were taken on individual tungsten-rich grains and also in areas of the matrix phase. A standard 180° bend test was used on as-sintered and also on cold-rolled samples. Workability

was evaluated by rolling 1/8 x 5/8 x 1 3/4 in. specimens at room temperature.

Materials which possessed satisfactory structures and adequate room-temperature properties were further evaluated at elevated temperatures. Transverse-rupture, tensile, and stress-rupture tests were conducted at temperatures up to 2000F under inert atmospheres. Test samples had cross-sections measuring about 0.1 x 0.2 in. Oxidation tests were performed at 2000F in a horizontal alundum tube. The oxide films which formed in air were somewhat tenacious for most alloys, and the specimens were held at temperature for 15 or 30 minutes. Test results are expressed in weight gain/$cm^2$/hr.

## III. DISCUSSION OF RESULTS

Efforts during this reporting interval have been devoted to improving the workability, fabricability, and elevated temperature properties of tungsten-base alloys studied under a previous program. Over 100 new compositions were evaluated at room temperature; of these, about 40 were considered promising and were further tested at elevated temperatures. Investigations of W-Ni-Fe compositions were continued in order to determine optimum compositions in the system. The fabricability and elevated temperature stability of these alloys were studied. A number of quaternary additions to the W-Ni-Fe base were made in order to improve high-temperature strength and stability. In addition, other tungsten-base ternary systems including W-Ni-Co, W-Ni-Cr, and W-Ni-Ru were evaluated. The scope of alloys studied is outlined below:

Binary Alloys:
Tungsten with Ni and Pd

Ternary Alloys
Tungsten-nickel with Fe, Co, Cr, Mo, and Ru.
Tungsten-iron with Co, Cr, and Mo.
Tungsten-cobalt with Cr and Ru.

Quaternary alloys:
Tungsten-nickel-iron with Al, C, Co, Cr, Mo, Pd, Ru, Th, Ti, V, and Zr.
Tungsten-nickel-cobalt with Cr, Mo, and Ru.

More complex alloys:
Tungsten-nickel-iron with Co-Cr, Co-Ru, Cr-Mo Mo-Ti, Mo-V, and Mo-Zr.

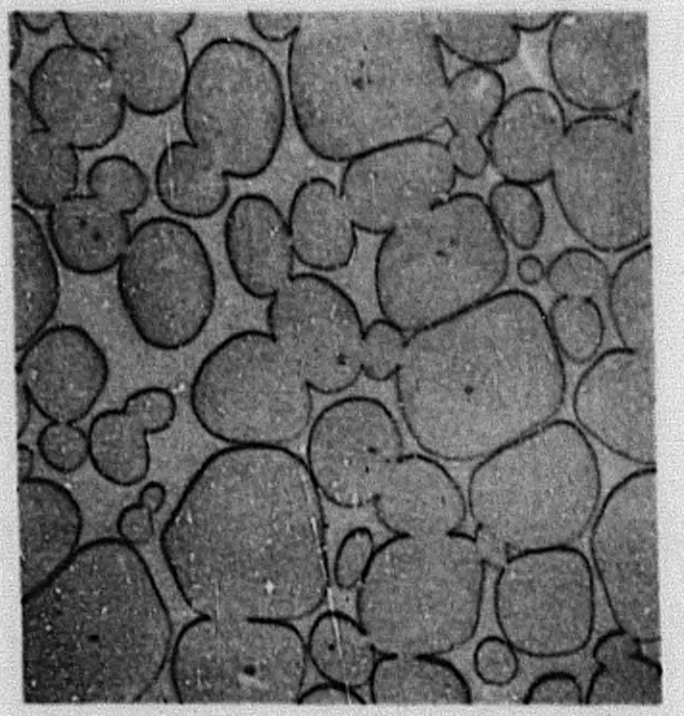
Some of the alloy systems listed above were represented by a single blend, whereas the more promising systems included ten or more variations in composition. Several sintering treatments were required in order to establish optimum heat treatments for many of the alloys. Some systems are in the initial stages of study; hence the compositions and thermal treatments have not been optimized.

A. W-Ni-Fe Alloys

Initial tungsten-base alloy studies were devoted to the W-Ni-Fe system. Compositions ranged from 90 to 99.5 wt% tungsten; optimum nickel-to-iron ratios were found to be about 3:2 at most tungsten levels. Sintering temperatures were 2680F for the lower-tungsten compositions, and up to 2840F for alloys above 98 wt% tungsten. Microstructures were characterized by rounded tungsten-rich grains in a Ni-Fe-W solid solution matrix for materials containing less than 98 wt% tungsten. Figure 1 illustrates a typical structure in a 90W-6Ni-4Fe* alloy. The matrix phase of this composition was found to contain 18 wt% tungsten; the tungsten-rich grains contained about 0.1 wt% nickel and less than 0.3 wt% iron. Electron-probe microanalysis techniques were used to further these determinations. Alloys at 98 to 99.5 wt% tungsten levels were characterized by greatly reduced areas of matrix, and the tungsten grains were generally equiaxed as shown in Figure 2 for a 99W-0.6Ni-0.4Fe specimen. The matrix phase appears to be discontinuous and the tungsten grains are very coarse. The density of this specimen was 19.02 g/cc. a value which approximates the theoretical density for this composition; dense material was obtained in all sintered W-Ni-Fe material containing up to 99.5 wt% tungsten.

Room-temperature properties of W-Ni-Fe alloys are markedly influenced by the tungsten contents. Results of transverse-rupture tests for these materials are presented in Figure 3. Strength levels decreased rapidly above 97 wt% tungsten, and the deflection values decreased uniformly with increasing tungsten contents above 90 wt%. Materials at the 90 wt% tungsten level passed a 1.2 t bend, using a standard 180° bend test. The 90W-6Ni-4Fe alloy had an ultimate tensile strength of about 130,000 psi with 20 to 25 per cent elongation; rapid cooling from the sintering temperature did not significantly affect these values. Materials containing

* Compositions are reported in weight per cent.



Neg. No. 19107 X250

Fig. 1
Microstructure of a 90W-6Ni-4Fe alloy. This material was sintered for 1 1/4 hr at 2680F, and consists of rounded tungsten-rich grains in a W-Ni-Fe solid solution matrix. Etched and repolished.

Neg. No. 18572 X250

Fig. 2
Microstructure of a 99W-0.6Ni-0.4Fe alloy. Sintering was conducted at 2840F for 1 1/2 hr; the tungsten grains are generally equiaxed, and the light-etching matrix areas are extremely small.
Etchant: $H_2O_2 + NH_4 + H_2O$.

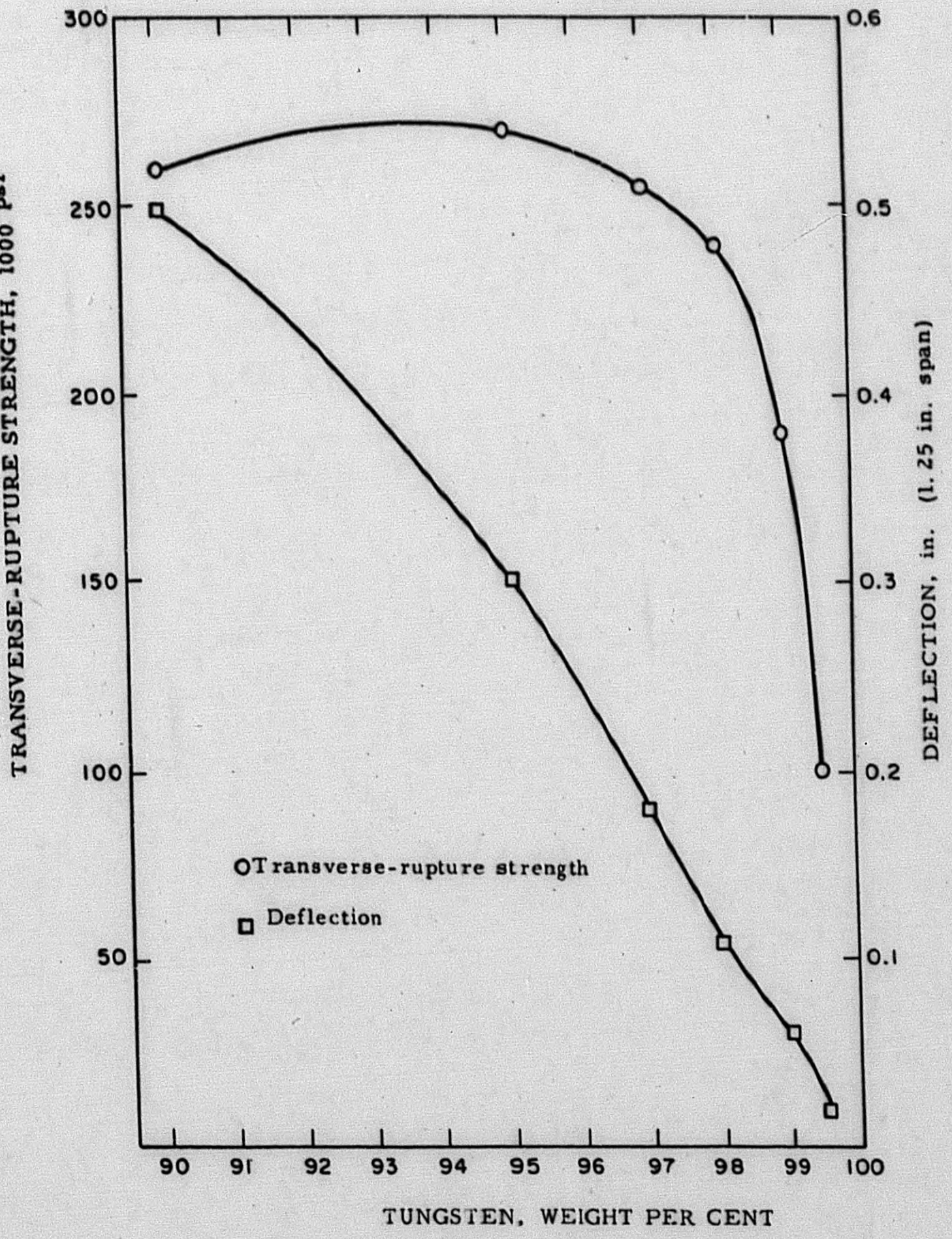



FIG. 3 - ROOM-TEMPERATURE TRANSVERSE-RUPTURE TEST DATA FOR W-Ni-Fe ALLOYS.

up to 95 wt% tungsten could be cold rolled to reductions in thickness of 85 per cent with only slight edge cracking. Ductility was reduced at higher tungsten levels: the 97W-1.8Ni-1.2Fe alloy could be reduced 35 per cent in thickness before edge-cracking developed.

Initial impact tests using standard Charpy "V" notched specimens showed the relatively low values of 3 to 3.5 ft-lb for the 90W-6Ni-4Fe material. Microstructures of the specimens exhibited some inclusions, possibly due to contamination during blending. Good ductility was observed in subzero temperature tensile tests. However, the low impact values indicate the possibility of a ductile-to-brittle transition on the basis of notch-sensitivity. These results suggest that additional impact tests and also notched-tensile tests at various strain rates be conducted on these alloys.

The W-Ni-Fe system is characterized by a large single-phase region in the nickel-rich corner, as shown in Figure 4. The matrix composition in a 90W-6Ni-4Fe alloy is approximately 49 wt% Ni - 33 wt% Fe - 18 wt% W. Intermediate phases exist in both the Fe-W and the Ni-W systems, although a peritectoid reaction at 1780F in the Ni-W system is very sluggish. Consideration has been given to the effects of long-time annealing treatments on the properties of the matrix phase in W-Ni-Fe compositions. Samples of a 90W-6Ni-4Fe alloy were annealed for 200 hours at temperatures ranging from 1450F to 1850F. Metallographic examination disclosed the presence of an intermediate phase in some matrix areas of specimens annealed below 1750F. Figure 5 illustrates the structure of a sample annealed for 200 hours at 1550F; the precipitate occurs throughout many of the matrix areas. Hardness values were similar to the as-sintered hardness. Annealing a sample for 100 hours at 1500F resulted in a slight increase in ultimate tensile strength from 130,000 psi for as-sintered material to 142,000 psi for the annealed specimen; room-temperature elongation was unchanged at 23%. The presence of an intermediate phase in the microstructures, however, should require further studies of thermal stability. Long-time heat treatment at temperatures below 1450F may possibly alter the room-temperature properties in W-Ni-Fe alloys.

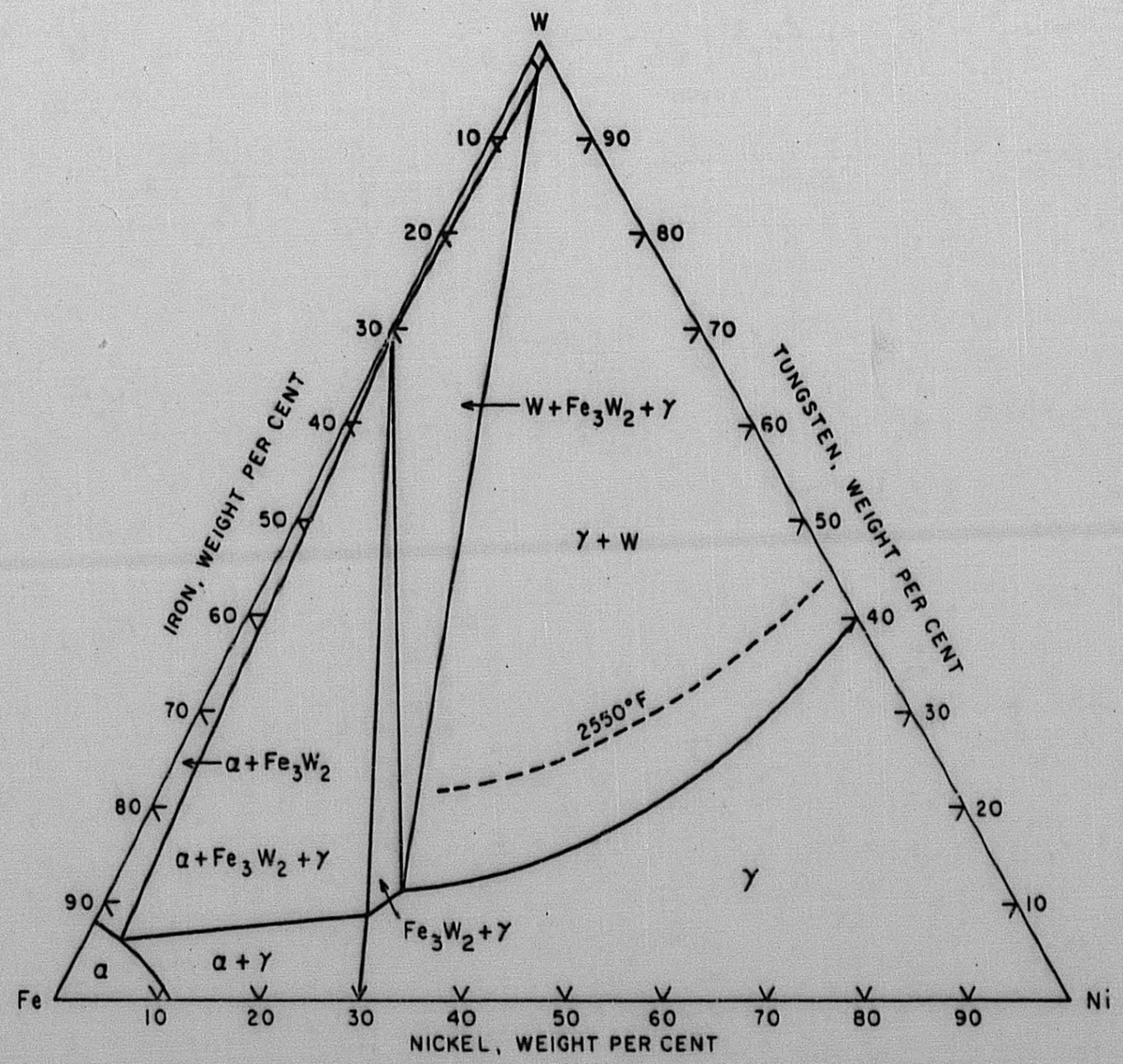



FIG. 4 - ROOM TEMPERATURE SECTION OF THE IRON-TUNGSTEN-NICKEL SYSTEM, (WINKLER AND VOGEL).

Neg. No. 19106 X500

Fig. 5
Microstructure of a 90W-6Ni-4Fe alloy annealed for 200 hr at 1550F. The annealing treatment produced an intermediate phase in some matrix areas.
Etchant: $HNO_3$ + HF + Glycerine.

Elevated temperature tensile properties of W-Ni-Fe alloys have been evaluated for compositions containing from 90 to 99 wt% tungsten. Figure 6 presents results of tensile tests conducted at 2000F. Tensile strength values rise to a maximum of 34,000 psi at 97 wt% tungsten, and decrease rapidly above 98 wt% tungsten. Elongation values fall slightly from 90 to 95 wt% tungsten, and a more rapid decrease is noted above 95 wt% tungsten. Stress-rupture tests of W-Ni-Fe alloys at 2000F showed a 100-hour life at a stress level of about 2,500 psi; this value was found for alloys containing 90, 95, and 97 wt% tungsten. The 100-hour rupture life at 1600F was at a stress level of about 10,000 psi for a 90W-6Ni-4Fe composition. The tensile and stress-rupture data are for materials in the as-sintered condition. Cold-working a 90W-6Ni-4Fe material produced an increase in short-time tensile strength from 26,000 psi to 32,000 psi at 2000F. However, stress-rupture tests on the cold-rolled specimens showed values similar to those reported for the as-sintered samples. Recrystallization of the worked materials was initiated after relatively short times under stress at elevated temperature, and the increase in strength levels resulting from rolling was lost.

Alloys of the W-Ni-Fe system are characterized by good room-temperature strength and ductility, and adequate tensile strength at 2000F. The relatively low stress-rupture levels at 2000F indicated the necessity for further alloy development in order to improve the long-time elevated-temperature properties. A number of quaternary additions to W-Ni-Fe were evaluated, and several other ternary and quaternary tungsten-base alloys systems were studied. The results of these investigations are discussed in the sections below.

## B. W-Ni-Fe-X Alloys

Quaternary additions to the W-Ni-Fe base were made for the purpose of improving elevated-temperature strength properties without undue loss of room-temperature ductility. The factors which contribute to the excellent ductility of the W-Ni-Fe materials include a strong bond between the matrix and the tungsten grains, the absence of intermetallic compounds, the matching of flow stresses of the tungsten grains and the matrix envelope, and limited solubility of the matrix in tungsten unless this can be matched by

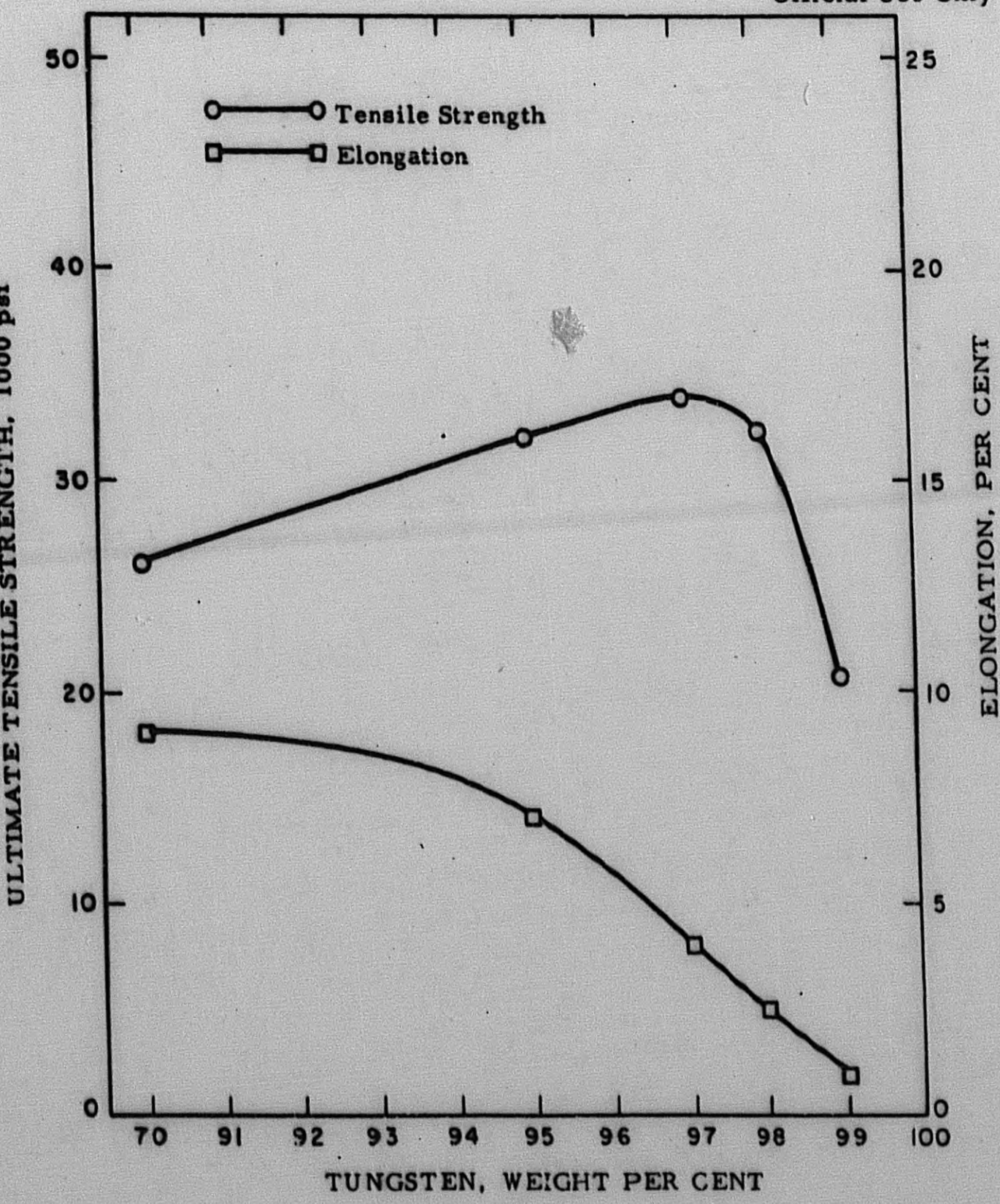



FIG. 6 - TENSILE TEST DATA FOR W-Ni-Fe ALLOYS AT 2000F.

a corresponding strengthening of the matrix phase. Elevated-temperature strength of these alloys may be improved by increasing the melting point of the matrix, by solid-solution strengthening of both phases, or by dispersion-strengthening of the matrix. A number of alloying additions were made on the basis of one or more of these criteria; in some cases elevated-temperature properties were improved at the expense of room-temperature workability.

1. W-Ni-Fe-Al Alloys

Aluminum was added to a 90W-Ni-Fe base at levels of 0.25 and 0.5 wt%. The sintered compacts contained numerous fine inclusions, probably the result of oxidation during sintering. Room-temperature strength and ductility of the W-Ni-Fe base were reduced by the aluminum additions; the transverse-rupture strength at 2000F was also decreased. Property data for these alloys will be presented under a subsequent section where test results for the various quaternary additions will be compared.

2. W-Ni-Fe-C Alloys

Several compositions containing 0.1 to 0.5 wt% carbon were prepared. The additions were made in the form of lampblack and also as WC. Test results were extremely variable, although in most cases the ductility of the W-Ni-Fe base was reduced. Chemical analysis of the compacts showed that most of the carbon was lost during sintering, probably due to traces of oxygen in the hydrogen-sintering atmosphere. Small quantities of carbon in solution in the matrix should result in higher strength, and vacuum or inert-atmosphere sintering may be required to avoid the loss of this element during heat treatment.

3. W-Ni-Fe-Co Alloys

Cobalt was added to the W-Ni-Fe base in amounts up to 5 wt%; tungsten levels varied from 90 to 97 wt%. Alloys at the 90 wt% tungsten level containing up to 2.5 wt% cobalt were stronger than the 90W-Ni-Fe base; the excellent ductility was maintained. The microstructure of a 90W-4.5Ni-3Fe-2.5Co sample consisted of rounded, tungsten-rich grains in a solid solution matrix. Increasing cobalt contents resulted in the formation

of a brittle intermediate phase around the tungsten grains. Figure 7 illustrates the three-phase structure in a 90W-4Ni-2.5Fe-3.5Co composition; the intermediate phase probably has the composition $W_6Co_7$, and is formed peritectically from the melt in W-Co alloys. The material shown in Figure 7 was weak and brittle at room temperature.

Figure 8 presents room-temperature transverse-rupture test data for W-Ni-Fe-Co alloys at tungsten levels ranging from 90 to 97 wt%. The cobalt contents were adjusted so that maximum strength was obtained at each tungsten level. Strength values reach a maximum at about 93 wt% tungsten, and these levels are somewhat higher than for W-Ni-Fe alloys (refer to Figure 3). Ductility, as indicated by deflection of test specimens under load, decreases with increasing tungsten contents beyond 93 wt%. The deflection values indicate good room-temperature workability up to at least 95 wt% tungsten.

Elevated-temperature strength properties of W-Ni-Fe-Co alloys are shown in Figure 9. Transverse-rupture strength values increase with increasing tungsten contents up to at least 97 wt% tungsten; the strength levels are slightly higher than those of the W-Ni-Fe ternary compositions. In addition, deflection values of the alloys containing cobalt are somewhat greater than were observed in the ternary base at similar tungsten levels.

The thermal stability of cold-rolled 90W-4.5Ni-3Fe-2.5Co material has been investigated by annealing the worked specimens for 2 hours at temperatures between 500F and 1900F. A rise in hardness occurred above 500F and reached a maximum at 1100F; the hardness curve decreased above this temperature. Hardness values at 1900F were considerably above the as-sintered value. Microstructures of these specimens show the presence of a two-phase matrix upon annealing near 1100F; partial recrystallization of the rolled structure occurred upon annealing at 1900F. These results indicate that the 2.5 wt% cobalt addition resulted in a lower recrystallization temperature than was found in the W-Ni-Fe base, and that a brittle intermediate phase is precipitated after relatively short annealing intervals.

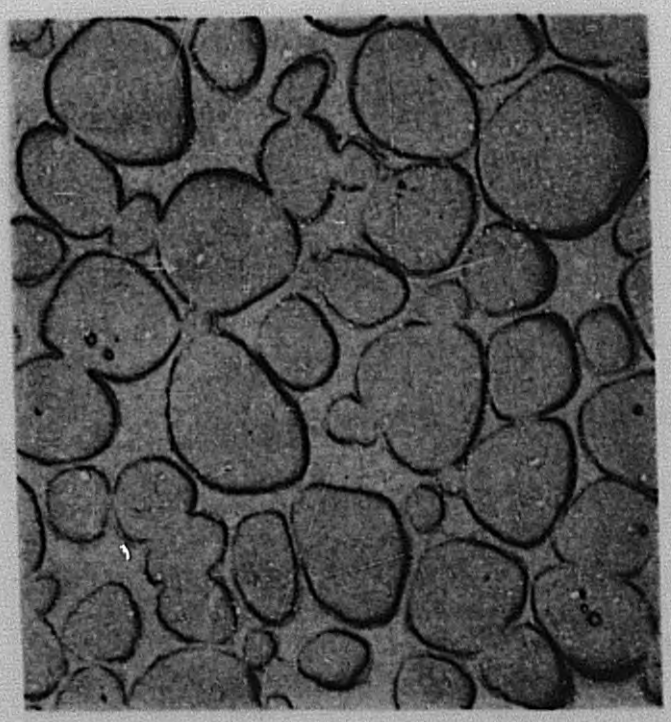


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Fig. 7

Microstructure of a 90W-4.5Ni-2.5Fe-3Co alloy. This material, sintered for 1 hr at 2750F, contains a brittle cobalt-tungsten intermediate phase in some matrix areas and around the tungsten grains. Unetched.

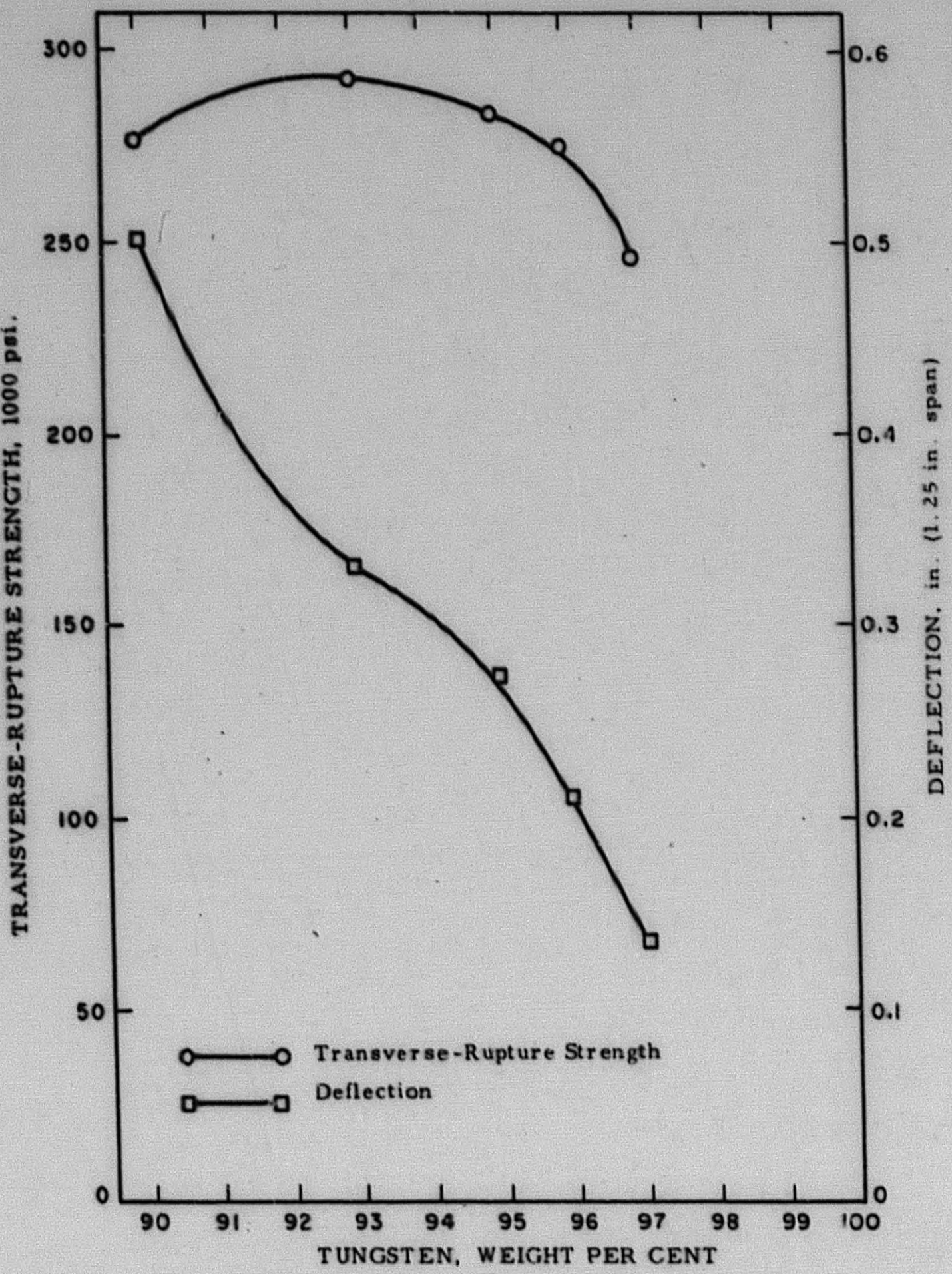



FIG. 8 - ROOM-TEMPERATURE TRANSVERSE-RUPTURE TEST DATA FOR W-Ni-Fe-Co ALLOYS

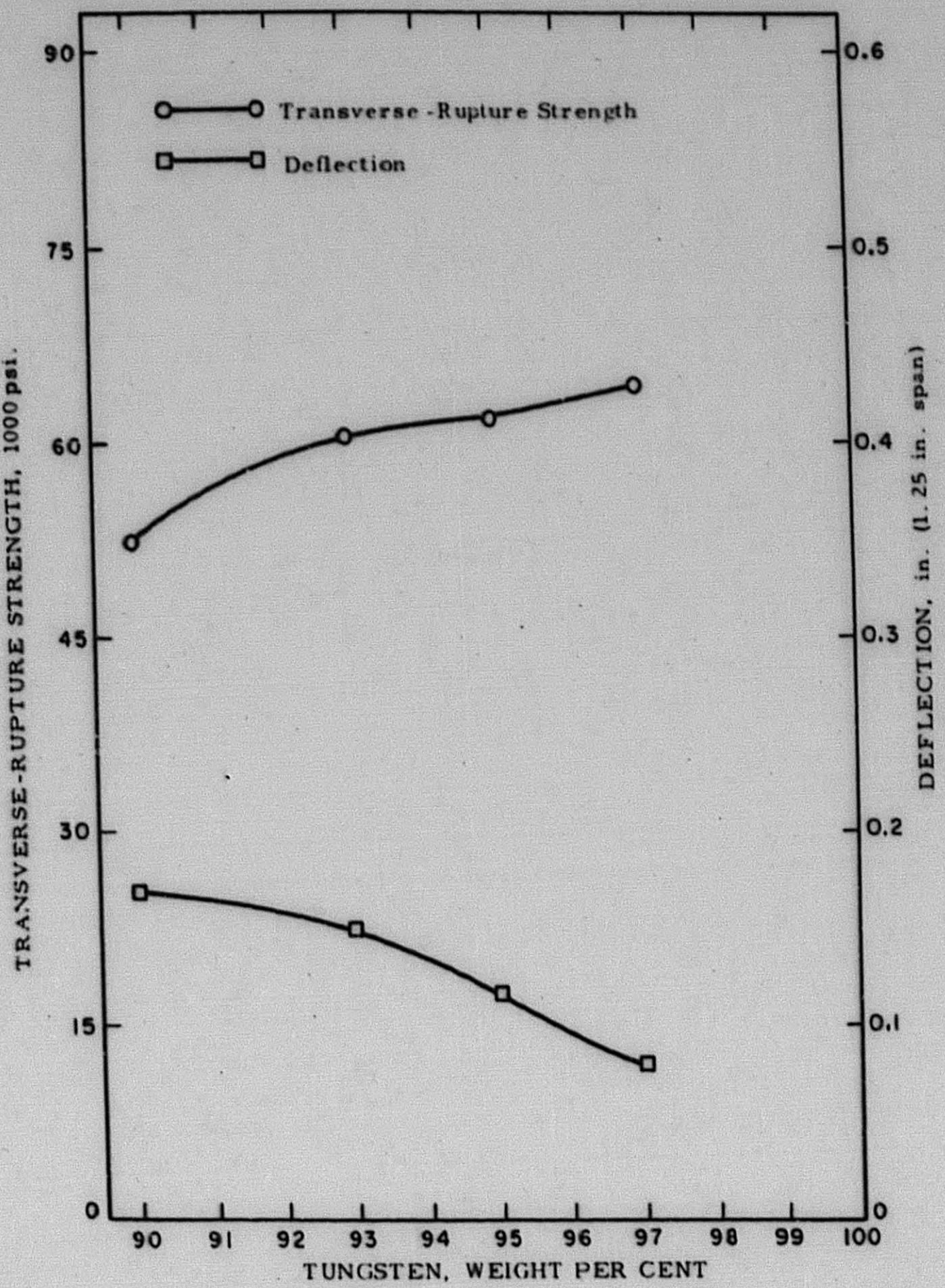



FIG 9 - TRANSVERSE-RUPTURE TEST DATA FOR W-Ni-Fe-Co ALLOYS AT 2000 °F

4. W-Ni-Fe-Cr Alloys

The element chromium was added to the W-Ni-Fe base in amounts ranging from 1 to 5 wt%; tungsten levels varied from 90 to 95 wt%. Room-temperature property measurements showed that chromium additions resulted in higher hardness, moderately greater strength, and lower ductility than were found in the W-Ni-Fe base. These properties may be attributed to solid-solution hardening effects, as tungsten and chromium have extensive solid solubility. The sintered compacts were finer-grained, and higher sintering temperatures were required to produce optimum properties. Microstructures resembled those of W-Ni-Fe alloys. Ductility was moderate in a 90W-4.5Ni-3Fe-2.5Cr composition and a 90W-3Ni-2Fe-5Cr material was not considered workable at room temperature.

Chromium additions to the W-Ni-Fe base usually resulted in decreased strength and ductility at 2000F. The reasons for this loss in strength are not understood, although the melting point of 2440F in the Ni-Cr system is about 180F lower than any in the Fe-Ni, Fe-W, and Ni-W binary systems.

5. W-Ni-Fe-Mo Alloys

Molybdenum, completely soluble in tungsten in the solid state, was added to the W-Ni-Fe base at levels up to 5 wt%. Microstructures of the sintered compacts resembled those of W-Ni-Fe alloys. Property data at room temperature show that increasing amounts of molybdenum result in higher strength and moderately reduced ductility. The addition of molybdenum produced slightly greater strength than was obtained by increasing the tungsten level an equivalent amount in the ternary base.

Elevated-temperature tests on W-Ni-Fe-Mo alloys show that molybdenum additions resulted in increased tensile strength. The effect of adding molybdenum was similar to that produced by the addition of an equal amount of tungsten to the W-Ni-Fe base. Stress-rupture test results at 2000F on 90W-Ni-Fe-Mo compositions containing 2.5 and 5 wt% molybdenum were slightly higher than values for W-Ni-Fe materials. The 100-hour rupture life was at a stress level of about 3,000psi for both quaternary alloys.

The elements Cr, Ti, V, and Zr were added to a 90W-Ni-Fe-Mo base at the 2 wt% level. Chromium additions produced a slight increase in strength and hardness at room-temperature; ductility was greatly decreased. Additions of titanium, vanadium, and zirconium resulted in decreased strength and ductility, and considerable amounts of porosity and unreduced oxides were present in the sintered compacts.

6. W-Ni-Fe-Pd Alloys

The solubility of palladium in tungsten is probably very low; although complete phase relationships are not available. Initial studies were conducted on a 90W-4.8Ni-3.2Fe-2Pd composition. The resulting microstructure was similar to those of the W-Ni-Fe system. Room-temperature property data show that the good ductility of the W-Ni-Fe base was very slightly reduced; the transverse-rupture strength was also slightly lower than that of the ternary base. Palladium absorbs large quantities of hydrogen at the temperatures involved in the sintering of these alloys. Some improvements in properties may be expected through the use of vacuum or inert-atmosphere sintering.

7. W-Ni-Fe-Ru Alloys

Ruthenium was added to a 90W-Ni-Fe base in amounts of 1, 1.5, 2, and 2.5 wt%: the nickel-iron ratio was maintained at 3:2. No other quaternary addition under study had such a pronounced effect on the room- and elevated-temperature strength properties. The results of room-temperature transverse-rupture tests for compositions containing ruthenium are presented in Figure 10. Strength values rise and deflection decreases with increasing ruthenium contents. These deflection values indicate adequate workability to at least 2 wt% ruthenium. The addition of 1 wt% ruthenium resulted in greater strength than the addition of 5 wt% of chromium, molybdenum, or tungsten to the 90W-Ni-Fe base. The 90W-4.8Ni-3.2Fe-2Ru material had an ultimate tensile strength of 156,000 psi with 8.5% elongation at room temperature. Microstructures of W-Ni-Fe-Ru alloys were similar to those of the W-Ni-Fe system except that the grain size was much smaller. Figure 11 illustrates the structure of the 90W-4.8Ni-3.2Fe-2Ru composition sintered for 1 1/4 hours at 2840F. The grain size may be compared with

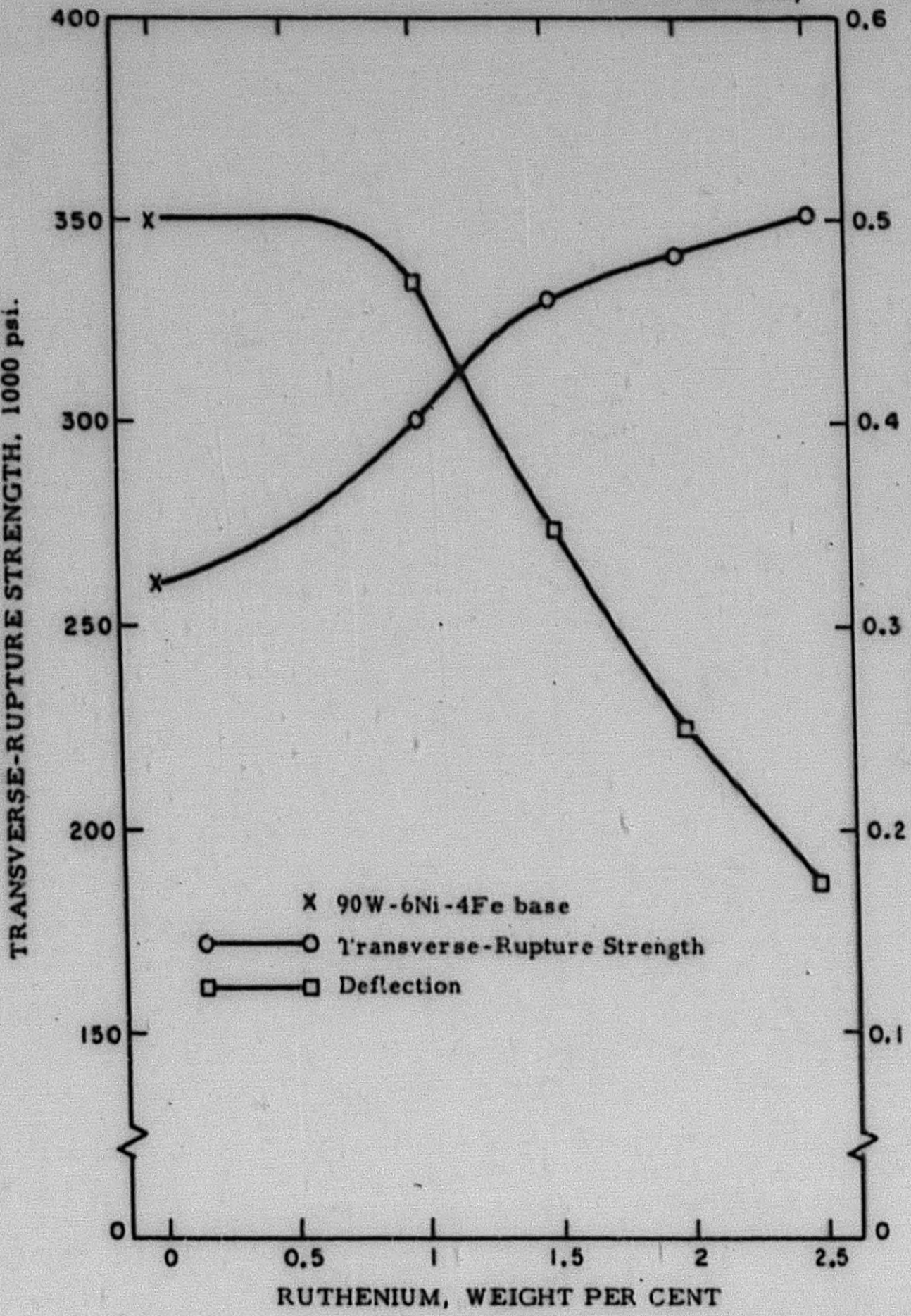



FIG. 10 - ROOM-TEMPERATURE TRANSVERSE-RUPTURE TEST DATA FOR 90W-Ni-Fe-Ru ALLOYS.

Neg. No. 19089 X250

Fig. 11
Microstructure of a 90W-4.8Ni-3.2Fe-2Ru alloy. The sintering treatment of 1 1/4 hr at 2840F produced a very fine grain size. Small porous areas are present in the matrix.
Etched and repolished.

that shown in Figure 1 for the 90W-6Ni-4Fe material which was sintered at a lower temperature; the effect of ruthenium in retarding grain growth was also noted at the 1 wt% level. Some fine porosity was present in the alloys containing ruthenium. This porosity may have been caused by the absorption and subsequent release of hydrogen during sintering. Vacuum or inert-atmosphere sintering may be required to produce clean microstructures; this may also result in an increase in tensile properties.

Elevated-temperature transverse-rupture, tensile, and stress-rupture tests show that alloys containing ruthenium are markedly superior to the 90W-Ni-Fe base. The 90W-4.8Ni-3.2Fe-2Ru material had ultimate tensile strength values of 73,300 psi at 1600F and 46,700 psi at 2000F; elongation values were about 4%. Unalloyed tungsten, by comparison, had a tensile strength of about 35,000 psi at 2000F.[2] Initial stress-rupture tests at 1600F for this alloy show a 100-hour rupture life at a stress level of 15,000 psi, some 5000 psi higher than was measured for the 90W-6Ni-4Fe material.

Cobalt additions to the W-Ni-Fe-Ru base resulted in lower strength and ductility at room temperature. However, the transverse-rupture strength of a 90W-4.5Ni-2Fe-2Ru-1.5Co alloy at 2000F was about 81,000 psi, higher than for any other composition tested under this program.

These initial property data for W-Ni-Fe-Ru alloys demonstrate the superiority of this system, although only a few compositions at one tungsten level have been studied. Further improvements may be expected by variations in tungsten and ruthenium contents and also by modifications of the nickel-to-iron ratios. Sintering in inert atmospheres or under vacuum may also result in stronger materials by eliminating the fine porosity found in hydrogen-sintered specimens.

8. W-Ni-Fe-Th Alloys

Although complete W-Th phase diagrams are not available, the solubility of thorium in tungsten is probably well below 1 wt%. One composition containing 1 wt% thorium was investigated at the 90 wt% tungsten level. Some oxidation of the thorium occurred during sintering, and the microstructures showed the presence of thorium particles which

did not dissolve in the matrix. Room-temperature tests of this 90W-5.4Ni-3.6Fe-1Th alloy show a transverse-rupture strength of 245,000 psi, slightly lower than that of the 90W-Ni-Fe base; deflection values were considerably lower in the material containing thorium. The decreased strength and ductility may be attributed to the presence of unreduced oxides in the sintered compacts. This effect has been noted in earlier studies of tungsten-base alloys containing Al, Nb, Ta, Ti, and other elements whose oxides are not reduced by hydrogen. Improved microstructures may be produced by vacuum or inert-atmosphere sintering of such alloys.

9. W-Ni-Fe-Ti Alloys

Titanium was added to a 90W-Ni-Fe base at levels of 0.5 and 2 wt%. The sintered compacts of both compositions exhibited some unreduced oxides and the alloy containing 2 wt% titanium was extremely porous. Room-temperature strength values of the 90W-5.7Ni-3.8Fe-0.5Ti material were slightly lower than the 90W-Ni-Fe base; a moderate decrease in ductility was noted. Elevated-temperature properties of these compositions were not determined because of the presence of oxides and porosity in the specimens.

10. W-Ni-Fe-V Alloys

Two compositions containing vanadium were studied; tungsten levels were maintained at 90 wt%, with vanadium additions of 0.5 and 2 wt%. The sintered compacts contained some porous areas which may have been caused by evolution of a volatile vanadium oxide when the matrix phase was liquid. Transverse-rupture strength values at room temperature were lower than for the 90W-Ni-Fe base, and the ductility was greatly reduced. Property data at elevated temperature were not obtained because of porosity in the test samples.

11. W-Ni-Fe-Zr Alloys

Zirconium was added to the 90W-Ni-Fe base at levels of 1 and 2.5 wt%. The microstructures contained excessive amounts of porosity and unreduced oxides; property data for these materials are not reported. Compositions containing zirconium will also require oxygen-free atmospheres during sintering.

## 12. W-Ni-Fe-X Comparative Data

The effects of various quaternary additions to a 90W-Ni-Fe base on room-temperature transverse-rupture strength are illustrated in Figure 12; a curve for increasing tungsten contents in the ternary alloy is also included. Ruthenium additions produced the greatest increase in strength. Somewhat smaller increases were noted for cobalt, chromium, and molybdenum additions; a similar strength increase was produced by increasing the tungsten content of the ternary alloy. Moderate decreases in strength were observed in compositions containing Al, Th, Ti, and V, elements whose oxides are not reduced in hydrogen during sintering. Higher strength values may be obtained for some of these alloys by eliminating the porosity and oxide inclusions which were present. Data for carbon and palladium additions are incomplete because optimum sintering treatments have not been established.

Figure 13 shows the effects of quaternary additions on transverse-rupture strength at 2000F. In general, the alloying additions which were best at room temperature also produced the highest strength at elevated temperature. Ruthenium additions resulted in the most drastic strength increase, with smaller rises noted for tungsten, molybdenum, and cobalt. Chromium, which promoted room-temperature strength, resulted in lower strength values at high temperature. Lower strength was also found in alloys containing thorium and titanium; some of the reduced strength may be due to the presence of oxides in the samples. Data were not obtained at 2000F for additions of Al, C, Pd, V, and Zr.

Room-temperature ductility of the 90W-Ni-Fe base was not significantly improved by any of the quaternary additions. Ductility was gradually lowered by increasing tungsten levels in the ternary alloys; additions of cobalt and molybdenum also produced similar moderate decreases in ductility. Slightly greater reduction in ductility was noted for ruthenium additions. The elements Al, Cr, Pd, Th, Ti, and V produced considerably larger reductions in the ductility of the W-Ni-Fe base.

Hardness values of the 90W-Ni-Fe base were only slightly increased by higher tungsten contents and also by the addition of molybdenum. Moderate

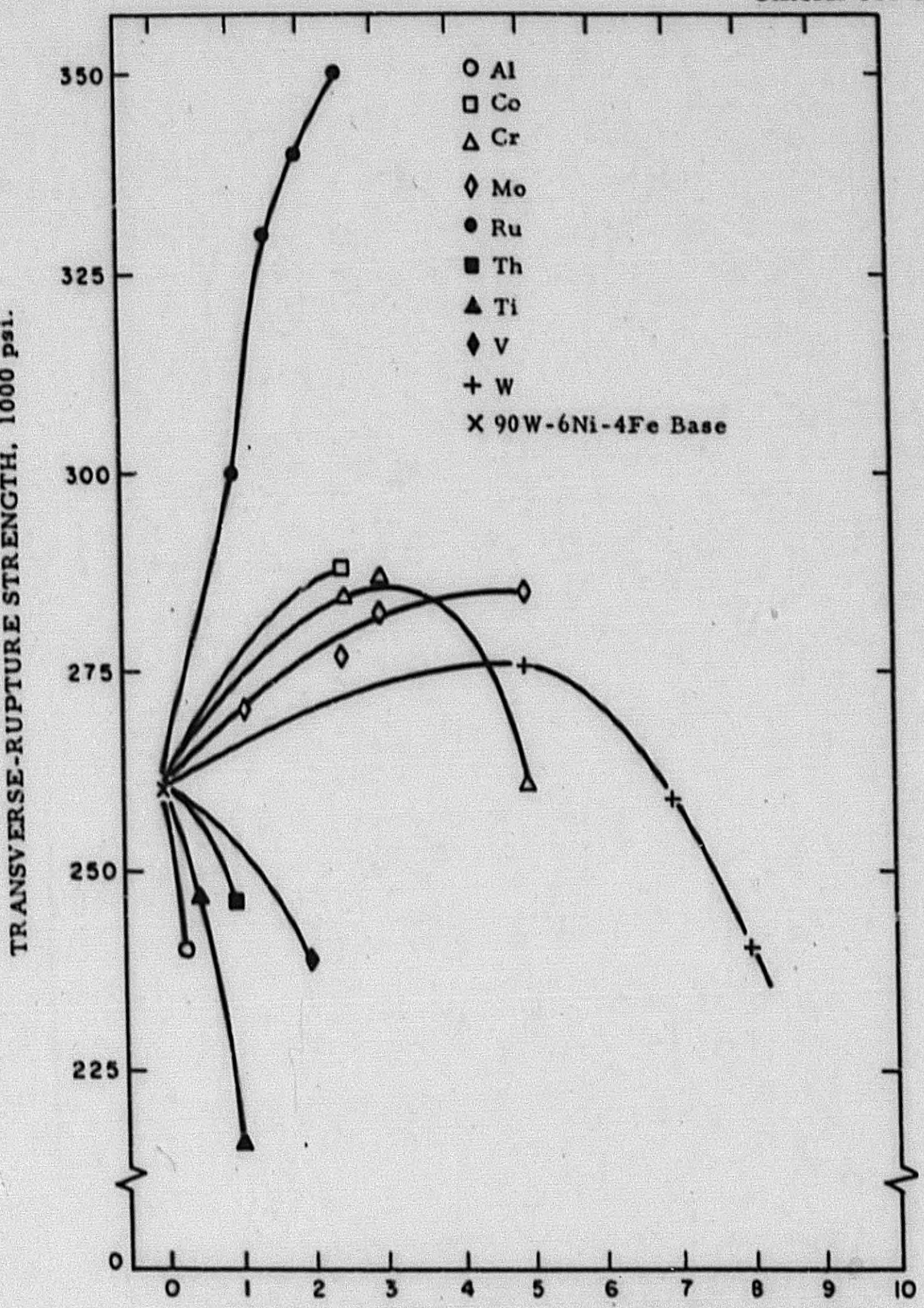



FIG. 12 - ROOM-TEMPERATURE TRANSVERSE-RUPTURE TEST DATA FOR 90W-Ni-Fe-X ALLOYS.

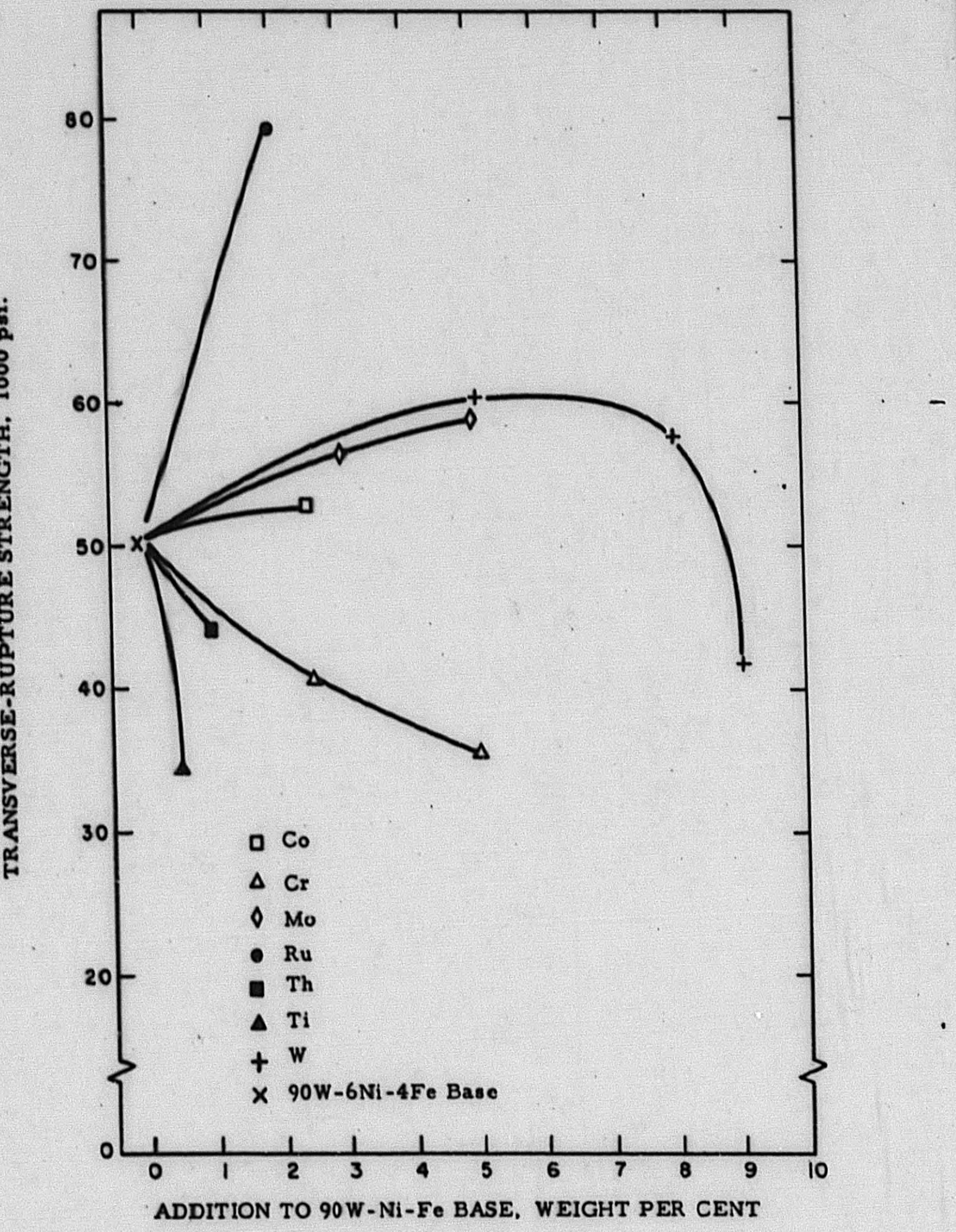



FIG. 13 - TRANSVERSE-RUPTURE TEST DATA FOR 90W-Ni-Fe-X ALLOYS AT 2000F.

increases in hardness were produced by additions of Co, Pd, Th, Ti and V. The elements chromium and ruthenium resulted in the highest hardness levels of the quaternary compositions under study.

A comparison of elevated-temperature stress-rupture properties, oxidation resistance, and stability of W-Ni-Fe-X alloys will be presented in a subsequent section of this report where data for other alloy systems will also be included.

## C. W-Ni-Co Alloy

Phase relationships in the W-Ni-Co system are somewhat similar to those in the W-Ni-Fe system. A two-phase liquid intermetallic compound field exists in the cobalt-tungsten system, similar to tungsten-iron. Alloys of 90W-Ni-Co which contain at least 6 wt% nickel have microstructures which resemble those of W-Ni-Fe alloys; the matrix phase appears to be a nickel-cobalt-rich solid solution. The 90W-6Ni-4Co alloy possessed good room-temperature ductility, and was cold-rolled to 80 per cent reduction in thickness with only minor edge cracking. Alloys containing more cobalt than nickel were weak and brittle at room temperature, and exhibited the intermediate phase constituent in the matrix. A series of ternary alloys was prepared with tungsten contents from 90 to 97 wt%; nickel-to-cobalt ratios varied from 1.5:1 to 2:1. Figure 14 presents results of room-temperature transverse-rupture tests for these compositions. Strength and ductility were at a maximum at 90 wt% tungsten, and decreased at higher tungsten levels. These materials had lower strength and ductility than were found in W-Ni-Fe alloys (compare with Figure 3).

The results of transverse-rupture tests on W-Ni-Co alloys at 2000F are shown in Figure 15. Strength values were moderately higher at all tungsten levels than were measured for W-Ni-Fe materials. A greater change was noted in the deflection values, where considerable ductility was found in the W-Ni-Co compositions. The ultimate tensile strength of a 90W-6Ni-4Co alloy was 42,000 psi, with 13 per cent elongation at 2000F. This material had the highest 2000F tensile elongation of all materials studied, and the tensile strength was exceeded only by compositions containing ruthenium.

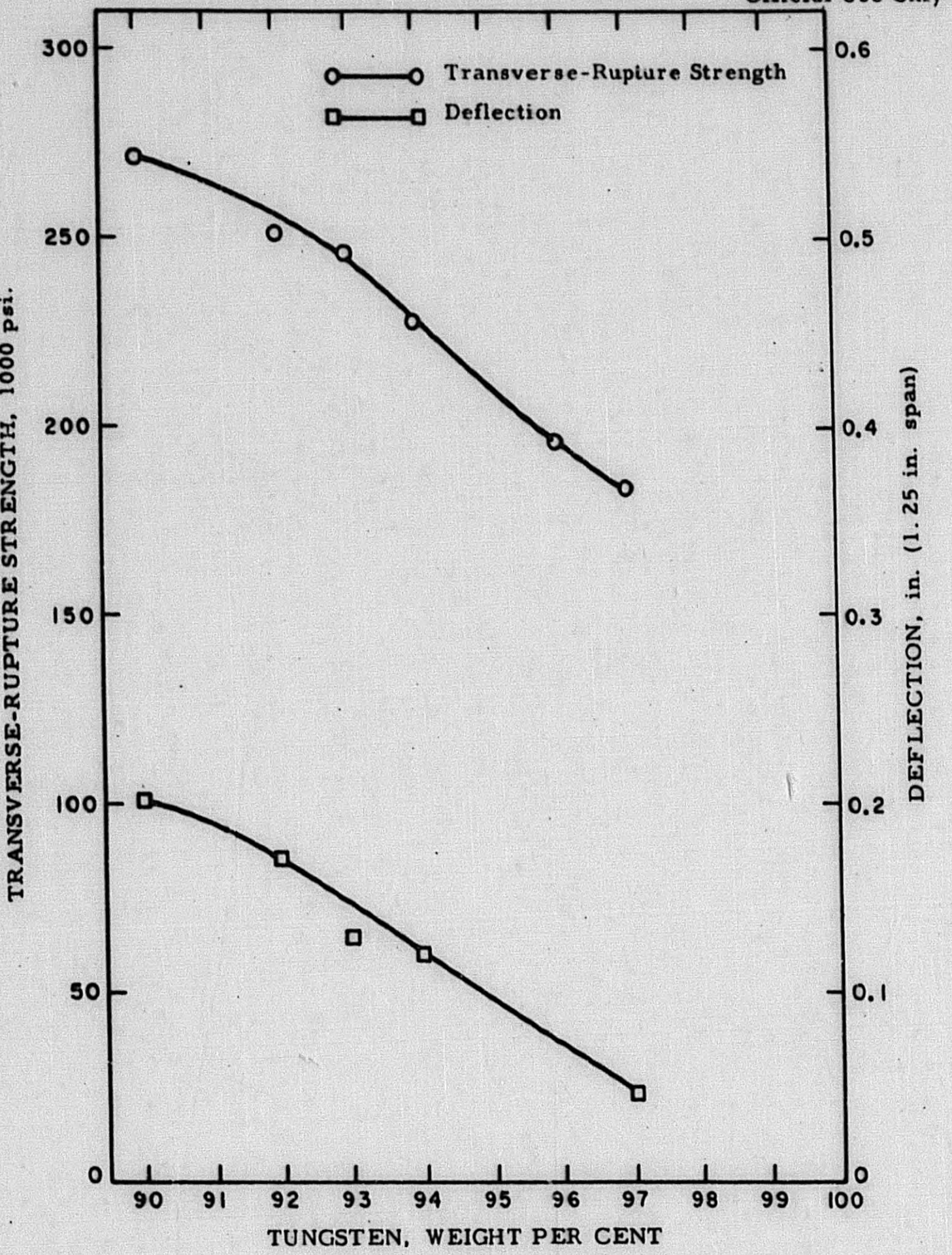



FIG. 14 - ROOM-TEMPERATURE TRANSVERSE-RUPTURE TEST DATA FOR W-Ni-Co ALLOYS.

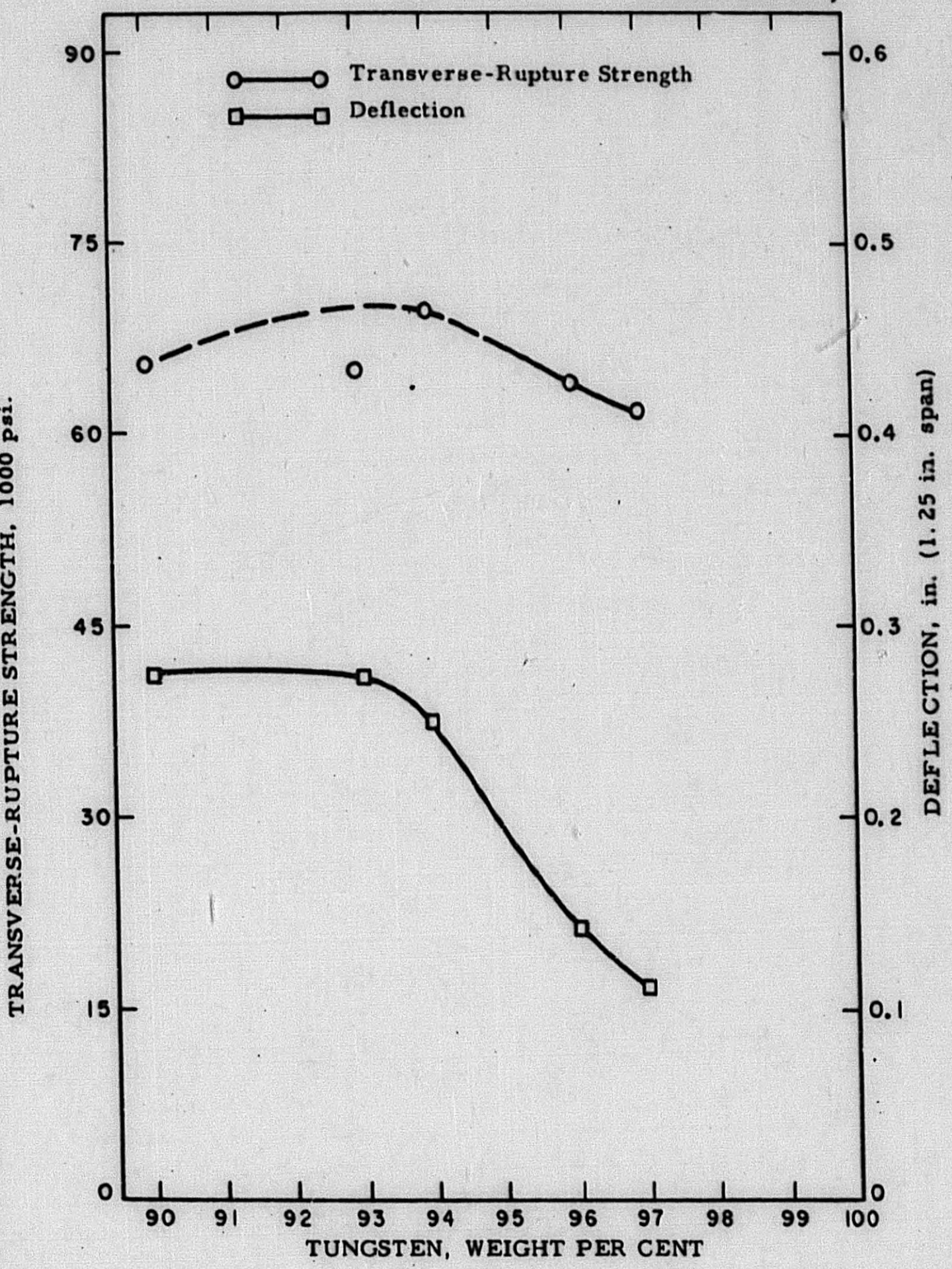



FIG. 15 - TRANSVERSE-RUPTURE TEST DATA FOR W-Ni-Co ALLOYS AT 2000F.

Elevated-temperature stress-rupture tests of the 90W-6Ni-4Co alloy showed a marked improvement over W-Ni-Fe materials at 2000F; the 100-hour rupture life was 4500 psi compared to values of about 2500 psi for the W-Ni-Fe ternary compositions. Tests were also conducted at 1600F on the 90W-6Ni-4Co material. The ultimate tensile strength was 10,500 psi with 10 per cent elongation; this strength level was some 24,000 psi higher than that of the 90W-6Ni-4Fe alloy. Stress-rupture results for the 90W-6Ni-4Co composition at 1600F show a 100-hour life at a stress level of 11,000 psi, a value slightly above that for the 90W-6Ni-4Fe material.

Ternary W-Ni-Co alloys which had been reduced 50 per cent in thickness by cold-rolling were annealed for 2 hours at temperatures between 500F and 1900F. The 90W-6Ni-4Co material exhibited a sharp increase in hardness levels above 500F; maximum values were attained between 800F and 1400F. Microstructures at these temperatures were similar to the as-rolled specimen. At 1900F a fine precipitate was noted in the matrix areas, although the original elongated grains of the rolled structure were unchanged.

Annealing treatments of 200 hours were conducted on as-sintered samples of the 90W-6Ni-4Co alloys. Temperatures ranged from 1450F to 1850F. The hardness values of the annealed materials ranged from 380 to 420 VPN (10kg) compared to 325 for the as-sintered specimens. A precipitate was noted in the matrix areas and also around the tungsten-rich grains on samples annealed at 1650F and higher. Although tensile tests were not conducted on the annealed specimens, the high hardness values suggest that the good room-temperature ductility of this alloy would be reduced. Alloys of 90W-6Ni-4Fe having the same annealing treatments also exhibited a precipitate in the matrix, but hardness levels were not increased. In the case of the 90W-6Ni-4Co material, the cobalt content was probably too high, and a composition such as 90W-7Ni-3Co may have improved thermal stability.

Chromium and ruthenium were added to the 90W-Ni-Co base. Additions of 1 and 2 wt% chromium produced a decrease in strength and ductility at both room temperature and elevated temperature compared to the 90W-6Ni-4Co base. Ruthenium at the 2 wt% level resulted in lower

room-temperature strength and ductility. However, the transverse-rupture strength at 2000F in the composition containing ruthenium was 72,100 psi, a value higher than was found in W-Ni-Co ternary alloys.

D. W-Ni-Cr Alloys

Tungsten and chromium form a continuous series of solid solutions above 2500F. Ternary W-Ni-Cr compositions containing 90 wt% tungsten were prepared with varying nickel-to-chromium ratios; alloys at the 95 wt% tungsten level were also studied. The W-Ni-Cr materials had microstructures similar to those observed in the W-Ni-Fe system. Property data at room temperature are shown in Figure 16 for 90W-Ni-Cr alloys. Transverse-rupture strength and deflection values reach a maximum for materials containing equal parts of nickel and chromium; hardness levels increased with higher chromium contents. All compositions were considerably less ductile than W-Ni-Fe alloys at room temperature. Materials containing chromium had some inclusions which were probably unreduced oxides of chromium. Sintering temperatures for these compositions were about 180F higher than for the W-Ni-Fe alloys.

At 2000F, the W-Ni-Cr materials were considerably weaker and less ductile than W-Ni-Fe compositions at similar tungsten levels. Reduced strength values were also noted in W-Ni-Fe-Cr alloys at elevated temperatures; elongation or deflection values were relatively low for all materials containing chromium. The nickel-chromium-rich matrix of W-Ni-Cr alloys was expected to improve oxidation resistance. Oxidation test results for these materials showed a relatively small improvement over other tungsten-base alloys; data are presented in a subsequent section of this report.

E. W-Ni-Ru Alloys

Ternary W-Ni-Ru compositions have been investigated to a limited extent. Three alloys were prepared at the 90 wt% tungsten level; ruthenium contents were 3, 4, and 6 wt%. Samples were sintered at temperatures up to 2840F. The microstructures revealed an extremely fine grain size compared to other alloys under study. Most of the specimens contained large, round pores which rendered property data unreliable. This

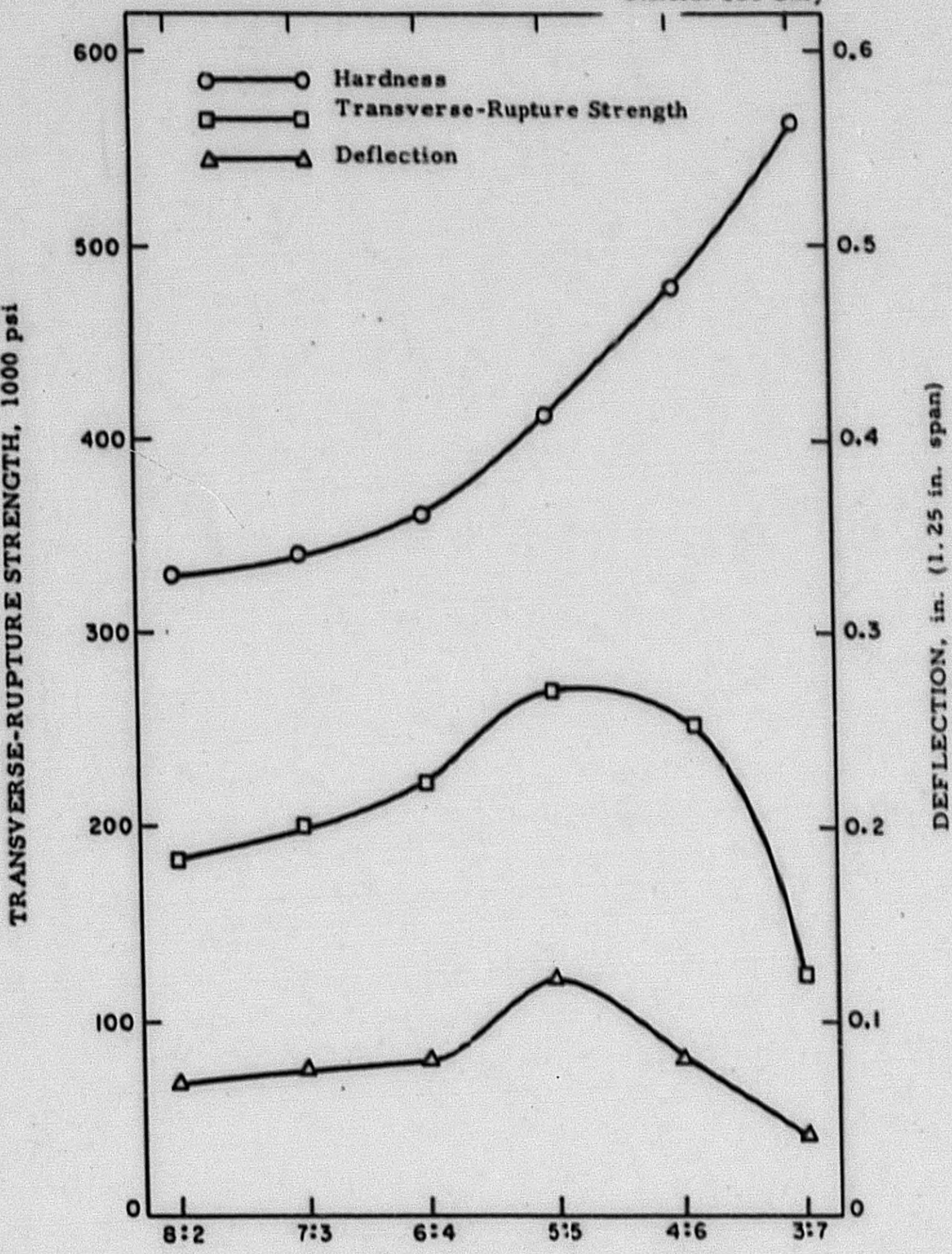



FIG. 16 - ROOM-TEMPERATURE TEST DATA FOR 90W-Ni-Cr ALLOYS.

blistering may be caused by absorption of hydrogen in ruthenium or by the presence of a volatile ruthenium oxide.

Compositions containing less ruthenium than nickel appeared to have single-phase matrix phases, although traces of another phase were detected in some of the 90W-6Ni-4Ru specimens. With higher ruthenium contents, a small amount of second phase was found to occur in the matrix as a fine, somewhat rounded precipitate. This phase was not identified; the existence of a W-Ru intermetallic compound is doubtful, according to Raub and Walter.[3] The hardness of W-Ni-Ru alloys increases with higher ruthenium contents. This effect was also noted in W-Ni-Fe-Ru materials. Limited room-temperature tests on porous samples show strength and ductility levels for a 90W-7Ni-3Ru alloy to be considerably below those of the 90W-6Ni-4Fe composition. Improved properties may be expected when suitable sintering techniques have been established for materials containing ruthenium.

## F. Other Alloy Systems

In addition to the alloys discussed above, several binary, ternary, and more complex tungsten-base alloys were also studied under this program. The systems described below were investigated to a very limited extent because initial results did not show promise, or because insufficient time was available to continue studies of the more promising materials.

### 1. W-Ni System

One binary composition, 90W-10Ni, was prepared for the purpose of investigating the effects of thermal treatment at temperatures below 1780F; an intermediate phase exists in the Ni-W system below this temperature.[4]

The microstructure of the as-sintered material exhibited rounded tungsten-rich grains in a solid-solution matrix. Microhardness tests showed that the matrix areas were somewhat harder than the tungsten grains, thereby accounting for the observed low ductility of this composition. The high hardness of the matrix is due to the solution of a large amount (40 wt%) of tungsten in nickel, according to the phase diagram shown in Figure 4. This diagram shows that iron reduces the solubility of tungsten in the matrix to about 18 wt% in the 90W-6Ni-4Fe alloy. The lower tungsten content in the

ternary matrix decreases the hardness and apparently the flow stress of this phase to below that of the tungsten grains, and a ductile alloy results. Each grain is surrounded by a ductile matrix which allows dissipation of dislocations so that slip can proceed.

The binary 90W-10Ni material was annealed for 200 hours at temperatures of 1550F to 1750F. Samples annealed at the lower temperature contained a single-phase matrix. Annealing at 1650F produced small amounts of a precipitate at the tungsten grains, and at 1750F large quantities of the nickel-tungsten intermediate phase were precipitated in the matrix and also around the tungsten grains. This precipitate resembled that found in 90W-6Ni-4Fe specimens annealed in the same temperature range. Addition of iron to the W-Ni did not prevent formation of the intermediate phase. Hardness values in the ternary composition were substantially unchanged, whereas a considerable hardness increase occurred in the 90W-10Ni material.

## 2. W-Pd System

A binary tungsten-palladium composition, 86W-14Pd, was selected for initial studies of this system; the high palladium content was required in order to provide a sufficient volume of matrix phase. Microstructures of specimens sintered in hydrogen exhibited a two-phase structure, and a considerable amount of porosity was present. This porosity may be due to the absorption and release of hydrogen during sintering; palladium is capable of absorbing large quantities of hydrogen. Initial test data show relatively low strength and ductility at room temperature, although these properties may be substantially increased by improved sintering techniques. The hardness of the sintered compacts was similar to that of 90W-Ni-Fe materials, indicating that the tungsten was not appreciably hardened by solution of palladium. This low solubility of matrix elements in tungsten is one requirement for good ductility, and the W-Pd or W-Pd-X materials appear to be promising in this respect.

3. Ternary Alloy Systems

The following ternary alloy systems were studied to a limited extent:

90W-Ni-Mo
90W-Fe-Co
90W-Fe-Cr
90W-Fe-Mo
90W-Co-Cr
90W-Co-Ru

None of the above systems possessed room-temperature ductility, and strength levels were low. Microstructures in the W-Ni-Mo alloys resembled those of W-Ni-Fe materials, although hardness values were higher. Structures in W-Fe-(Co, Cr, Mo) alloy systems showed evidence of intermediate phases; and these materials were porous, brittle, and weak at room-temperature. Similar results were noted for the W-Co-(Cr, Ru) systems. Room-temperature property data are not available because of the porous nature of the test specimens. In view of the existence of intermediate phases and/or lack of room-temperature ductility, no further experimental work was conducted on these ternary systems.

G. Comparative Data

A number of ternary and more complex tungsten-base alloy systems have been described in detail under preceding sections of this report. Some of these alloys exhibit superior properties at room temperature whereas other materials possess higher strength properties at elevated temperatures. The purpose of this "Comparative Data" section is to provide a convenient evaluation of the more promising compositions developed to date. Over one hundred alloys were studied during this reporting interval; a number of these compositions represented minor variations in alloy content or preparation methods. Complete property data for all materials investigated are presented in the Appendix.

1. Room-Temperature Properties

The results of room-temperature transverse-rupture tests on representative ternary and quaternary tungsten-base alloys are presented in Figure 17. Each alloy system is represented by one composition at the 90 wt% tungsten level except in the case of W-Ni-Fe materials which are shown at 90 and 95 wt% tungsten contents. Data for a commercial "Heavy Alloy," 90W-6Ni-4Cu, are included for comparison. Figure 17 shows that the highest strength levels occurred in the W-Ni-Fe-Ru composition, while maximum ductility was found in the 90W-6Ni-4Fe and the 90W-Ni-Fe-Co alloys. All materials were considerably stronger and more ductile that the 90W-6Ni-4Cu alloy.

2. Elevated Temperature Properties

Tensile test data for several tungsten-base alloys at 1600F and 2000F are presented in Figure 18. These results show a considerably greater spread of strength values than was found at room temperature (Figure 17). The strongest material at 2000F was the 90W-4.8Ni-3.2Fe-2Ru alloy which had an ultimate tensile strength nearly twice that of the 90W-6Ni-4Fe composition. The 90W-6Ni-4Co material was also considerably stronger at 2000F than the ternary W-Ni-Fe alloys. At 1600F, tensile strength values ranged from about 50,000 psi for 90W-6Ni-4Fe to about 73,000 psi for the 90W-4.8Ni-3.2Fe-2Ru and the 90W-6Ni-4Co compositions. These values were much higher than the level of 17,500 psi for the 90W-6Ni-4Cu alloy at this temperature. Tensile tests were not conducted on the 90W-6Ni-4Cu material at 2000F, because data at lower temperatures indicate that the strength level approaches zero at a temperature near 2000F.

A comparison of tensile strength values for three alloys at various temperatures is shown in Figure 19. The 90W-4.8Ni-3.2Fe-2Ru composition has the highest strength levels of any composition studied under this program. Data for the 90W-6Ni-4Fe alloy show considerably lower strength levels at all temperatures, although this material is considerably stronger than the commercial "Heavy Alloy," 90W-6Ni-4Cu.

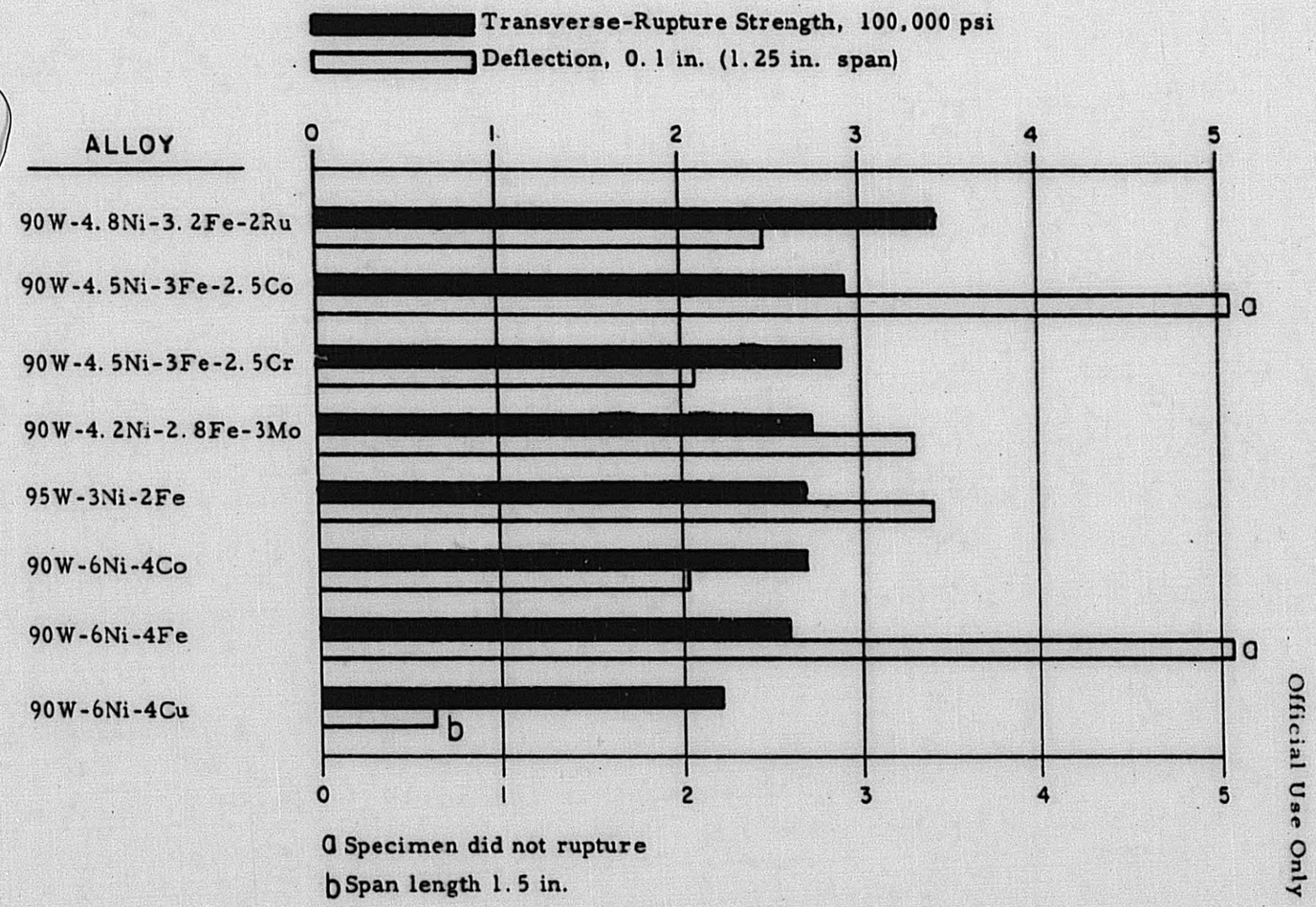



FIG. 17 - ROOM-TEMPERATURE TRANSVERSE -RUPTURE TEST DATA FOR TUNGSTEN-BASE ALLOYS

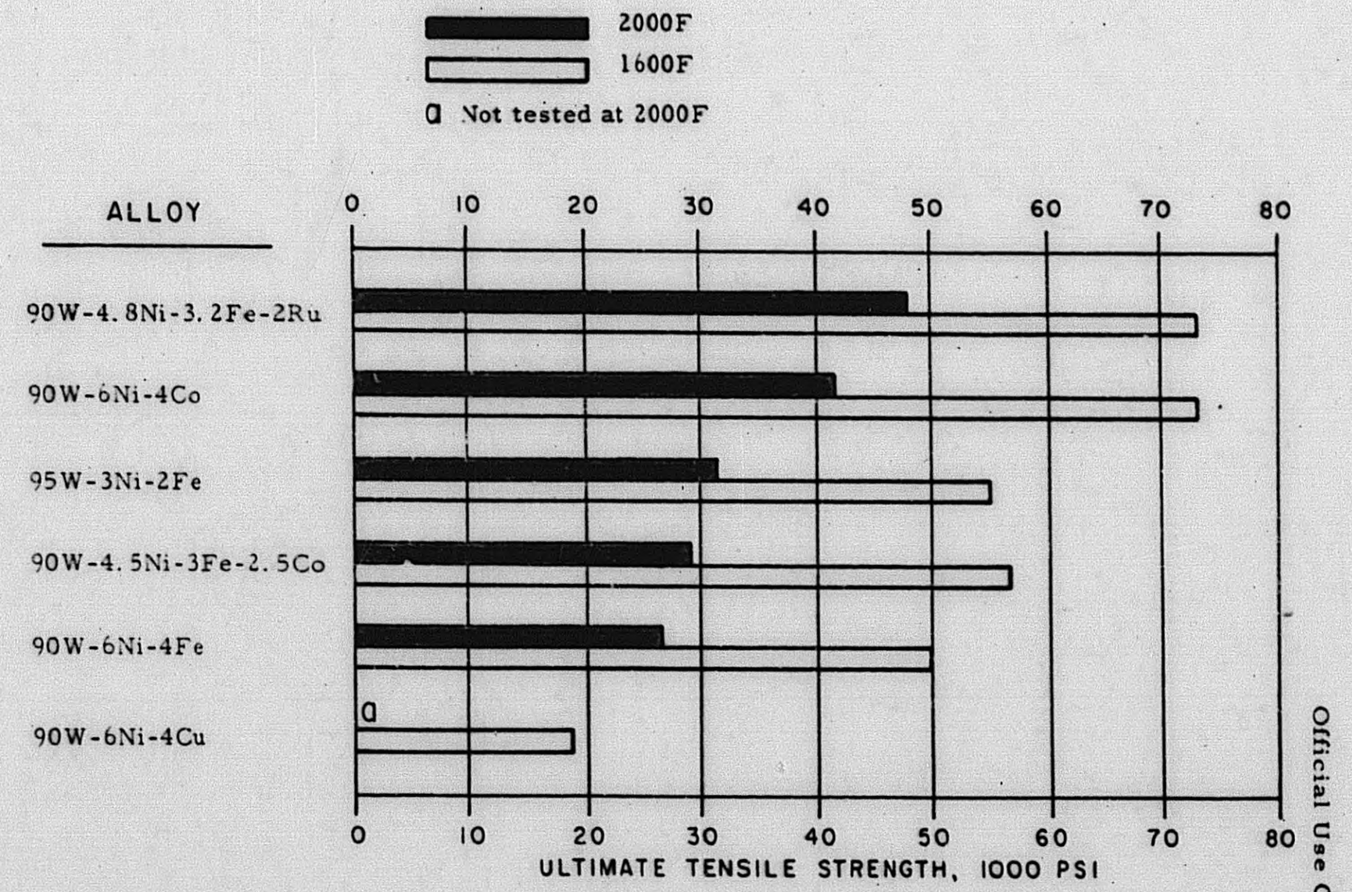



FIG. 18 - ELEVATED-TEMPERATURE TENSILE TEST DATA FOR TUNGSTEN-BASE ALLOYS.

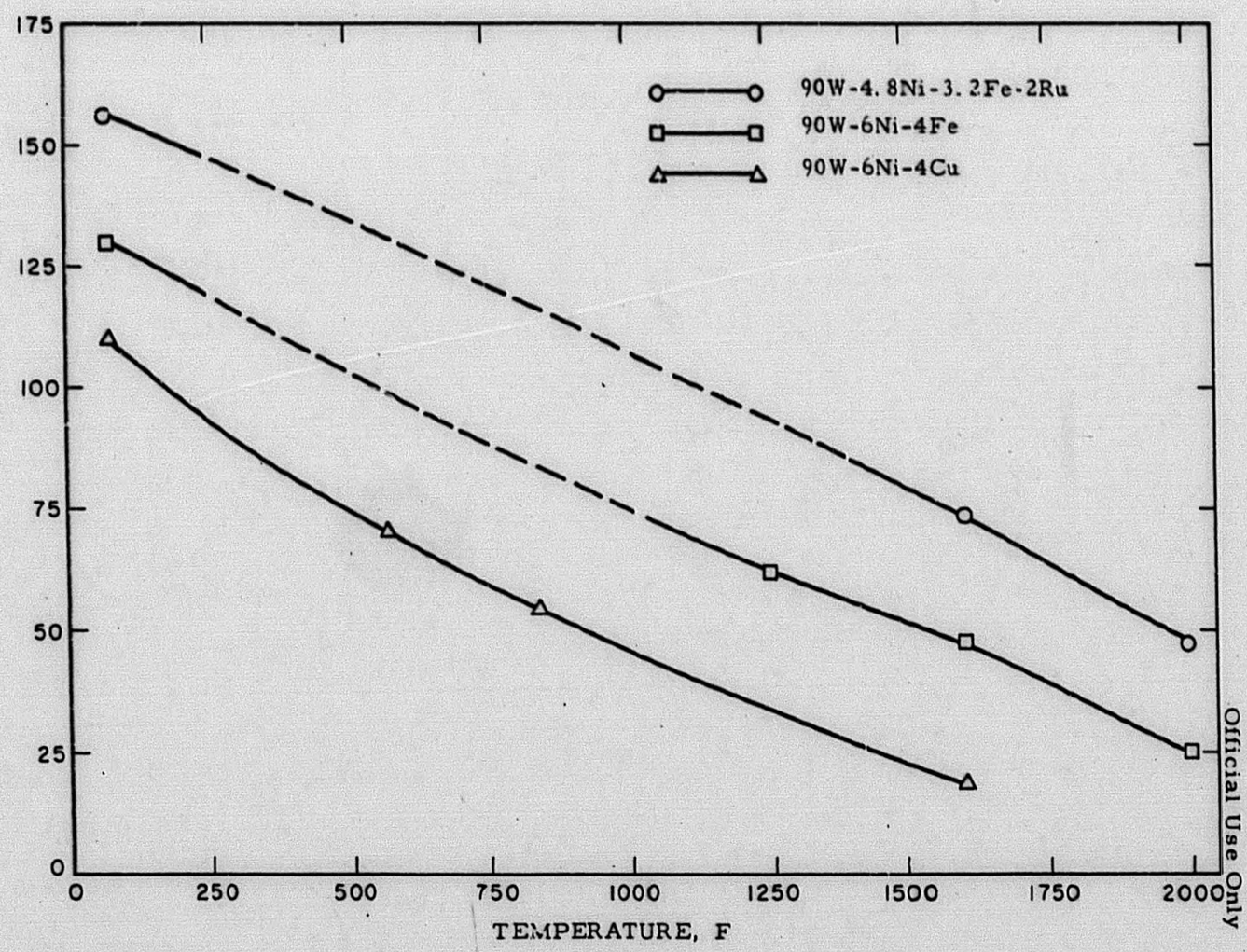



FIG. 19 - TENSILE TEST DATA FOR TUNGSTEN-BASE ALLOYS.

Although stress-rupture data are not complete for the W-Ni-Fe-Ru material, initial results at 1600F indicate that this composition was greatly superior to W-Ni-Fe and W-Ni-Co alloys. A sample of the 90W-4.8Ni-3.2Fe-2Ru material failed in 102 hours at a stress level of 15,000 psi. The 100-hour rupture life of 90W-6Ni-4Co at this temperature was at a stress-level of 11,000 psi, and the 90W-6Ni-4Fe alloy 100-hour life was at a stress of about 10,000 psi. At 2000F, the 100-hour rupture life values of various alloys were as follows:

| Alloy | Stress level, psi |
|---|---|
| 90W-6Ni-4Fe | 2,500 |
| 95W-3Ni-2Fe | 2,500 |
| 92W-3Ni-2Fe-3Cr | 3,000 |
| 90W-3Ni-2Fe-5Mo | 3,000 |
| 90W-6Ni-4Co | 4,500 |

A slight increase in stress-rupture life was produced by the chromium and molybdenum additions to the 90W-Ni-Fe base; larger increases are expected for ruthenium additions on the basis of results at lower temperatures.

## 3. Oxidation Resistance and Protection

### a. Oxidation Test Results

The oxidation resistance of unalloyed tungsten is very poor at temperatures of 2000F. Previous work on tungsten-base alloys containing at least 90 wt% tungsten has shown that these materials also have poor resistance to oxidation. Several additional alloys were studied during the current reporting interval. Included in these tests were an 85W-9Ni-6Fe alloy, and also an arc-melted 18W-49Ni-33Fe composition approximately that of the matrix phase in W-Ni-Fe alloys.

Sintered specimens of ternary and quaternary tungsten-base compositions were surface ground and heated in air at 2000F. The oxide films after 15 minutes at temperature usually ranged from 0.02 to 0.04 in. thick, and were somewhat tenacious. Results of these tests were as follows:

| Alloy | Weight gain (mg /cm$^2$/hr) |
|---|---|
| 90W-6Ni-4Fe | 227 |
| 90W-6Ni-4Co | 231 |
| 90W-5Ni-5Cr | 193 |
| 90W-4.8Ni-3.2Fe-2Ru | 196 |
| 85W-9Ni-6Fe | 110 |
| 18W-49Ni-33Fe | 9 |

The values for the higher tungsten alloys are considerably higher than results for similar materials tested under the initial program on tungsten-base alloys. These higher values are the result of modifications of the testing techniques whereby a greater supply of air was available to the specimens.

The four compositions at the 90 wt% tungsten level had relatively high weight gains; little improvement was shown by the 90W-5Ni-5Cr alloy whose matrix phase was expected to offer superior oxidation resistance. Microstructures of alloys containing 90 wt% tungsten show many adjoining tungsten-rich grains which would offer a path for oxidation. The 85W-9Ni-6Fe composition oxidized at about one-half the rate for the materials at the 90 wt% tungsten level. The increased matrix volume in this lower-tungsten alloy resulted in fewer contacts between tungsten grains. These results demonstrate that alloy variation produced little effect on the resistance to oxidation of 90W-base alloys, and that the tungsten-rich phase in these materials is probably semi-continuous. The oxidation resistance of the 18W-49Ni-33Fe matrix material shows that the poor behavior of the alloys is apparently due either to oxidation of the interconnecting tungsten-rich grains or to reactions between the oxides formed on the matrix and dispersed phases which reduce the oxidation resistance of the composite. Oxidation-resistant alloys containing somewhat less than 85 wt% tungsten might therefore be developed if the former mechanism is the one operative.

b. Oxidation Protection

The relatively poor oxidation resistance of the alloys under study was not improved by alloying. Limited efforts were undertaken to investigate protective coatings for use in air at 2000 F.

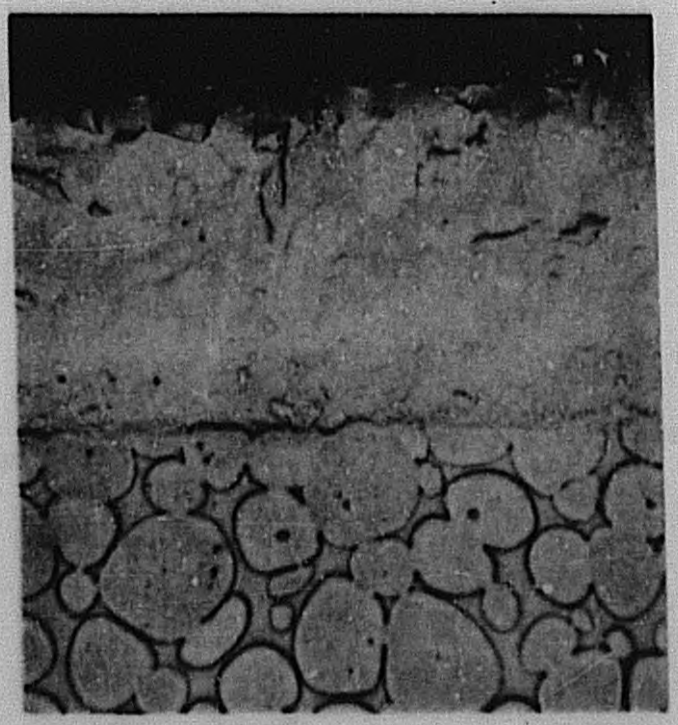
Electrodeposited chromium was applied to the surfaces of a 95W-3Ni-2Fe alloy. A modified chromic acid bath, Metal & Thermit Corporation No. CF 520, was used, with a plate thickness of 0.002 in. Initial tests after 10 hours at 2000F showed good protection was afforded except at the location of a few small surface defects. Some diffusion of nickel and iron into the chromium may have occurred at the test temperature; nickel would be expected to improve the oxidation resistance of chromium.

Experimental work using fused metal coatings gave improved results. A commercial oxidation-resistant brazing alloy, "Nicrobraz" (AMS 4775), was applied as a powder in collodion to the surface of 95W-3Ni-2Fe compacts. The coated specimens were fused for 15 minutes at 2190F in dry hydrogen. A photomicrograph of the coating and base metal is shown in Figure 20. A small diffusion layer is noted between the coating and the tungsten-rich grains. Weight gains of less than 0.1 mg/cm$^2$/hr occurred upon heating the coated samples in air at 2000F. Duplex coated specimens having a base layer of "Nicrobraz" followed by a 0.001 in. chromium plate were also tested. After 100 hours in air at 2000F an average weight gain of 0.07 mg/cm$^2$/hr was noted. A section of this test sample is shown in Figure 21. Most of the chromium plate has oxidized whereas the "Nicrobraz" material is relatively unaffected except for the presence of small inclusions, possibly oxides. The diffusion layer between this coating and the tungsten alloy has increased to a depth of about 0.002 in.

Roll-bonding was also investigated as a possible method for oxidation protection of tungsten-base alloys. Nichrome sheet was successfully bonded to a 90W-6Ni-4Fe alloy sheet by hot-rolling at 2190F. The tungsten-base alloy sheet was reduced 60 per cent in thickness during hot working, although it is believed that good bonds may be obtained with much smaller reductions. Oxidation tests have not been conducted on the roll-clad specimens.

## H. Material Processing Techniques

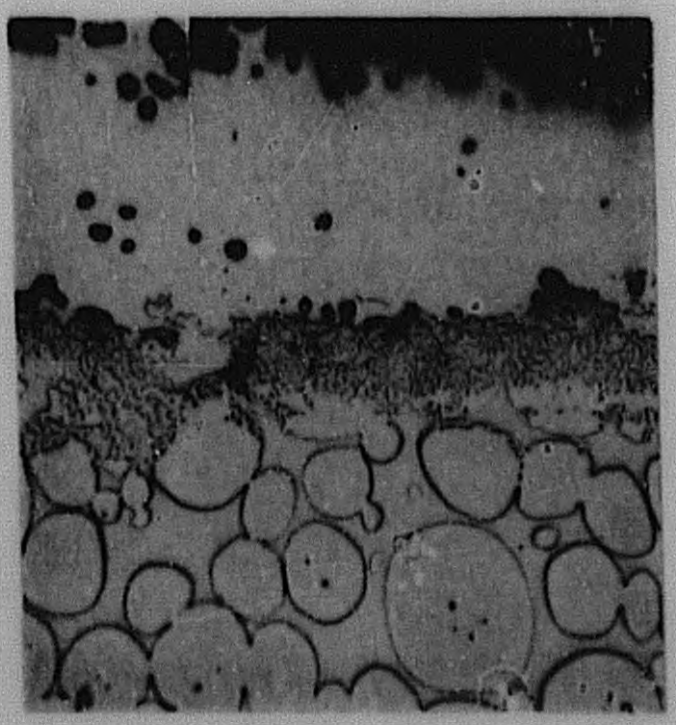
The property data reported above were obtained from samples prepared by cold-pressing followed by sintering in hydrogen. Alloys of the W-Ni-Fe system have also been prepared by other techniques which are adaptable to the fabrication of large parts having complex shape. Initial



Neg. No. 17532 X250

Fig. 20

95W-3Ni-2Fe alloy coated with "Nicrobraz" (AMS 4775). A thin diffusion zone exists at the interface. Unetched.



Neg. No. 17533 X250

Fig. 21

Oxidation test sample of 95W-3Ni-2Fe alloy coated with "Nicrobraz" and chromium plate. After 100 hrs in air at 2000F, the chromium has oxidized, and some oxides are present in the "Nicrobraz" layer. The diffusion layer at the tungsten alloy surface has increased to about 0.002 in. Unetched.

experimental work on hot-pressing in induction-heated graphite dies has shown this process to be feasible, although a carbide layer was formed on the surfaces of the compacts and some matrix material was lost by squeeze-out. Studies showed that when 90 W-6Ni-4Fe powders were loosely filled in an alundum crucible and heated in hydrogen at 2680F for 1 hour, full sintered density was attained. The excellent sinterability of cold-pressed W-Ni-Fe alloys containing up to 99.5 wt% tungsten also indicates good wettability of the tungsten particles by the liquid matrix. Accordingly, such techniques as slip casting and powder-metal extrusion appeared to be feasible. In addition, the fluidity of the matrix phase indicated that joining of sintered specimens could be accomplished by heating the area of contact to a temperature above the melting point of the matrix. The sections below discuss methods used in the fabrication and joining of W-Ni-Fe alloys; the techniques described are probably adaptable to most of the other tungsten-base alloys under study.

1. Slip Casting

Slip casting, commonly used in the fabrication of ceramics, has been applied to metal powders to a somewhat limited extent. The process described herein yielded castings of adequate green strength and density in a 95W-3Ni-2Fe alloy. The vehicle and binder consisted of a solution of collodion in alcohol (Fisher Catalog No. C-409) to which acetone was added to give a slip of the desired viscosity. About 3 cc of the collodion solution was used for 100 grams of alloy powder; an excess of acetone was added to give a thin slurry which thickened by evaporation of acetone upon stirring. The pH of the slip was near 7. Standard pottery plaster molds were used. Wall thicknesses of 1/8 inch were attained in about 1 minute after the slip was poured into the mold. After pouring out the excess slip, the castings were dried at room temperature for at least 1 hour before removal from the mold. Shrinkage upon drying was less than 1 per cent; this amount was sufficient to permit easy removal of the casting from the mold. After drying for 24 hours at room temperature, the castings were sintered in hydrogen using heating schedules identical to those for cold-pressed materials. Full sintered density was attained in the castings, although the linear shrinkage was about 24 per cent compared to 16 per cent for specimens cold-pressed

at 20 tsi. Figure 22 shows a small slip-cast cup of 95W-3Ni-2Fe; the as-sintered wall thickness is approximately 0.05 inch.

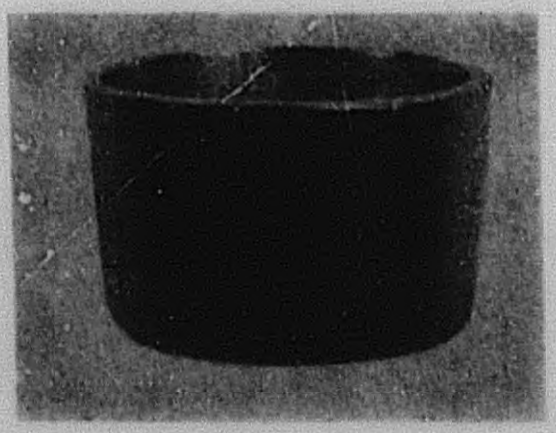
Slip casting techniques were also applied to the fabrication of larger compacts, including specimens which filled the entire mold cavity. This process represents an economical method for the preparation of large shapes, as inexpensive plaster molds are substituted for costly metal compacting dies. Initial results with large, solid specimens cast in plaster molds show promise; a few air bubbles were entrapped in the castings. These may be eliminated by vacuum-treatment of the slip before pouring and by suitable agitation of the slip after pouring into the mold.

2. Joining Method

The joining of W-Ni-Fe and other tungsten-base alloy compacts may be accomplished by placing the specimens in contact and heating the contact area to a temperature above the melting point of the matrix phase. One method involves the combined sintering and joining of cold-pressed compacts; the as-pressed specimens are held together during sintering. Other methods are concerned with the joining of previously sintered material. Several techniques are available, including induction heating, resistance welding, and tungsten-electrode inert-gas welding. Very little contact pressure is required on account of the high fluidity and wettability of the liquid matrix.

Figure 23 shows the welded area between two pieces of sintered 90W-6Ni-4Fe alloy. This joint was made by placing two surface-ground specimens in contact and heating the assembly to about 2730F for 10 minutes. Induction heating and a $90N_2$-$10H_2$ atmosphere were used. The joined area is evidenced only by the presence of a few finer tungsten-rich grains along a horizontal line in the photograph. Initial tensile test results indicate that such joints are nearly as strong as the parent material. Excellent mechanical properties are to be expected since the joint is practically indistinguishable from the original material.

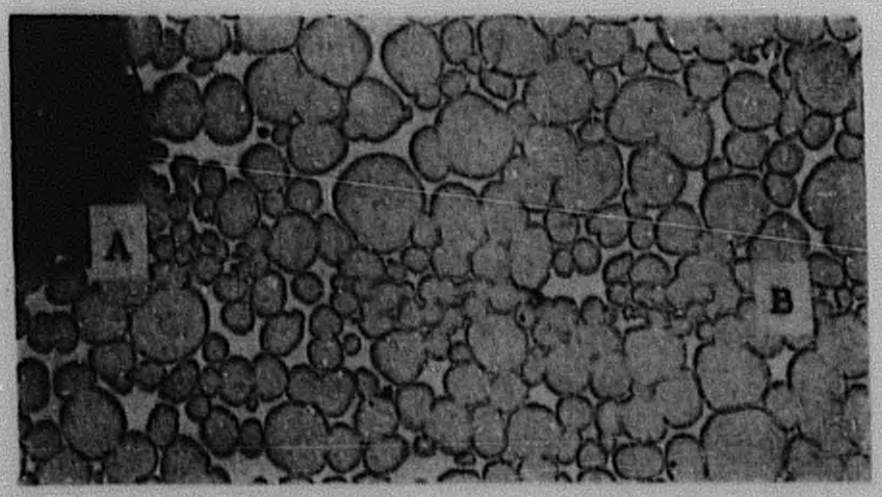
Spot-welding was also used to form sound joints between specimens of sintered W-Ni-Fe materials. The alloys welded contained up to 95 wt% tungsten, and microstructures revealed that sufficient matrix



Neg. No. 19040 X1.5

Fig. 22

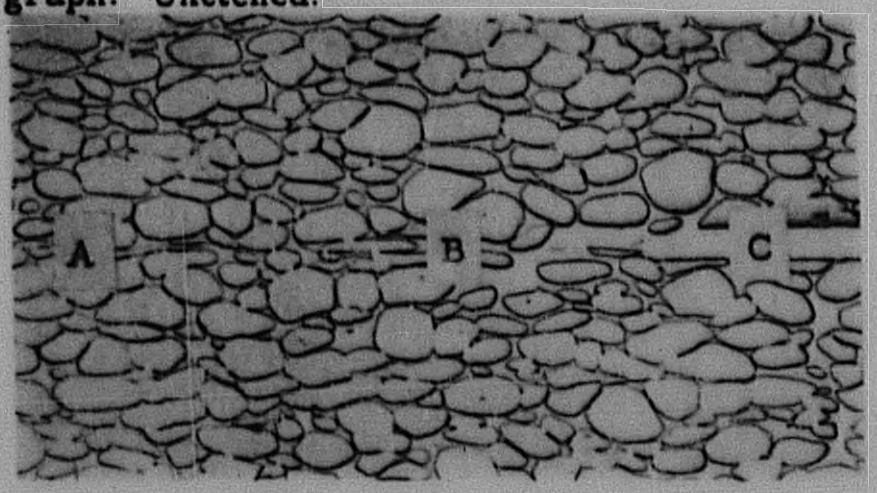
Slip-cast, sintered cup of 95W-3Ni-2Fe alloy.




Neg. No. 18574 X150

Fig. 23

Microstructure of a fused joint in 90W-6Ni-4Fe alloy. Two sintered specimens were held together and induction heated to 2730F for 10 minutes. Some small tungsten-rich grains are present in the joined area which extends along a line from A to B in the photograph. Unetched.




Neg. No. 18573 X150

Fig. 24

Microstructure of a spot-weld in cold-rolled 90W-6Ni-4Fe alloy. The two rolled sheets were in contact from A to B; the matrix flowed into the open area (B to C) which was not directly between the electrode tips. Unetched.

phase was present even at the higher tungsten level to effect bonding. Cold-rolled 90W-6Ni-4Fe sheet specimens about 1/16 in. thick were successfully spot-welded as illustrated in Figure 24. The wide band of matrix phase was outside of the area of contact between the two sheets, and the deformation of the tungsten grains occurred during cold-rolling to a 60 per cent reduction in thickness prior to welding. Several spot-welded samples have been tested by pulling the sheets apart; failures in most cases occurred outside of the welded area.

Initial experimental work using tungsten-electrode inert-gas welding indicates that this method also may be used to join sintered W-Ni-Fe materials. Microstructures through welded joints show satisfactory bonds in several of the specimens of 90W-6Ni-4Fe alloy. Improved results may be expected when optimum current and voltage requirements have been established.

## IV. SUMMARY

This program was concerned with the development of alloys containing at least 90 wt% tungsten which are readily fabricable and are capable of operating at temperatures up to 2000F. Samples from which property data were obtained were prepared by cold-pressing blended metal powders, followed by liquid-phase sintering in a hydrogen atmosphere. The alloys were evaluated at room-temperature by hardness, transverse-rupture, tensile, and bend tests. Elevated-temperature tests included transverse-rupture, tensile, stress-rupture, and oxidation resistance.

Alloys of the W-Ni-Fe system were studied at tungsten levels of 90 to 99.5 wt%. Microstructures consisted of tungsten-rich grains in a matrix of nickel and iron which contained about 18 wt% tungsten in solution. Properties of these alloys may be summarized as follows:

1. Room-temperature tensile strength attained a maximum of 130,000 psi at 95 wt% tungsten, and decreased rapidly above 97 wt% tungsten.

2. Room-temperature ductility decreased uniformly with increasing tungsten contents. The tensile elongation of the 90W-6Ni-4Fe

alloy was 20-25%. Fabricability was excellent; alloys containing up to 97 wt% tungsten could be cold-rolled.

3. Ultimate tensile strength values at 2000F attained a maximum of 34,000 psi at 97 wt% tungsten, and decreased sharply above 98 wt% tungsten. Tensile elongations were lower than at room temperature.

4. An intermediate phase was precipitated in the matrix of a 90W-6Ni-4Fe alloy after annealing for 200 hours at temperatures near 1550F; the effects of this compound on properties have not been fully investigated.

5. The 100-hour rupture life at a stress level of about 2500 psi was found in W-Ni-Fe alloys containing 90, 95, and 97 wt% tungsten.

A number of quaternary additions were made to a 90W-Ni-Fe base in an effort to improve elevated-temperature properties. The effects of additions of Al, C, Co, Cr, Mo, Pd, Ru, Th, Ti, V, and Zr are summarized below:

1. Cobalt additions up to 2.5 wt% increased the room-temperature strength without loss of ductility. Higher cobalt contents resulted in the formation of a brittle intermediate phase, probably $W_6Co_7$. A moderate increase in strength and ductility at 2000F was produced by the addition of 2.5 wt% cobalt to the 90W-Ni-Fe base; a cold-rolled 90W-4.5Ni-3Fe-2.5Co composition was recrystallized after annealing for 2 hours at 1900F.

2. Chromium additions to the 90W-Ni-Fe base resulted in considerably reduced ductility and increased hardness at room temperature; strength levels reached a maximum at 3 wt% chromium. At 2000F, the W-Ni-Fe-Cr alloys were not as strong and ductile as W-Ni-Fe materials.

3. Molybdenum additions up to 5 wt% produced moderate increases in strength levels at room temperature and at 2000F; ductility was gradually lowered with increasing molybdenum contents.

4. Ruthenium additions to the 90W-Ni-Fe base resulted in the strongest materials developed under this program. Strength levels for a 90W-4.8Ni-3.2Fe-2Ru alloy were some 20 per cent higher than the strongest W-Ni-Fe composition at room temperature, and adequate ductility was maintained. At 2000F, the alloy containing ruthenium had an ultimate tensile strength of 46,700 psi; this value is considerably higher than for annealed, unalloyed tungsten. Initial stress-rupture results at 1600F show a 100-hour life at a stress level of 15,000 psi, a 50 per cent improvement over the ternary base. Further property improvements may be expected in the W-Ni-Fe-Ru system when compositions and sintering treatments are optimized.

5. Al, C, Pd, Th, Ti, V, and Zr were also added to the 90W-Ni-Fe base. On the basis of limited test data, no significant property improvements were produced by any of these quaternary additions. Some oxidation of Al, Th, Ti, V, and Zr occurred during sintering, due to traces of water vapor and oxygen in the hydrogen atmosphere used for sintering. As a result, the sintered compacts usually contained oxide inclusions and porosity which affected the test results. The oxides of these elements are not reduced in hydrogen at temperatures used in these studies; inert-atmosphere or vacuum sintering may be required to produce clean microstructures. Quaternary alloys containing carbon and palladium are in preliminary stages of investigation, and satisfactory sintering treatments have not been established.

Experimental work under this program also included investigations of other ternary and more complex tungsten-base alloy systems. The results of these studies are as follows:

1. W-Ni-Co alloys having nickel-to-cobalt ratios of at least 3:2 had microstructures which resembled those of W-Ni-Fe compositions. Larger amounts of cobalt resulted in the appearance of a brittle intermediate phase. The room-temperature strength and ductility of W-Ni-Co materials were somewhat lower than in

W-Ni-Fe alloys, although a 90W-6Ni-4Co composition was readily cold-workable. Elevated-temperature tensile strength values of W-Ni-Co alloys were higher than for all other compositions studied with the exception of alloys containing ruthenium. Tensile elongations were considerably higher than in other materials under investigation. The 100-hour rupture life of the 90W-6Ni-4Co alloy at 2000F was at a stress level of about 4,500 psi, some 80 per cent higher than for W-Ni-Fe alloys. Annealing the as-sintered 90W-6Ni-4Co material for 200 hours in the range of 1450F to 1850F produced hardness increases associated with the formation of an intermediate phase.

2. W-Ni-Cr compositions exhibited structures similar to those in the W-Ni-Fe system. Equal amounts of nickel and chromium were found to yield optimum properties at the 90 wt% tungsten level. The 90W-5Ni-5Cr alloy at room temperature was considerably harder and much less ductile than the 90W-6Ni-4Fe material. At 2000F, tensile strength values in W-Ni-Cr compositions were some 20 to 40 per cent below those of W-Ni-Fe alloys at similar tungsten levels.

3. Other ternary alloy systems investigated included W-Ni-(Mo-Ru), W-Fe-(Co, Cr, Mo), and W-Co-(Cr, Ru). Of these, only the W-Ni-Ru materials, which were fine grained and moderately strong, appeared to offer promise on the basis of preliminary results. The remaining alloy systems were characterized by low strength and ductility, and intermediate phases were present in some compositions.

Oxidation tests at 2000F on several tungsten-base compositions showed that alloying had little effect on oxidation resistance. An arc-melted 18W-49Ni-33Fe sample having a composition similar to that of the matrix phase in a 90W-6Ni-4Fe alloy was also tested; this material had a weight gain value only 4 per cent of that of the 90W-6Ni-4Fe composite. These results indicate that the tungsten-rich phase in the high-tungsten alloys is semi-continuous and that rapid oxidation proceeds through adjacent tungsten grains.

Oxidation protection of W-Ni-Fe alloys was investigated to a limited extent; electrodeposition, fused alloy coatings, and roll bonding were included in these studies. Chromium plate, 0.002 in. thick, gave good protection for 10 hours at 2000F except at the location of a few defects in the plating. A fused coating of "Nicrobraz" (AMS 4775) showed improved results. One sample coated with "Nicrobraz" and an outer layer of electrodeposited chromium had a weight gain of 0.07 mg/cm$^2$/hr after 100 hours at 2000F. Nichrome sheet was satisfactorily bonded to a 90W-6Ni-4Fe specimen by rolling at 2190F.

Several material processing techniques were studied in addition to the conventional cold-pressing and hydrogen-sintering method used in the preparation of test specimens. Powders of 90W-6Ni-4Fe which were loosely filled in an alumina crucible sintered to full density upon heating above the melting point of the matrix phase. Initial results using slip-casting techniques were promising. The binder and vehicle consisted of a collodion-alcohol solution to which acetone was added. Standard pottery plaster molds were used, and the pH of the slip was near 7. The sintered castings of a 95W-3Ni-2Fe alloy were strong, ductile, and fully dense. Casting large sections by this method appears to be feasible.

Sintered W-Ni-Fe materials were joined by several methods; these techniques depend on heating the area of contact between adjoining specimens to a temperature at which the matrix phase becomes liquid. Induction-heated joints were almost indistinguishable from the parent material. Spot-welding gave satisfactory results on as-sintered and on cold-rolled specimens containing up to 95 wt% tungsten. Limited efforts using tungsten-electrode inert-gas welding indicate some promise for this method.

## V. LOGBOOKS AND CONTRIBUTING PERSONNEL

Data for this report are recorded in ARF Logbooks C-8488, -8489, -8490, -8491, -8492, -8553, -8554, -8884, -8885, -8886, and -9113.

The following personnel have been the principal contributors to the planning and execution of this work:

| | | |
|---|---|---|
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| J. R. Dvorak | - | Metallography |
| A. L. Hess | - | Project Technician |
| F. C. Holtz | - | Project Leader |
| J. J. Connelly, Jr., LCDR | - | AEC Project Leader |
| R. J. Van Thyne | - | Supervisor |

Respectfully submitted,

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## TABLE I

## ROOM-TEMPERATURE PROPERTIES OF W-Ni-Fe-X ALLOYS

| Composition(wt%) | | | | Sintering Temp(F)[a] | Hardness VPN(10Kg) | Transverse-Rupture Test Data | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | Rupture Strength(psi) | Yield Point(psi)[b] | Deflection, in.[c] (1.25 in. span) |
| W | Ni | Fe | Other | | | | | |
| 90 | 6 | 4 | | 2680 | 300-315 | 260,000 | 175,000 | 0.5+[d] |
| 93 | 4.2 | 2.8 | | 2680 | 305-315 | 270,000 | 180,000 | 0.38 |
| 95 | 3 | 2 | | 2760 | 310-320 | 270,000 | 185,000 | 0.35 |
| 97 | 1.8 | 1.2 | | 2840 | 320-330 | 255,000 | 185,000 | 0.18 |
| 98 | 1.2 | 0.8 | | 2840 | 330 | 240,000 | 185,000 | 0.12 |
| 99 | 0.5 | 0.5 | | 2840 | 330 | 190,000 | 180,000 | 0.06 |
| 99.5 | 0.25 | 0.25 | | 2840 | 335 | 100,000 | 100,000 | 0.02 |
| 90 | 5.85 | 3.9 | 0.25Al | 2680 | 305 | 239,000 | 160,000 | 0.24[d] |
| 90 | 4.5 | 3 | 2.5Co | 2680 | 315 | 290,000 | 185,000 | 0.5+[d] |
| 90 | 4 | 2.5 | 3.5Co | 2760 | 360 | Brittle | | |
| 90 | 3 | 2 | 5Co | 2760 | 415 | Brittle | | |
| 93 | 3.15 | 2.1 | 1.75Co | 2680 | | 300,000 | 187,000 | 0.38 |
| 95 | 2.5 | 1.5 | 1Co | 2680 | | 280,000 | 182,000 | 0.28 |
| 95 | 2.25 | 1.5 | 1.25Co | 2680 | 321 | 284,000 | 184,000 | 0.29 |
| 95 | 2.75 | 1 | 1.25Co | 2680 | | 277,000 | 179,000 | 0.26 |
| 95 | 3 | 0.5 | 1.5Co | 2680 | | 234,000 | 182,000 | 0.14 |
| 96 | 1.8 | 1.2 | 1Co | 2680 | 325 | 276,000 | 189,000 | 0.21 |
| 97 | 1.5 | 0.75 | 0.75Co | 2760 | 339 | 244,000 | 191,000 | 0.13 |
| 90 | 3 | 2 | 2Co-2Cr | 2840 | 373 | 180,000 | 172,000 | 0.06 |
| 90 | 5.4 | 3.6 | 1Cr | 2760 | 312 | 276,000 | 176,000 | 0.27 |
| 90 | 4.5 | 3 | 2.5Cr | 2840 | 350 | 280,000 | 195,000 | 0.20 |
| 90 | 4 | 3 | 3Cr | 2840 | 382 | 283,000 | 230,000 | 0.14 |
| 90 | 3.5 | 1.5 | 5Cr | 2840 | 460 | 261,000 | 220,000 | 0.08 |
| 92 | 3 | 2 | 3Cr | 2840 | 406 | 290,000 | 250,000 | 0.12 |
| 95 | 2 | 1 | 2Cr | 2840 | 433 | 252,000 | 220,000 | 0.07 |
| 90 | 3.5 | 1.5 | 2Cr-3Mo | 2840 | 390 | 285,000 | 216,000 | 0.15 |

**TABLE I (con't)**

| Composition(wt%) | | | | Sintering Temp. (F)[a] | Hardness VPN(10Kg) | Transverse-Rupture Test Data | | |
|---|---|---|---|---|---|---|---|---|
| W | Ni | Fe | Other | | | Rupture Strength(psi) | Yield Point(psi)[b] | Deflection, in. (1.25 in span)[c] |
| 90 | 4.2 | 2.8 | 3Mo | 2840 | 331 | 273,000 | 180,000 | 0.33 |
| 90 | 3 | 2 | 5Mo | 2840 | 342 | 285,000 | 216,000 | 0.2 |
| 9. | 3 | 2 | 3Mo | 2840 | | 255,000 | 180,000 | 0.18 |
| 95 | 1.5 | 1 | 2.5Mo | 2840 | 342 | 247,000 | 204,000 | 0.12 |
| 99 | 0 25 | 0.25 | 0.5Mo | 2840 | | Brittle | | |
| 90 | 3.5 | 1.5 | 3Mo-2Ti | 2840 | 317 | 205,000 | 165,000 | 0.06 |
| 90 | 3.6 | 2.4 | 2Mo2V | 2760 | 325 | 210,000 | 166,000 | 0.07 |
| 90 | 3.5 | 1.5 | 3Mo2Zr | 2760 | | Blistered | | |
| 90 | 4.8 | 3.2 | 2Pd | 2680 | | 240,000 | 152,000 | 0.30 |
| 90 | 5.4 | 3.6 | 1Ru | 2680 | 330 | 299,000 | 200,000 | 0.48 |
| 90 | 5.1 | 3.4 | 1.5Ru | 2760 | 366 | 331,000 | 227,000 | 0.37 |
| 90 | 4.8 | 3.2 | 2Ru | 2840 | 373 | 339,000 | 231,000 | 0.25 |
| 90 | 4.5 | 3 | 2.5Ru | 2840 | 397 | 351,000 | 238,000 | 0.20 |
| 90 | 4.5 | 2 | 2Ru-1.5Co | 2760 | 370 | 294,000 | 208,000 | 0.22 |
| 90 | 5.5 | 1 | 2Ru-1.5Co | 2760 | 357 | 219,000 | 181,000 | 0.08 |
| 90 | 5.4 | 3.6 | 1Th | 2760 | 317 | 244,000 | 167,000 | 0.26 |
| 90 | 5.7 | 3.8 | 0.5Ti | 2680 | 305 | 243,000 | 164,000 | 0.23 |
| 90 | 5.25 | 2.25 | 2.5Ti | 2760 | | Blistered | | |
| 90 | 4.8 | 3.2 | 2V | 2680 | 312 | 238,000 | 182,000 | 0.15 |
| 90 | 5.4 | 3.6 | 1Zr | 2680 | | Blistered | | |
| 90 | 5.25 | 2.25 | 2.5Zr | 2760 | | Blistered | | |

a. 1 to 1.5 hours at temperature.
b. Yield point calculated from transverse-rupture stress strain curve; refer to text, page 5.
c. Maximum deflections for specimens approximately 0.125 in. thick.
d. Specimen did not rupture in test.

TABLE III

ROOM-TEMPERATURE PROPERTIES OF W-Ni-Cr BASE ALLOYS

| Composition (wt%) | | | | Sintering Temp. (F)[a] | Hardness VPN(10Kg) | Transverse-Rupture Test Data | | |
|---|---|---|---|---|---|---|---|---|
| W | Ni | Cr | Other | | | Rupture Strength(psi) | Yield Point(psi)[b] | Deflection, in. (1.25 in. span)[c] |
| 90 | 8 | 2 | | 2840 | 327 | 184,000 | 167,000 | 0.07 |
| 90 | 7 | 3 | | 2840 | 342 | 197,000 | 173,000 | 0.08 |
| 90 | 6 | 4 | | 2840 | 363 | 220,000 | 200,000 | 0.08 |
| 90 | 5 | 5 | | 2840 | 415 | 270,000 | 240,000 | 0 12 |
| 90 | 4 | 6 | | 2840 | 480 | 255,000 | 239,000 | 0.08 |
| 90 | 3 | 7 | | 2840 | 572 | 117,000 | 115,000 | 0.04 |
| 95 | 2.5 | 2.5 | | 2840 | 394 | 234,000 | 230,000 | 0.05 |
| 90 | 4.5 | 4.5 | 1Co | 2840 | 442 | 252,000 | 215,000 | 0.10 |

a. 1.5 to 2.0 hours at temperature.
b. Yield point calculated from transverse-rupture stress-strain curve; refer to text, page 5.
c. Maximum deflections for specimens approximately 0.125 in. thick.

TABLE II

ROOM-TEMPERATURE PROPERTIES OF W-Ni-Co BASE ALLOYS

| Composition(wt%) | | | | Sintering Temp. (F)[a] | Hardness VPN(10Kg) | Transverse-Rupture Test Data | | |
|---|---|---|---|---|---|---|---|---|
| W | Ni | Co | Other | | | Rupture Strength(psi) | Yield Point(psi)[b] | Deflection, in. (1.25 in. span)[c] |
| 90 | 8 | 2 | | 2680 | 320 | 210,000 | 180,000 | 0.10 |
| 90 | 6 | 4 | | 2680 | 325 | 272,000 | 192,000 | 0.20 |
| 90 | 5.5 | 4.5 | | 2680 | 333 | 162,000 | 160,000 | 0.05 |
| 90 | 4 | 6 | | 2680 | 410 | 59,000 | 59,000 | 0.01 |
| 90 | 2 | 8 | | 2760 | | 55,000 | 55,000 | 0.01 |
| 92 | 5.3 | 2.7 | | 2760 | | 248,000 | 185,000 | 0.17 |
| 92 | 5 | 3 | | 2760 | 325 | 254,000 | 183,000 | 0.18 |
| 93 | 4.7 | 2.3 | | 2760 | 325 | 247,000 | 190,000 | 0.13 |
| 94 | 4 | 2 | | 2760 | 325 | 228,000 | 185,000 | 0.12 |
| 94 | 3.7 | 2.3 | | 2760 | 325 | 218,000 | 190,000 | 0.07 |
| 96 | 2.7 | 1.3 | | 2760 | 325 | 195,000 | 175,000 | 0.07 |
| 97 | 2 | 1 | | 2760 | 325 | 179,000 | 175,000 | 0.04 |
| 90 | 6 | 3 | 1Cr | 2760 | 323 | 198,000 | 164,000 | 0.09 |
| 90 | 6 | 2 | 2Cr | 2760 | | 221,000 | 186,000 | 0.11 |
| 90 | 4.5 | 3 | 2.5Cr | 2760 | 405 | Brittle | | |

a. 1 to 1.5 hours at temperature.
b. Yield point calculated from transverse-rupture stress-strain curve; refer to text, page 5.
c. Maximum deflections for specimens approximately 0 125 in. thick.

TABLE IV

ROOM-TEMPERATURE PROPERTIES OF MISCELLANEOUS TUNGSTEN-BASE ALLOYS

| Composition(wt%) | Sintering Temp(F)[a] | Hardness VPN(10Kg) | Transverse-Rupture Test Data: Rupture Strength(psi) | Yield Point(psi)[b] | Deflection, in. (1.25 in. span)[c] |
|---|---|---|---|---|---|
| 90W-10Ni | 2760 | 363 | 135,000 | 130,000 | 0.03 |
| 86W-14Pd | 2760 | 312 | 86,000 | 85,000 | 0.01 |
| 90W-6Co-4Cr | 2760 | | Brittle, porous | | |
| 90W-8Co-2Cr | 2760 | | Brittle, porous | | |
| 90W-7Co-3Ru | 2760 | | Brittle, porous | | |
| 90W-4Fe-6Co | 2760 | | Brittle, porous | | |
| 90W-7Fe-3Co | 2760 | | Brittle, porous | | |
| 90W-6Fe-4Cr | 2760 | | 44,000 | 44,000 | 0.01 |
| 90W-4Fe-6Cr | 2760 | | 41,000 | 41,000 | 0.01 |
| 90W-7Fe-3Mo | 2760 | | Brittle, porous | | |
| 90W-7Ni-3Mo | 2760 | 354 | 136,000 | 130,000 | 0.03 |
| 90W-5Ni-5Mo | 2760 | 342 | 139,000 | 135,000 | 0.02 |
| 90W-7Ni-3Ru | 2760 | 440 | 119,000 | 115,000 | 0.03 |
| 90W-6Ni-4Ru | 2840 | 470 | 76,000 | 75,000 | 0.02 |
| 90W-4Ni-6Ru | 2840 | 473 | Brittle | | |
| 92W-6Ni-2Ru | 2760 | 375 | 181,000 | 175,000 | 0.04 |
| 90W-6.5Ni-2Ru-1.5Co | 2760 | 373 | 161,000 | 155,000 | 0.04 |

a. 1 to 1.5 hours at temperature.
b. Yield point calculated from transverse-rupture stress-strain curve; refer to text, page 5.
c. Maximum deflections for specimens approximately 0.125 in. thick.

## TABLE V

## PROPERTIES OF TUNGSTEN-BASE ALLOYS AT 2000F

| Composition(wt%) | Transverse Rupture Strength(psi) | Deflection, in. (1.25 in. span)[a] | Ultimate Tensile Strength(psi) | Elong. % |
|---|---|---|---|---|
| 90W-8Ni-2Co | 62,500 | 0.25 | | |
| 90W-6Ni-4Co | 64,600 | 0.25 | 42,600 | 12 |
| 90W-5.5Ni-4.5Co | 73,900 | 0.25+[b] | | |
| 92W-5.3Ni-2.7Co | 62,000 | 0.25 | | |
| 92W-5Ni-3Co | 61,600 | 0.25 | | |
| 93W-4.7Ni-2.3Co | 63,000 | 0.25 | | |
| 94W-4Ni-2Co | 69,600 | 0.23 | 36,700 | 7 |
| 96W-2.7Ni-1.3Co | 61,400 | 0.12 | | |
| 97W-2Ni-1Co | 61,600 | 0.09 | | |
| 90W-6Ni-3Co-1Cr | 49,300 | 0.13 | | |
| 90W-6Ni-2Co-2Cr | 45,500 | 0.06 | | |
| 90W-5.5Ni-2.5Co-2Mo | 62,800 | 0.25 | | |
| 90W-4Ni-6Cr | 36,000 | 0.03 | | |
| 90W-4.5Ni-4.5Cr-1Co | 46,900 | 0.02 | | |
| 90W-6Ni-4Fe | 50,000 | 0.17 | 27,000 | 9 |
| 95W-3Ni-2Fe | 62,000 | 0.11 | 31,800 | 7 |
| 98W-1Ni-1Fe | | | 34,000 | 2.5 |
| 99W-0.5Ni-0.5Fe | 42,300 | 0.02 | 21,000 | 0.5 |
| 90W-5.85Ni-3.9Fe-0.25Al | 41,200 | 0.19 | | |
| 90W-4.5Ni-3Fe-2.5Co | 52,400 | 0.17 | 27,400 | 7 |
| 93W-3.15Ni-2.1Fe-1.75Co | 61,000 | 0.15 | | |
| 95W-2.5Ni-1.5Fe-1Co | 59,000 | 0.11 | | |
| 95W-2.25Ni-1.5Fe-1.25Co | 61,200 | 0.11 | | |
| 95W-3Ni-0.5Fe-1.5Co | 62,400 | 0.13 | | |
| 97W-1.5Ni-0.75Fe-0.75Co | 65,600 | 0.08 | | |

TABLE V (con't)

| Composition(wt%) | Transverse Rupture Strength(psi) | Deflection, in. (1.25 in. span)[a] | Ultimate Tensile Strength(psi) | Elong. % |
|---|---|---|---|---|
| 90W-4.5Ni-3Fe-2.5Cr | 41,000 | 0.06 | | |
| 92W-3Ni-2Fe-3Cr | | | 30,000 | 1.5 |
| 95W-2Ni-1Fe-2Cr | 45,500 | 0.01 | | |
| 90W-4.2Ni-2.8Fe-3Mo | 56,400 | 0.20 | | |
| 90W-3Ni-2Fe-5Mo | | | 31,400 | 2.5 |
| 95W-1.5Ni-1Fe-2.5Mo | 58,600 | 0.02 | | |
| 90W-4.8Ni-3.2Fe-2Ru | | | 46,700 | 3 |
| 90W-4.5Ni-3Fe-2.5Ru | 77,800 | 0.10 | | |
| 90W-4.5Ni-2Fe-2Ru-1.5Co | 80,700 | 0.11 | | |
| 90W-5.5Ni-1Fe-2Ru-1.5Co | 73,700 | 0.09 | | |
| 90W-5.4Ni-3.6Fe-1Th | 44,800 | 0.20 | | |
| 90W-5.25Ni-2.25Fe-2.5Ti | 34,000 | 0.17 | | |
| 90W-6.5Ni-2Ru-1.5Co | 72,100 | 0.10 | | |

a. Maximum deflection for specimen approximately 1.25 in. thick.

b. Specimen did not rupture in test.





END