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THE PILOT PRODUCTION AND EVALUATION OF CHROMIUM ALLOY SHEET AND PLATE

by

L. J. Goetz, J. R. Hughes and W. F. Moore

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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LAMP METALS AND COMPONENTS DEPARTMENT
LAMP DIVISION
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FINAL REPORT

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ABSTRACT

Approximately 180 lbs. of chromium alloy sheet and plate of a nominal composition of Cr-5w/o W-0.05w/o Y were produced by a process based on inert atmosphere induction melting, hot extrusion and warm rolling. Utilizing alloy material from this pilot production program, a second program was conducted which identified the temperature limits for warm rolling the Cr-5W-0.05Y alloy and examined the relationships between interstitial impurity content and mechanical properties.

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I SUMMARY

Approximately 180 lbs. of chromium alloy sheet and plate of a nominal composition of Cr-5w/o W-0.05w/o Y were produced in a pilot production program. Nine 100-lb. heats of this alloy were converted to thin gauge sheet by a process based on inert atmosphere induction melting, hot extrusion and warm rolling. To avoid contamination from oxidation and nitridation during extrusion and rolling, a coextruded molybdenum cladding was employed. Due to thermal expansion mismatch between the molybdenum and the alloy, extensive alloy cracking was encountered during extrusion and rolling. This was overcome in extrusion by a stress-relief anneal and slow cooling treatment of the extrusion. Similarly, the rolling process used slow heating and cooling rates and special practices designed to minimize thermal cycling during rolling.

Utilizing alloy material from the manufacturing program, a second program was conducted which identified the temperature limits for warm rolling the Cr-5W-0.05Y alloy and examined the relationship between interstitial impurities and alloy transition temperature and strength. A qualitative correlation was found between alloy yield strength at 1000°F (540°C) and carbon plus oxygen content. Although not conclusive, evidence of a relationship between oxygen content and transition temperature is indicated.

II INTRODUCTION

This is a final report of work performed on two chromium alloy manufacturing programs conducted during the period May 25, 1965, to January 20, 1967. The primary objective of the first program, performed under NASA contract NAS 3-7901, was to manufacture 180 lbs. of chromium alloy sheet and plate of a nominal composition of Cr-5w/o W - 0.05w/o Y (hereinafter referred to as Cr-5W). This material was subsequently used by NASA in other and separate work directed toward the development of oxidation and nitridation resistant coatings for chromium base alloys. The objective of the second program, conducted under NASA contract NAS 3-7919, was to obtain certain metallurgical data on Cr-5W alloy material which would better define and optimize secondary working processes for the manufacture of this alloy in particular, and other chromium alloys in general.

Consolidation and primary fabrication work, i.e., melting and extrusion, of the chromium alloy ingots required for the production program, was performed at the General Electric Research and Development Center in Schenectady, New York. Secondary fabrication, i.e., rolling, and testing work for this program was conducted by the General Electric Refractory Metal Rolling Operation plant in Cleveland, Ohio, a part of the Lamp Metals and Components Department. All work performed in obtaining metallurgical data for the second program was conducted under the direction of the Refractory Metal Rolling Operation plant and was performed either in this facility or in the specialized test facilities of the Refractory Metals Laboratory of the Lamp Metals and Components Department.

In this report the work of the two programs is described chronologically. The approach employed and performance and experience obtained in producing 180 lbs. of the Cr-5W alloy is discussed first. Some of the material from this program was then used to obtain data in the second program and this effort is discussed by tasks and/or phases.

III PRODUCTION OF 180 LBS. OF Cr-5W ALLOY (NAS 3-7901)

A. APPROACH

The General Electric Company, through the interest and efforts of several separate departments has maintained a continuing interest and activity in chromium base alloys. In the course of these previous efforts, several full-scale quantities of chromium alloys, primarily of the C207 type (Cr-7W-.05Y-ZTC), have been produced. Since the objectives of this program did not include process development work, proven processing methods for chromium alloys were required and previous processing experience was used wherever possible. It was on this basis that the induction melting and extrusion techniques were chosen. Previous General Electric work in the secondary fabrication of chromium alloys was performed largely at hot work temperatures; that is, above the recrystallization temperature. Because the Cr-5W alloy was allocated for use in coating development programs, it was essential that the finished product be free of surface contamination and have a low ductile-brittle transition temperature for bend test evaluation of coatings. For these reasons, it was decided to depart from previous experience and fabricate the required quantities of sheet and plate by rolling at temperatures below the recrystallization temperature to achieve a low DBBTT and with the extrusion cladding intact during rolling to eliminate atmospheric surface contamination.

Table I illustrates in condensed outline form the original chemical, physical and metallurgical requirements for the Cr-5W alloy sheet and plate of this program.

B. INGOT MELTING

1. General

A number of characteristics of chromium and chromium-base alloys must be considered in melting and casting these materials:

1) Chromium is a reactive metal and in the molten state reacts readily with oxygen and nitrogen in the atmosphere and with ceramic crucible materials.

2) Chromium has a high vapor pressure and cannot be melted in high vacuum.

3) Chromium and most chromium-base alloys are susceptible to cracking due to thermal shock, and extreme care must be taken during casting to avoid cracks.

4) Chromium and chromium-base alloys exhibit a large amount of shrinkage during solidification.

From a consideration of these characteristics, it is obvious that chromium-base alloy melting and casting is not a simple, routine process.

In induction melting these alloys, a ceramic crucible with a minimum tendency to react with chromium must be used. Melting must be carried out in a protective atmosphere, but not in high vacuum. Casting must be accomplished in such a way that there is sufficient directional cooling from the bottom to keep all the shrinkage cavity at the top of the ingot, but cooling at any point must not be drastic enough to cause cracking. A further obstacle to the directional freezing of the actual ingot shape described in this report is the fact that it has no taper. The ingot was designed without taper in order to minimize the loss of expensive material in machining the tapered ingot to the cylindrical shape required for extrusion.

2. Equipment

Melting and casting operations were carried out in a vacuum induction melting furnace with a capacity for melting and tilt-pouring 200 lbs. of steel within the chamber. The power source was a 200 KW, 1920 CPS motor-generator set. Lowest pressure attainable in the chamber was approximately 10 microns Hg.

The crucible material deemed best suited to melting this alloy was zirconia stabilized with 8% yttria. The nominal 150-lb. size was used. The pouring lip and pouring basin were both made of lime-stabilized zirconia.

The mold consisted of a coarse-grained lime-stabilized zirconia tube, 5 in. ID x 5-3/4 in. OD x 20 in. long. Actually, since a 14 in. length of uniform density tubing was the maximum length commercially available, it was necessary to cement together a 14-in. length and a 6-in. length. The capacity of this mold is 100 lbs. of the Cr-5W alloy. In order to cool the casting directionally from the bottom, but not chill it so rapidly that it would crack, a copper stool, plasma-spray-coated with a thickness of approximately 15 mils of zirconia, was used as a mold bottom. To further enhance the possibilities for directional freezing, the bottom 13 in. of the zirconia tube was packed in steel grit and the top 8 in. was packed in highly insulating zirconia bubble grain. In addition, a calrod hot top heater encircled the top

6 in. of the zirconia tube.

The entire mold set-up is shown schematically in Figure 1.

3. Melt Stock

The raw materials used in melting the Cr-5W alloy are as follows:

Chromium	-	"Elchrome H.P." electrolytic chromium obtained from Union Carbide Corp.
Yttrium	-	Nuclear grade sponge obtained from Lunex Co.
Tungsten	-	99.97% pure sintered bar obtained from General Electric Company.

Chemical analysis specifications and actual lot analyses of the various lots of chromium used are shown in Table 2. This table also relates the lot analysis to the specific ingots made from each lot. Actual analyses were not available for the tungsten and the yttrium. However, a vendor's typical analysis of the yttrium is given in Table 3.

4. Preparation of Charge

Because of the poor packing factor of electrolytic chromium and the resulting poor coupling obtained in induction melting, it is necessary to briquet this material. For optimum charging of the 150-lb. size crucible, briquets of 2 in. diameter, 3 in. diameter and 3-3/4 in. diameter were required in addition to several pounds of loose, unbriquetted material. The tungsten and 0.5w/o yttrium were briquetted with the chromium and placed in the initial crucible charge. This quantity of yttrium was added as a "getter" for oxygen and nitrogen and was not expected to be retained. The yttrium actually needed to meet chemical specification was added as a late addition.

5. Melting Procedure

When the vacuum chamber reached a pressure in the range of 10-20 microns of mercury, power was applied in vacuum and the charge was allowed to outgas at a temperature approaching the melting point. The pressure typically increased to approximately

80 microns and then started to decrease. Power was then shut off and the chamber was allowed to pump back down to approximately 10 microns. Power was again applied and the charge heated in vacuum to a temperature just below the melting point. Argon was then admitted to a pressure of 2/3 of atmospheric and melting was completed at this pressure. Pressures typically leveled off in a 25-45 micron range before admission of argon to the chamber. The charge was held molten for a period of 15 minutes to allow for uniform distribution of the tungsten and to permit the yttrium to accomplish its gettering function. At this point, the final yttrium addition of 0.059w/o was made, and the heat was held molten for five minutes to insure complete homogeneity. It was then poured and allowed to cool over night in the vacuum chamber.

6. Ingot Quality

In general, surface condition of the ingots varied from good at the bottom to fair at the top and poor in the hot top section. The condition of the ingot surface is dependent upon the amount of metal-mold reaction that takes place. Since the lower portions of the ingot cool most rapidly, they exhibit very little metal-mold reaction and thus have the best surface condition. The metal in the hot top portion of the ingot is purposely maintained molten as long as possible to aid in feeding the shrinkage cavity. Consequently, the metal-mold reaction is greatest in this portion and the resulting surface condition is poor. Figure 2 is typical of the surface condition of these ingots after sandblasting.

The ingots were examined radiographically for internal defects. It was found that 13-14 in. of sound ingot were consistently obtained.

A slice approximately 3/8 in. thick was cut from the bottom of each ingot, and from this slice samples were taken for chemical analysis. Vacuum fusion gas analysis was employed for oxygen, nitrogen and hydrogen. The tungsten and yttrium were analyzed gravimetrically, the carbon and sulfur by combustion, and the phosphorus by a spectrophotometric method. Results of ingot analyses are shown in Table 4.

As can be seen from this table, one ingot No. 74-100, was prepared from recycled scrap. This was attempted in an effort to re-use process scrap and thereby increase overall process yields. The charge was prepared from approximately equal quantities of surface machined and degreased hot top croppings from heats No. 47-100, 57-100, 58-100, 64-100 and 67-100. Since these sections were massive in comparison to the chromium flake and had been deoxidized by previous melting, it was concluded that a yttrium addition to the charge would not be needed. Accordingly, only the 0.059w/o final yttrium addition was made during melting. This was apparently not adequate and the final ingot chemistry results show a very low residual yttrium content and high oxygen content. However, this approach still appears to be technically feasible and certainly merits further consideration. Possibly a more successful method might be based on the addition of one cleaned hot top, approximately 20 lbs. in weight, to each virgin charge of 80 lbs.

C. EXTRUSION

1. Preparation of Ingots for Extrusion

Following radiography the hot top was removed from each ingot by cutting on a power hack saw at a point just below the bottom of the primary shrink cavity. This sawed surface and the sawed surface at the bottom end of each ingot, from which a slice for chemical analysis had been taken, were examined by the "dye-check" method for hairline cracks which an x-ray could well have missed. Cracks of this type were discovered at the bottom end of two of the nine ingots. On each of these ingots an additional slice was saw-cut from the bottom end and the resulting surface was again examined using dye-check. In both instances the second cut yielded a surface which was free of any indications in the dye-check test.

The ingots were then lathe-turned until the circumference was free of surface defects or until these defects were few enough so that they could be spot-conditioned using a hand grinder. A chamfer was then machined on each ingot at the bottom end, which corresponded to the nose or front end of the extrusion billet. At this point an extrusion can, made from molybdenum powder by hydrostatic pressing, sintering and rough machining, was finish-machined to fit the turned ingot (extrusion billet) very closely.

2. Canning

The molybdenum can, two molybdenum plugs and the extrusion billet were then degreased and assembled for loading into a welding chamber. The chamber was pumped down to a vacuum of approximately 100 microns, then back-filled with helium just prior to welding. The molybdenum can was then sealed using a tungsten electrode by making a circumferential arc weld between the can and one of the moly plugs which had been inserted into the can at the back end of the extrusion billet. The welded assembly, ready for extrusion, is shown in Figure 3.

3. Extrusion

The canned billets were extruded through hot-work steel dies having a conical entry and a rectangular orifice with nominal dimensions of 1 in. x 4-1/4 in. The dies were coated by plasma spraying with zirconia to a nominal thickness of 0.015 to 0.020 in. Fiske 604 and Corning 7052 glass were used as lubricants, the Fiske 604 being swabbed heavily on the die and in the container just prior to each extrusion.

The billets were heated to a nominal temperature of 1425°C in a hydrogen atmosphere furnace, transferred to a 1250-ton Loewy horizontal press and extruded. The resulting data are given in Table 5. The surfaces of the extruded bars, without exception, looked and felt very smooth as sandblasted, although three of the bars exhibited a few shallow longitudinal striations in the molybdenum cladding. No quantitative measurements of surface roughness were made. The zirconia die coating did an excellent job of preventing any pickup of die steel on the extruded bars. Some minor spalling of the coating was encountered; however, and the dies were recoated after each extrusion to remove any chance of die pickup.

Extrusion produced an excellent bond between the Cr alloy and the molybdenum cladding. None of the cut sections made on the extruded bars showed the slightest evidence of any separation between cladding and core. The molybdenum was left on the bars throughout subsequent processing.

The first bar produced was air-cooled from the extrusion temperature to approximately 700°C, then buried in sand to cool from 700°C to room temperature. The bar was sandblasted and shipped to the General Electric Company's Refractory Metal Rolling Operation in Cleveland, Ohio, for further processing. On sectioning the bar, some hairline cracks were discovered, which prevailed over a significant portion of the length of the bar and resulted in a substantial lowering of the yield of sound material obtained. Some minor modifications in processing procedure were made in extruding each of the next four ingots. These bars were sand cooled from the extrusion temperature to room temperature, but cracks were still found, varying in form and severity from bar to bar.

It was concluded that the cracks were not being formed during extrusion, but resulted from a combination of residual stresses from the extrusion and the stresses generated during cooling as a consequence of the difference between the thermal expansion coefficients of molybdenum and chromium. Since there was an appreciable thickness of molybdenum cladding (approximately 0.080 in.) on the edges of the extruded bars, and the coefficient of thermal expansion of molybdenum is lower than that of chromium, a tensile stress is developed in the chromium core on cooling. This combination of residual stresses was evidently enough to cause the chromium alloy to crack as it cooled below the ductile-brittle transition temperature. As a result, the remainder of the extruded bars were stress relieved by placing them, while still hot, in a furnace at 1050°C and holding for one hour. At this point, the furnace power was turned off and the bar was allowed to cool slowly with the furnace. The bars were removed from the furnace at a temperature of 250°C or below and sand cooled to room temperature. These bars were also sandblasted before shipping to the Refractory Metal Rolling Operation for further processing. The amount of cracking in all four of the stress-relieved bars was greatly reduced from that in the first five, and some of the stress-relieved bars exhibited no cracking at all. Thus, for this particular alloy, it appears that the use of a stress relief anneal immediately following extrusion results in a substantially higher yield of good extrusion.

4. Discussion of Extrusion Data

Extrusion temperatures were determined with an optical pyrometer. The extrusion ratio, taking into consideration the thickness of the die coating, was nominally 5.25 to 1, but varied slightly from this value because the dies used had slightly different orifice sizes, and the thickness of the zirconia coating varied by a few mils from die to die. The "K" factor, or "extrusion constant" was based on the breakthrough force, and was calculated for each ingot according to the empirical formula $P = K \ln R$ where $P =$ extrusion pressure and $R =$ extrusion ratio.

In an effort to minimize cracking in the alloy in the nose of the extrusion, one billet, No. 57-100 was extruded in tandem with a steel nose block. As can be seen from Table 5, the force required to extrude this ingot was significantly higher than that required for any other ingot. This occurred because the nose block was heated to only 850°C, rather than 1425°C, in order that the strength of the steel would at least match that of the Cr-5W alloy. Apparently, the steel block chilled the nose of the alloy billet and consumed some lubricant from the die face, resulting in a higher extrusion force. After sectioning this extrusion for rolling, it was concluded that the use of the steel nose block had not reduced the severity of cracking from extrusion.

The cropped and machined ingots weighed from 49.9 to 58.9 lbs. and ranged in size from 4.5 to 4.7 in. in diameter by 12-1/8 in. to 13-1/2 in. long. The canned ingots, ready for extrusion, weighed from 80.6 to 91.7 lbs. and measured between 14-1/4 in. and 15-7/8 in. long. The extruded bars ranged from 0.928 to 1.053 in. in thickness, from 3.927 in. to 4.183 in. in width, and from 70 in. to 77-3/8 in. in overall length. On a given bar the thickness usually varied approximately 0.015 in. and the width approximately 0.030 to 0.040 in.

D. ROLLING

1. Equipment

All rolling work for this program was performed in the pilot plant facilities of the General Electric Refractory Metal Rolling Operation plant in Cleveland, Ohio. The mill in this facility is a 50 hp, two-high or four-high combination mill with a 7 in. roll face. All rolling for this program was performed in the two-high configuration using 12 in. diameter work rolls. Associated with the rolling mill in the same facility are several resistance heated, high-temperature furnaces, equipped for atmospheres of hydrogen, argon, nitrogen or air. A hydrogen atmosphere was used for all work with the Cr-5W alloy. Auxiliary operations, such as cutting, pickling, inspecting, etc., were performed using production sized equipment of the Refractory Metal Rolling Operation plant.

2. Extrusion Cracking

Macroscopic examination of cut sections from the first extrusion, heat No. 47-100, revealed the presence of essentially two cracks through the thickness of the Cr-5W alloy, running parallel to each other and the axis of extrusion. Each crack was within 3/8 in. to 1/2 in. of one edge of the extrusion, but did not extend into or go through the molybdenum cladding. The microstructure of this extrusion, as well as all subsequent extrusions, was fully recrystallized with an approximate grain size of 128 gr/mm^2 (ASTM No. 4). The extrusion cracks were clearly transgranular and invariably occurred in approximately the same location on heats 47-100 through 67-100, inclusive. In each extrusion, roughly 2/3 of the extruded length was affected in this manner. In order to isolate and identify the degree of cracking present in each extruded section prior to rolling, the cut sections were wet belt sanded and macroetched electrolytically in a saturated oxalic acid solution. After this treatment, any cracks present could be seen with the unaided eye and readily identified. This inspection method was used on all extrusions rolled during this program. An example of a section prepared by this technique is shown in Figure 4.

After a suitable rolling process had been developed, it was observed that sections of Cr-5W alloy could be rolled to finish size without removing the extrusion cracks prior to rolling. The finished rolled strip or plate contained separations in the alloy at the same geometric location as the original flaws, but no further propagation occurred during rolling. The cladding material did not fail during rolling and the finished rolled section with cladding was usually in one piece.

3. Cutting

With the aid of the electrolytic oxalic acid macroetch described above, it was rapidly established that the Cr-5W alloy is easily heat checked and/or cracked in cutting or grinding operations and must be cut by methods using liberal coolant flow, light cuts and minimum tool vibration. The best techniques found for cutting thick sections of Cr-5W alloy are: 1) a cutting fluid lubricated and cooled bandsaw, or 2) cutting fluid lubricated and cooled machining slitter. The former method was used for sectioning all extruded alloy. Thin sections of material can be cut best using a water cooled silicon carbide abrasive saw and this method was employed for the finish cutting of all rolled sheet and plate. Except for wet belt sanding, no satisfactory grinding or lapping methods were found.

4. Rolling Process

Initial rolling trials, using Cr-5W material from heat No. 47-100, were complicated to some extent by a lack of accurate information on the recrystallization temperature and transition temperature for this alloy. This was further compounded by the previously described cracks present in the extrusion, cracking occurring during the rolling process from thermal shock and/or the thermal expansion mismatch between the molybdenum cladding and the Cr-5W alloy, and heat checking or cracking generated in sawing the extruded sheet bar. To some extent these different factors were not completely identified until several Cr-5W heats had been processed to final gauge. Thus, the extruded alloy in heat No. 47-100 was largely consumed in performing process development work.

It was planned to roll the Cr-5W alloy with the co-extruded molybdenum cladding intact in order to minimize oxidation and nitridation and thus insure the absence of contamination in the rolled strip. Initial rolling trials showed this approach to be feasible, but indicated that allowances must be made to compensate for the thermal expansion mismatch between cladding and alloy during the thermal cycling encountered during rolling. Although initial rolling temperatures in the range of 900°C to 1350°C were employed, repeated catastrophic failures by longitudinal cracking were encountered in rolling until thermal cycling was effectively minimized. Preliminary test work on partially rolled material from these first experiments established that the ductile-brittle transition and recrystallization temperatures for alloy warm worked approximately 90% were on the order of 400°C and 1100°C, respectively. These measurements then, roughly identified the maximum and minimum temperature limits in which the Cr-5W alloy could be rolled without generating cracks from thermal expansion or causing recrystallization and an increase in the transition temperature. While the latter effect was controlled by using a rolling temperature schedule which was substantially below 1100°C the former effect could only be achieved with a carefully controlled rolling practice. In summary, the process steps instituted which successfully avoided cracking from clad-alloy expansion mismatch are as follows:

1) Preparatory to rolling, heat the extrusion or sheet bar sections from room temperature to the rolling temperature at a rate not in excess of 200°C per hour.

2) Use high rolling speeds, i.e., 150 sfpm, and a thick outer can or cladding of mild steel, i.e., 0.125 in. thick, to avoid excessive chilling in the roll bite.

3) Keep out-of-furnace handling time short during rolling and do not allow the rolled material to cool below 500°C during the rolling process.

4) After rolling is completed, furnace cool to approximately 300°C and then cool in preheated (300°C) sand or bubbled alumina to room temperature.

A typical cut section of a Cr-5W alloy extrusion and another section double clad with the loose outer jacket of mild steel box section is shown in Figure 5. The rolling direction is transverse to the axis of extrusion and parallel to the long axis of the box section, i.e., open ends of the mild steel clad are the nose and tail of the rolled piece.

As can be seen in Figures 4 and 5, the interface between the molybdenum cladding and the Cr-5W alloy in the extruded condition is very irregular and deeply striated as is typical of some clad extrusions. It was primarily for this reason that the extruded alloy was cross-rolled rather than rolled parallel to the extrusion direction. It was found that cross-rolling effectively leveled the striations on finished strip and plate to the extent that additional surface finishing was not necessary. Straight-rolled material retained these striations essentially unchanged and would have required on the order of 0.020 in. removal of material on each surface in order to obtain a reasonable surface finish. In addition, the molybdenum cladding on the nose and tail (original extrusion edges) of a cross-rolled sheet bar remained quite ductile throughout the rolling process and thus afforded additional protection against cracking from insufficient rolling reduction, i.e., "alligatoring", and from incipient cracks in the nose and tail of the Cr-5W alloy. Although an early and limited attempt was made to roll the Cr-5W alloy without the molybdenum cladding, this resulted in an "alligator" type of crack failure; presumably caused by the stresses generated from an insufficient reduction in thickness for each pass. While the reductions taken were on the order of 20%, it is possible that heavier reductions would have been more satisfactory. Due to limitations in roll diameter and the ability to "bite" the sheet bar, larger reductions could not be obtained.

The rolling process developed and used throughout this program is outlined in Table 6. As can be seen from this table, rolling temperatures decrease in stages from 1050°C to 900°C in order to avoid recrystallization during rolling. Since these temperatures are consistently from 100°C to 150°C below the start of recrystallization temperature for this alloy, all rolled structures are wrought. Although the combined initial cladding thickness for steel and molybdenum was on the order of 0.175 in. per surface or 0.350 in. total, little difficulty was encountered in obtaining finished alloy thickness within the ±4% thickness tolerance. Alloy thickness within each extrusion was very uniform and only minor modifications in the rolling schedule were made for succeeding extrusions. Side spread during rolling from the extrusion thickness to the 0.062 in. final thickness was about 1/4 in. on a starting sheet bar width of 2-1/4 in.

The effect of thermal expansion mismatch between cladding and alloy was found to be more severe for 0.250 in. rolled alloy plate than for 0.062 in. alloy sheet. Since the thickness of molybdenum cladding on the extreme nose and tail of rolled alloy plate is increased by rolling, the finished alloy strip tends to exhibit some nose and tail cracks from the large thermal contraction stresses in these areas. While the depth of cracking is approximately the same for 0.062 in. and 0.250 in. thick product, i.e., 1 to 2 inches, the percentage losses on 0.250 in. plate are much higher. To solve this problem, the molybdenum cladding on the nose and tail of all extrusion sections rolled to 0.250 in. thick was reduced from approximately 0.100 in. thick to 0.020 in. thick by bandsawing off the extra molybdenum. This practice alleviated the thermal contraction stresses to the extent that all cracking from this source was eliminated.

From the last ingot processed, heat No. 84-100, approximately 4 lbs. of 0.027 in. thick alloy strip were produced. This was accomplished by the addition of five more passes of 20% reduction each at 900°C to the rolling schedule of Table 6. No difficulty was encountered in rolling or finishing this thinner gauge material.

As can be seen from Table 4, several heats of alloy, numbers 56-100, 58-100, 70-100 and 74-100, have higher carbon and oxygen contents and lower yttrium contents than the remaining ingots. All of the first three heats listed above showed to a varying degree a tendency towards edge cracking during rolling. Of these, heat No. 56-100, which was later rejected because of chemistry, had the poorest fabricability. Only four to five lbs. of sound material was obtained from the whole extrusion. The effect was minor for heats No. 58-100 and 70-100 and the process yields were affected only slightly. It was because of this adverse experience that heat No. 74-100, with a similar ingot chemistry, was not extruded or processed further.

As can be seen from Table 7, the average ductile-brittle-bend-transition temperatures of ingots No. 56-100, 58-100, and 70-100 are higher than all other heats measured. These temperatures are, however, substantially below the temperatures used for rolling. Since chromium is strain-rate sensitive(1), it is probable that, under the extremely high strain rates used for rolling, the transition temperatures of the three subject heats were increased to the point where brittle behavior was generated during rolling. It is likely that in a chromium alloy with reactive metal additions, such as Ti, Zr, Hf, or Ta, oxygen and carbon levels of kind reported here would not be harmful during processing.

5. Cleaning

After cooling to room temperature, the rolled sections were immersed for several hours in a room temperature solution of 60 v/o H_2O, 15 v/o HNO_3 and 15 v/o H_2SO_4 to remove the steel and molybdenum cladding. It was found that the Cr-5W alloy and the oxide of this alloy which formed on the bare edges of the rolled strip were extremely resistant to attack by any inorganic acids, and that the clad alloy could be immersed in any acid or mixture of acids, including HF, without any danger of encountering alloy removal.

Using X-ray fluorescence techniques, it was observed that the surfaces of the rolled and acid cleaned Cr-5W alloy strip contained a significant amount of molybdenum from the cladding. Since this contamination would have probably had an adverse or complicating effect on any subsequent coating development program using this material, it was necessary to remove the molybdenum contamination. This was accomplished by electroetching the rolled and acid cleaned alloy strip in a saturated oxalic acid solution using a D.C. current density of one to two amps/in.^{2} at about 11 volts. The removal of 0.0005 in. of alloy per surface was accomplished in approximately 2.5 minutes under these conditions and the molybdenum content was reduced by this treatment to the amount present in cast ingot. The electroetching operation also enlarged any cracks present in the strip such that they were easily visible and facilitated inspection of the material prior to cutting.

After electroetching, the Cr-5W sections were cut to finish size using 1/16 in. wide SiC abrasive cutting wheels with a liberal flow of cooling water. Figures 6 and 7 show typical examples of Cr-5W alloy finished to 0.062 in. and 0.250 in. thick, respectively.

6. Inspection and Evaluation

Drill cuttings were obtained from finished strip from each heat and submitted for chemical analysis. The values obtained, in conjunction with the ingot analyses are shown on Table 4.

As can be seen from Table 4, there is considerable variation between ingot and product analyses, especially for yttrium, oxygen, carbon, and nitrogen determinations. While a good portion of this may be due to within ingot variation and/or segregation, it is likely that a significant part is due to a lack of analytical development and experience with chromium base alloys. In future production work of this type multiple determinations would be helpful in minimizing variation from both sources.

In contrast to the target objectives of Table 1, all chemistry objectives with the exception of yttrium and, to a lesser extent tungsten, were maintained. The actual product tungsten values varied from 4.72 w/o to 5.20 w/o; very close to the contract requirement of 4.8 w/o to 5.2 w/o. Product yttrium values showed considerably more variation; ranging from 0.03 w/o to 0.18 w/o as compared to the contract requirement of 0.04 w/o to 0.07 w/o. It is concluded that this range in yttrium content is characteristic of the process used and that a new innovation in melt technique or melting method would be required to obtain closer yttrium control.

The molybdenum and steel cladding used during processing were completely effective in eliminating atmospheric surface contamination on the Cr-5W alloy strip. The chemical analyses of Table 4 show no oxygen or nitrogen contamination trends from ingot to product. In addition, knoop hardness traverses were obtained across the thickness of recrystallized 0.062 in. strip rolled from each heat. Figure 8 shows a typical set of knoop hardness traverses. No trend toward surface hardening from contamination could be observed from these tests.

Table 7 is a summary of the ductile-brittle-bend transition temperature test results obtained on rolled 0.062 in. strip from all heats processed in this program. Only the transverse tests were performed during the manufacturing program covered in this section of the report. The longitudinal results were developed during the second program (NAS 3-7919), and a discussion of all results is included in the report of that work, under Task III. Individual test results are given in the Appendix, Tables I to XII. In contrast to the contract requirements listed in Table I, a consistently low ductile-brittle transition temperature was not achieved and the maximum 200°F variation was exceeded.

During the initial stages of manufacturing, the contract requirements of Table 1 for length and width of sheet and plate were not achieved. However, once all problems of cracking were resolved, it was found that with careful rolling practice and a sound sheet bar, the length and width requirements on sheet and the width requirements on plate could be readily achieved or exceeded. Due to the limited width of extruded sheet bar and the necessity for cross-rolling, the maximum length obtainable for plate was on the order of 9 in. The surface finish requirements of 80 micro-inches, RMS, were attained for all 0.062 in. strip without surface conditioning. Because of the lower percent reduction in rolling and the roughness of the alloy-molybdenum interface in the extrusion, the surface roughness of 0.250 in. plate was higher than 80 micro-inches, but less than 125 micro-inches, RMS.

No appreciable difficulty was encountered in maintaining the contract tolerances for thickness, width, length and flatness.

E. PROCESS YIELDS

Tables 8 and 9 show in summary the actual product yields obtained in this program and the source of processing losses.

The overall yield for typical heats of 26.4% is probably a maximum for the process of this program and could only be improved upon by further work on the two major sources of loss, the ingot pipe and finished product cutting trim. Of these two, the percentage of cutting trim losses could be rapidly reduced merely by increasing the width of sheet bar for rolling. While other problems, especially with equipment and the thermal expansion mismatch could be encountered in this approach, the benefits would probably outweigh the development effort required. It is clear that the problem of ingot shrinkage pipe will require more serious attention if total process efficiency is ever to exceed 50%. One effort made to solve this problem was attempted in the form of heat No. 74-100. The charge for this heat was composed exclusively of hot tops, i.e., shrinkage pipes, from previous heats. While this attempt was not successful, this approach still appears to be technically feasible and definitely warrants further consideration and effort.

Of the 194.15 lbs. of Cr-5W alloy produced, a total of 184 lbs. were delivered to NASA. This quantity was composed of 51.5 lbs. of 0.250 in. plate, 128.7 lbs. of 0.062 in sheet, and 3.8 lbs. of 0.027 in. sheet.

IV MANUFACTURING DATA ON Cr-5W ALLOY SHEET (NAS 3-7919)

A. BACKGROUND AND PROGRAM ORGANIZATION

Based on the experience obtained in processing the first several heats of Cr-5W alloy produced in the manufacturing program discussed above, it was apparent that further work would be necessary in order to completely isolate the source of some of the cracking problems encountered in rolling, obtain an optimum rolling schedule, and identify better the effect of alloy chemistry on fabricability and/or transition temperature. Since the effect of warm work was being used in an attempt to achieve a lowered transition temperature in the rolled product, the matallurgical properties of interest in a successful rolling schedule were the recrystallization temperature, the transition temperature and the presence or absence of strain aging. The presence of a strain aging peak from interstitial contaminants has been reported for pure chromium at about 500°C[2] and it has been observed [3] that warm working at or above this peak results in no improvement in transition temperature with increasing warm work. Thus to produce a low transition temperature in pure chromium, initial warm working should be done well above 500°C to depress the transition temperature to below 500°C. Further warm work is done below 500°C to insure a further depression in the transition temperature. It was assumed that interstitial contaminants could play a similar role in the Cr-5W alloy, and might contribute to the high transition temperatures observed.

In summary, then, the factors selected for further investigation can be enumerated as follows:

1. The effect of warm work on the recrystallization temperature.
2. The effect of warm work on the ductile-brittle transition temperature.
3. The temperature for strain aging, if any is present.
4. The effect of interstitial contaminants on the ductile-brittle transition temperature.

The experimental program to evaluate the above relationships is divided into three tasks. Task I is directed toward obtaining baseline data on the transition temperature of wrought and recrystallized Cr-5W alloy, measuring the effect of strain aging, and determining the effect of warm work on the recrystallization and transition temperatures of Cr-5W. Task II and Task III consist of determining the heat-to-heat variation in strength properties and bend transition temperature, respectively, for the Cr-5W alloy previously manufactured and attempting to correlate these properties to alloy chemistry.

B. TECHNICAL APPROACH

1. Task I

Although a bend test is frequently used to determine the transition temperature of metals, the results obtained are strongly dependent on test geometry and are subject to substantial error. In addition, a bend test is not likely to yield data sufficiently accurate to identify a strain aging peak. For these reasons, it was decided to use the tensile test as the criteria for transition temperature and to determine strain aging.

To measure the effect of warm work on transition and recrystallization temperature, warm work reductions of 0%, 30%, 50%, 70%, 90%, and 93% were selected for investigation. For the purposes of this work, it was decided to simulate the 0% reduction or extruded sheet bar condition by using recrystallized 0.062 in. thick material. To obtain the other reductions, Cr-5W alloy extrusion sections were to be rolled to the required thickness by the standard practice of Table 6. The Cr-5W alloy thicknesses corresponding to these reductions are as follows:

1) 0% - recrystallized 0.062 in. alloy
2) 30% - 0.630 in. thick
3) 50% - 0.448 in. thick
4) 70% - 0.270 in. thick
5) 90% - 0.090 in. thick
6) 93% - 0.062 in. thick

To define end points in the effect of warm work on transition temperature, such that the testing of intermediate reductions would be minimized, and to insure accuracy in the detection of any strain aging peak, the 0% and 93% reduction conditions were selected for tensile testing across the total range of room temperature to 1100°C. Then based on these data, the intermediate reduction conditions would only be tensile tested at temperatures spanning the suspected location of the transition temperature.

To determine the effect of warm work on recrystallization temperature, small coupons cut from the 30% to 93% reduction samples were exposed for 15 minutes at temperatures in the range of 900°C to 1200°C and evaluated metallographically and by hardness tests for evidence of recrystallization. Since normal reheat times for fabrication or rolling work are usually small, the purpose of short time exposure was to provide data useful in fabrication.

All the above data served to identify the effect of warm work on transition and recrystallization temperatures and locate the presence of any strain aging. To assure homogeneous data, it was planned to obtain all the reduction conditions from one typical heat of the Cr-5W alloy. This arrangement was later modified somewhat by the addition of a 97%, or 0.027 in. thick reduction from another heat. However, the investigation of this condition was limited to determining tensile transition and recrystallization temperatures.

2. Task II

To obtain a more accurate estimate of the effect of interstitial contaminants on the mechanical properties of Cr-5W alloy, it would have been desirable to prepare samples of alloy with deliberate additions of contaminants. Lacking this material, the next best approach was to test alloy from all heats produced and attempt to correlate these results with the natural variation in contaminant levels encountered on these melts. Since the nine heats processed contained a substantial variation in interstitial content, it was anticipated that some effect on mechanical properties could be observed. To detect effects on alloy strength, it was planned to obtain tensile tests on 0.062 in. wrought material from each heat at a standard test temperature to be determined from the data of Task I. By virtue of the careful control of the rolling schedule for all material produced, it was considered likely that lot-to-lot variations in the degree of warm work would be minor and would not adversely affect a correlation between strength properties and the level of interstitial impurities. A subsequent addition to the work of this task was to perform tensile tests at the same temperature on 97% reduced, i.e., 0.027 in. thick alloy material.

3. Task III

Similar to task II, it was desired to obtain a correlation between transition temperature and the level of interstitial contaminants. Since variation in transition temperature, rather than the value of transition temperature was the attribute of interest, it was concluded that bend testing would be a satisfactory and more economical method of measurement. Especially since transverse bend test data for all heats were already determined during the prior manufacturing program. Thus it was planned to obtain longitudinal bend transition temperature measurements on 0.062 in. wrought alloy strip from all available heats, combine this data with the previously obtained transverse transition temperature measurements and attempt to correlate transition temperature to interstitial content. A later supplemental addition to this work was to determine the longitudinal and transverse bend transition temperature of 97% reduced, i.e., 0.027 in. thick alloy strip.

C. EXPERIMENTAL RESULTS

1. Task I

For the work of this task all Cr-5W alloy material was obtained from heat No. 83-100 except for the 0.027 in. thick strip which was obtained from heat No. 84-100. Rolling of the various reductions was accomplished in accordance with the rolling schedule and process of Table 6. Tensile specimens for the various thicknesses of alloy were prepared to meet the dimensional requirements of Figures 27 and 28. Sheet specimens of the type of Figure 27 were prepared by electrical discharge machining, while round specimens of the type of Figure 28 were lathe machined to size. All specimens were cut parallel to the rolling direction. After machining, all specimens were electroetched in a saturated oxalic acid solution and then were inspected for flaws using dye penetrant techniques. Recrystallized tensile specimens were prepared by vacuum annealing for 1 hour at 1050°C in a vacuum of less than 1 x 10^{-5} torr. The tensile test gauge length was 1/2 in. and the specimens were tested at a crosshead speed of 0.0025 in./min. to 0.6% offset and a crosshead speed of 0.025 in./min. after 0.6% offset was reached. Tests at room temperature were conducted in air, while tests up to 250°C were performed in a silicone oil bath. Above 250°C tensile tests were obtained in a furnace under a vacuum equal to or less than 1 x 10^{-4} torr. Annealing studies to determine recrystallization temperature were performed with alloy coupons at least 1/4 in. x 1/4 in. x thickness in size, in a muffle furnace with a hydrogen atmosphere. The time at temperature for annealing was 15 ± 1 minutes; and due to the size of the specimens, the heating and cooling times were essentially negligible. After annealing, the coupons were cut and ground to remove surface contamination. Six to eight room temperature hardness readings were obtained for each coupon using the Rockwell 30N scale; the individual values being averaged to obtain annealed hardnesses. Metallographic specimens were prepared parallel to the rolling direction by conventional techniques; the final etch being performed electrolytically in a saturated oxalic acid solution.

Figures 9 and 10 and Tables 10 and 11 summarize and recapitulate the tensile test results obtained on 0.062 in. wrought and recyrstallized Cr-5W alloy tested across the temperature range of room temperature to 1100°C. For maximum accuracy, replicate specimens were tested at 100°C intervals throughout this temperature range. As can be seen from Figures 9 and 10, both recrystallized and wrought material shows slight elongation minima and yield and ultimate strength maxima at about 700°C. In addition, the 700°C tensile test, stress-strain chart records for recrystallized material show slight discontinuous yielding serrations characteristic of a strain aging peak. However, neither of the effects noted were sufficiently pronounced to conclusively establish a strain aging peak at this temperature. It is, however, concluded that for Cr-5W alloy with total interstitial impurities similar to that contained in heat No. 83-100, i.e., 100 to 150 ppm, strain aging is not present to the extent that it would be a factor in any conventional warm working process.

The tensile strengths and ductility obtained in both the wrought and recrystallized 0.062 in. material of Figures 9 and 10 compare well with values previously reported[4] for similar alloys. As can be seen, the tensile transition temperatures of the wrought and recrystallized alloy are approximately 100°C and 175°C, respectively. Although this value for wrought material agrees closely with the longitudinal bend transition temperature for this heat (No. 83-100) as reported in Table 7, the value for recrystallized material was surprisingly low. Since it is known that the transition temperature of chromium is sensitive to strain rate(1), it was decided to verify the recrystallized tensile transition temperature value by further tests at a higher strain rate. To do this simply and quickly, longitudinal specimens of 0.062 in. Cr-5W alloy from heat No. 83-100 were vacuum furnace recrystallized at 1050°C for 1 hour. From this material the bend transition temperature was determined in accordance with the standard bend test procedure of this program. The ram speed in this test is 1 in./min., thus providing several orders of magnitude higher strain rate than performed in the previous tensile tests. As can be seen from Table 7, the bend transition temperature of 100°C is even lower than that obtained by tensile testing. Thus it was established that the low tensile transition temperature measured for recrystallized alloy was not necessarily caused by a low testing strain rate and is apparently valid. It has been previously reported, however, that the ductile-brittle transition temperature of chromium should decrease with decreasing grain size(5). This effect has also been noted for other BCC metals. Since the vacuum-furnace-recrystallized 0.062 in. thick alloy material has a grain size of approximately 1500 gr/mm^2 compared to a grain size in the extrusion of 128 gr/mm^2, it is reasonable to conclude that refinement in recrystallized grain size has contributed to the low transition temperature observed. Thus, using the recrystallized 0.062 in. material to simulate extruded Cr-5W alloy was not successful and these recrystallized data do not give a true indication of the transition temperature of extruded alloy.

Another interesting effect observed in the tensile test data on recrystallized 0.062 in. thick alloy is the retention of significantly large amounts of residual plastic ductility at temperatures below the transition temperature. As can be seen from Figure 10 and Table 11, the recrystallized alloy shows approximately 6% tensile elongation at temperatures as low as 20°C. In a similar manner, the bend tests on 0.062 in. recrystallized alloy as summarized in the Appendix, Table IX, also clearly show appreciable plastic deformation at temperatures below the transition temperature. Although this behavior is not particularly unusual, the amount of ductility retained is quite substantial. The trend of the above data indicates that purely brittle failure would not occur until temperatures well below room temperature were reached.

To measure the effect of warm work on the ductile-brittle transition temperature, the alloy material with rolling reductions of 30%, 50%, 70%, 90%, and 97% was tensile tested at temperatures ranging from 20°C to 400°C. The data obtained from this work is summarized on Table 12 and in Figures 25 and 26. For completeness and continuity, the low-temperature tensile data previously described for wrought 0.062 in. thick alloy, i.e., 93% reduced condition, has been repeated on this table and these figures. As was expected, it was found that increasing amounts of warm work increase strength and lower the ductile-brittle transition temperature. The tensile transition temperatures corresponding to the warm work conditions evaluated are approximately as follows:

% Reduction and Thickness	Transition Temperature °C
30%/0.630 in.	350
50%/0.448 in.	350
70%/0.270 in.	185
90%/0.090 in.	125
93%/0.062 in.	100
97%/0.027 in.	150

Based on these data, it is confirmed that the 0.062 in. recrystallized tensile results did not accurately simulate and measure the transition temperature of alloy in the extruded condition. However, from the trend exhibited above, it is estimated that this transition temperature would be approximately 400°C. In addition, it should be noted that these values are typical of an alloy heat with reasonably low interstitial impurity content. Based on the close agreement between longitudinal bend and tensile transition temperature measurements for this heat, and the higher interstitial impurity levels and wrought bend transition temperatures observed in other heats as shown on Tables 4 and 7, it is probable that other heats with poorer chemistry had much higher transition temperatures in the extruded condition and throughout the rolling process. This factor combined with the strain rate sensitivity of chromium and the high strain rates used during rolling may entirely account for the clearly brittle behavior observed during the rolling of certain heats, particularly No. 56-100.

From the 90%, 93% and 97% warm work transition temperature data described above, it would appear that warm work in excess of 93% can cause a rise in the transition temperature. While this effect is not unusual, or unexpected, the data is not homogeneous or conclusive since the 97% condition was obtained from another heat, i.e., No. 84-100. To assess this effect further, longitudinal bend transition temperature measurements were performed on 90% warm worked alloy from heat No. 83-100. Then recapitulating the available bend test results for heats No. 83-100 and 84-100 as a function of warm work, the following is obtained:

Longitudinal Bend Transition Temperature °C
(from Table 7)

	Heat No. 83-100	Heat No. 84-100
90%/0.090 in.	350	-
93%/0.062 in.	100	200
97%/0.027 in.	-	275

While the above data do not reflect effects from texture or preferred orientation, i.e., transverse transiverse transition temperature, it appears that excessive warm work will increase the transition temperature. In addition, note in Table 12 that the tensile ductility above the transition temperature for the 97% condition (4%), is less than that for the 93% worked condition (10%). It is thus possible that excessive warm work may reduce ductility.

Table 13 and Figures 11 to 24, inclusive, summarize the effect of warm work on recrystallization temperature and annealed hardness in the Cr-5W alloy. As expected, the recrystallization temperature, as defined by 1% to 10% area percent of strain-free material in the alloy microstructure, decreases regularly with increasing warm work as follows:

% Reduction and Thickness	Recrystallization Temperature (15 min.) (°C)
30%/0.630 in.	1150
50%/0.448 in.	1125
70%/0.270 in.	1075
90%/0.090 in.	1050
93%/0.062 in.	1025
97%/0.027 in.	1025

While the decrease in temperature is not as great as has been observed in molybdenum and tungsten, the effect should be taken into consideration in any warm work processing schedule. Normally, a conservative and safe practice is based on reheat temperatures at least 50°C below the start of recrystallization. As can be seen from Table 6, the rolling process of this program readily meets this criteria. Possibly, all the rolling reheat temperatures could be shifted upward by 50°C to achieve maximum ductility during rolling.

In summary, the work of Task I did yield data on the Cr-5W alloy useful in defining a rolling schedule based on warm work and in evaluating the effect of strain aging in such a rolling process. While little if any significant strain aging effects were observed in the alloy heat investigated, the upper and lower temperature limits for warm working an initially recrystallized alloy structure were adequately defined. Although these data would indicate that the temperature span of ductility useful for rolling is on the order of 750°C to 900°C, i.e., the difference between transition and recrystallization temperature, the transition temperature can probably be elevated appreciably by variations in alloy interstitial content and the inherent strain rate sensitivity of chromium. It is likely that these two factors could reduce the temperature span of ductility to 300°C to 400°C. Thus a conservative warm working process should use temperatures as close to the recrystallization temperature as it is possible to control. Finally, very limited data indicates that a decrease in initial recrystallized grain size results in a lowering of the ductile-brittle transition temperature. On the basis of this information, it is considered probable that an intermediate recrystallization which refined the alloy grain size during the warm working process, might result in a wrought product with a transition temperature well below ambient. Furthermore, typical manufacturing processes for molybdenum and tungsten also utilize a final stress-relief anneal which greatly enhances the ductility of finished wrought sheet. Certainly, this technique also is worthy of investigation.

2. Task II

For the work of this task, two tensile specimens, meeting the dimensional requirements of Figure 27, were prepared from 0.062 in. thick wrought Cr-5W alloy from eight heats of alloy produced during the manufacturing program. Two additional specimens were prepared from

0.027 in. thick wrought alloy from heat No. 84-100. All specimens were cut parallel to the rolling direction and were prepared and tested by the same procedures as previously outlined in Task I. Since it was the objective of this phase to attempt to correlate tensile properties with the natural heat-to-heat variation in interstitial impurity levels, it was important that accurate tensile data be obtained. To avoid the test error and scatter associated with a tensile test performed on a material in the brittle condition, it was decided to conduct all tests at a temperature which would yield ductile behavior for all heats. Accordingly, based on the tensile data of Task I and the transverse bend transition temperature measurements for all heats, a test temperature of 540°C (1000°F) was selected.

Table 14 summarizes the tensile test results for the eight heats of wrought 0.062 in. thick Cr-5W alloy and for the 0.027 in. thick material from one heat. Ingot No. 47-100 was not included since strip from this heat was not available. As expected, the test temperature of 540°C was above the transition temperature for all heats and substantial elongation was obtained on all tests. Interestingly enough, the higher degree of warm work present in the 0.027 in. thick alloy from heat No. 84-100 did not result in an improvement in strength and ductility over that measured for 0.062 in. thick material from the same heat. This would tend to confirm the observation discussed previously in Task I that the 97% or 0.027 in. thick alloy condition contains excessive warm work.

The data of Table 14 was compared to the chemistry results of Table 4 in an attempt to correlate interstitial impurity levels to mechanical properties. To avoid introducing variation from errors in the tensile test procedure into this correlation, only the highest yield and ultimate strength values from each heat were utilized. A correlation with tensile elongation was not attempted since the between-heat variation in ductility was essentially negligible. In order to accurately assess the degree of correlation achieved and facilitate the work, a short computer program was employed. This program performed a least-squares fit of the data to six curve types and calculated the index of correlation for each curve equation. The index of correlation was used as a criteria for determining whether any correlation existed. Because of the limited number of data points available, i.e., very few degrees of freedom, it was assumed that a correlation was not significant unless the index of correlation for the least squares fit was above 75%.

Using the above procedure, comparisons were made between yield strength and ultimate strength versus carbon, oxygen and nitrogen content in ingot and product and all possible combinations of these impurities. As a result of this work, the only significant correlation determined was between tensile yield strength and the product carbon plus oxygen impurity levels. Even this correlation was efficient for only seven heats and required the exclusion of data from heat No. 56-100 in order to exceed the 75% index of correlation criteria. The data points for this comparison are summarized on Table 15. No correlation was found for ultimate tensile strength or for nitrogen content. Since the nitrogen content showed little variation between heats, this latter result is not unexpected.

While the correlation between tensile yield strength and carbon plus oxygen content appears tenuous, it is not likely that this relationship is purely accidental. In view of the low number of data points comprising the correlation and the unknown quantity of sample variance present in the chemistry data, the correlation could not be expected to be much better. In short, it is concluded that some correlation definitely exists between interstitial oxygen and carbon content and yield strength in the Cr-5W alloy, but a more rigorous experimental approach would be required in order to obtain a quantitative assessment of the degree of correlation.

3. Task III

In the work of this task, longitudinal bend transition temperature measurements were obtained on 0.062 in. wrought alloy strip, from all available heats. In addition, longitudinal and transverse bend transition temperature measurements were performed on wrought 0.027 in. material from heat No. 84-100. Then these data were combined with the transverse transition temperature measurements obtained from the previous manufacturing program and compared to the heat chemistry results in an attempt to realize a correlation between transition temperature and interstitial impurity levels.

All bend tests performed in both programs were conducted in accordance with paragraph 5 of MAB-192-M "Evaluation Test Methods for Refractory Metal Sheet Material", except that the specimen size and test span for all tests was fixed at 0.500 in. x 2.0 in. x thickness and 1.25 in., respectively. The bend or punch radius used was four times the specimen thickness (4t) and the longitudinal edges of the specimens were polished and electroetched in a saturated solution of oxalic acid prior to test. Edge radii from polishing were about 0.010 in. Bend tests were performed in an air convection oven with thermocouple temperature measurements of both the air atmosphere and test fixture. Throughout both programs, the transition temperature was defined as the minimum temperature at which a full 90° or greater bend angle could be obtained without specimen cracking or breakage.

All bend transition temperature determinations from both programs are reported in Table 7, while individual test results are summarized in the Appendix, Tables I through XII. Specimen failure invariably occurred by cracking on the tensile side of the specimen with the crack running roughly parallel to the axis of the bend and the thickness of the specimen. Frequently, the crack showed some minor offsetting at the neutral axis of the specimen. Specimen failure by delamination was not observed in any tests.

Using the same computer method previously discussed in Task II, the combined longitudinal and transverse bend transition temperature data of Table 7 were compared to the chemistry results of Table 4 in an attempt to correlate interstitial impurity levels to transition temperature. Although no correlations were found which had an index of correlation in excess of 75%, a lower but consistent degree of correlation occurred repeatedly for seven out of eight heats between transition temperature and oxygen content. For examining this relationship further, Table 16 recapitulates transition temperature and alloy product oxygen content data ordered by increasing average transition temperature. While the correlation is weak, previously described observations of the effect of oxygen on sheet rolling and tensile strength tend to support the presence of some degree of correlation. Although there is work reported in the literature[6] which indicates that rather high levels of oxygen do not affect the transition temperature of pure chromium, this data may not apply to the Cr-5W alloy. Thus, while it cannot be concluded from the work of this program that there is a proven relationship between transition temperature and oxygen, carbon and/or nitrogen content, there are several indications that oxygen may affect transition temperature.

As can be seen from Table 7 for ΔT, there is a wide variation between heats in the degree of anisotropy or texture present in the wrought alloy sheet. In the case of two wrought conditions listed, i.e., heat No. 83-100 and 0.027 in. alloy from heat No. 84-100, the anisotropy was so large that the transverse transition temperature could not be accurately determined. There does not appear to be any pattern to the degree of anisotropy or relationship to any other factor and it is conluded the reason for this wide variation is not determinable from the data available. It is apparent, however, that in any future work with wrought alloys of this type, bend tests and/or mechanical property tests should be obtained in both directions in order to be meaningful.

V CONCLUSIONS

A. CONTRACT NAS 3-7901

1. Using previous developmental experience with chromium base alloys and a secondary fabrication process based on warm work, approximately 180 lbs. of Cr-5W alloy sheet and plate were successfully produced under a pilot production type of program. In the course of this work, it was found that several processing variables required development work in order to obtain maximum process yields. Problems with extrusion cracking were solved by stress-relief annealing and slow-cooling the extruded bar. The rolling process requires the minimization of thermal cycling, an outer cladding on the rolled sections and slow heating and cooling rates in order to avoid alloy cracking from thermal expansion mismatch between the alloy and the co-extruded molybdenum cladding. Finally, methods of cutting the alloy were selected which avoided cracking from thermal shock and mechanical vibration.

2. Although the Cr-5W alloy sheet and plate process demonstrated capability for fulfilling the bulk of the original specification requirements for product chemistry, mechanical properties and dimensions; certain requirements could not be met. These were: 1) alloy yttrium content, 2) a low and reproducible ductile-brittle-bend transition temperature and, 3) the length of plate product. Of these, the first two would clearly require further development work in order to obtain closer control.

B. CONTRACT NAS 3-7919

1. As an adjunct to the development work performed during the production program, a second program was conducted which had the objectives of defining the temperature limits for an optimum rolling process for Cr-5W alloy and of investigating the effect of interstitial contaminant levels on transition temperature and mechanical properties.

This work showed that the temperature schedule used for rolling Cr-5W alloy was essentially correct and that alloy of typical chemistry did not exhibit strain-aging to a degree which is detrimental in a warm working process. Secondly, it was found that the yield strength of wrought Cr-5W alloy at 540°C tends to increase with increasing oxygen plus carbon content. Finally, while there seems to be some relationship between ductile-brittle bend transition temperature and oxygen content, this correlation is not conclusive.

2. Some evidence was found that the transition temperature of recrystallized Cr-5W alloy decreases with decreasing grain size and that warm work in excess of 93% does not increase wrought strength or result in a further lowering of the transition temperature. Based on the first observation above, it is reasonable to expect that an intermediate recrystallization treatment to refine grain size during the warm working process could result in sub-ambient transition temperature in wrought alloy sheet of the Cr-5W type; and that this approach warrants further investigation.

3. Wrought Cr-5W alloy sheet exhibits a substantial and extremely variable anisotropy or texture in transition temperature.

4. The development of full-scale production processes for chromium base alloy sheet of the Cr-5W type will require further additional work in the following areas: 1) improvement of overall processing yields, 2) the control of alloy chemistry; particularly yttrium and interstitial content, and 3) reproducibility of analytical chemistry results.

VI ACKNOWLEDGMENTS

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TABLE I

A SUMMARY OF REQUIRED PROPERTIES FOR Cr-5W SHEET AND PLATE

A. CHEMICAL COMPOSITION

Element	Amount	Element	Amount
Tungsten	4.8 - 5.2 w/o	Sulfur	100 ppm max.
Yttrium	0.04-0.07 w/o	Phosphorus	50 ppm max.
Carbon	150 ppm max.	All Other	1000 ppm max.
Oxygen	150 ppm max.	Chromium	Balance
Nitrogen	150 ppm max.		

B. SIZE AND DIMENSIONAL REQUIREMENTS

1. Sheet - 0.062 in. thick x 2 in. to 3-1/2 in. wide x 12 in. long
2. Plate - 0.250 in. thick x 4 in. wide x 12 in. long
3. Tolerances - Thickness: ±4%
   - Width: ±1/32 in.
   - Flatness: 6% max.
   - Surface Finish: 80 micro-inches, RMS, maximum
4. Quantity Mix - 130 lbs. of 0.062 in. sheet and 50 lbs. of 0.250 in. plate.

C. METALLURGICAL REQUIREMENTS

1. Sheet and plate to be in worked condition.
2. No evidence of surface contamination as determined by Knoop hardness traverse across strip thickness.
3. A low transverse ductile-brittle bend-transition temperature with a maximum variation of not more than 200°F from the average temperature for all material.

TABLE 2

CHEMISTRY OF "ELCHROME HP" CHROMIUM

Element	Specification Limit - Wt. %	Vendor's Typical Analysis- Wt. %	Lot No. 37468 Heat No. 47-100	Lot No. 37550 Heat Nos. 56-100 57-100 58-100	Lot No. 37556 Heat No. 64-100	Lot No. 37560 Heat No. 67-100	Lot No. 37561 Heat No. 70-100	Lot No. 37570 Heat Nos. 83-100 84-100
C	0.008 Max.	0.006	0.008	0.007	0.005	<0.005	0.005	0.005
S	0.008 Max.	0.004	0.007	0.008	0.007	0.007	0.006	0.007
Si	0.02 Max.	0.009	0.008	<0.01	0.004	0.003	0.001	0.004
Fe	0.02 Max.	0.009	0.003	0.004	-	0.001	0.003	0.003
O	0.015 Max.	0.008	0.004	0.003	0.010	0.003	0.002	0.006
N	0.010 Max.	0.007	0.010	0.009	0.006	0.006	0.004	0.006
H	0.0005 Max.	0.0003	0.0003	<0.001	<0.001	<0.010	<0.001	<0.001
Cr	99.9 Min.	99.95	99.95+	99.95	99.94	99.95+	99.95+	99.95+

TABLE 3

NUCLEAR GRADE YTTRIUM SPONGE
VENDOR'S TYPICAL ANALYSIS

Element	PPM
O	100-150
N	0-10
H	0-10
C	50-150

OTHER ELEMENTS BY SPECTROGRAPHIC ANALYSIS - (PPM)

Gd	<1	Mg	<5	Na	<3
Dy	7	Al	6	Li	3
Yb	<100	Ta	<20	Ba	10
Sm	10	Pb	<20	K	6
Eu	<5	Ni	10	Ti	<3
Lu	<5	Cu	30	Co	<1
Tm	<5	Ca	<10	Zn	<100
Tb	<100	Cr	60	Sn	<3
Ho	<100	V	<3	Zr	<20
Si	<10	Be	<10	Cd	<30
Fe	6	B	<3		
Mn	1	Cb	<30		

TABLE 4

CHEMICAL ANALYSES OF Cr-5W ALLOY PRODUCED FOR NAS 3-7901

Heat No.[1]		W w/o	Y w/o	C ppm	O ppm	N ppm	H ppm	S ppm	P ppm	Remarks
47-100	I	5.21	0.11	145	140	9	2	30	20	
	P	4.99	0.09	28	72	44	6	30	20	
56-100	I	4.87	0.08	100	172	33	2	50	20	Rejected for chemistry
	P	4.92	0.024	134	342	55	1	10	30	
57-100	I	4.82	0.085	90	88	30	2	40	20	
	P	4.72	0.140	58	94	57	4	30	50	
58-100	I	4.78	0.054	80	136	49	1	40	20	
	P	4.86	0.070	79	82	44	1	20	<10	
64-100	I	4.86	0.120	20	116	23	2	20	50	
	P	4.82	0.110	80	49	35	8	50	10	
67-100	I	4.83	0.11	80	90	17	1	40	<10	
	P	4.90	0.12	<10	41	30	5	70	20	
70-100	I	4.88	0.03	40	112	7	1	30	10	
	P	5.20	0.04	42	119	20	1	50	<10	
74-100	I	5.10	0.01	75	278	57	1	40	20	Remelted scrap. Rejectable for chemistry and not processed further
	P	-	-	-	-	-	-	-	-	
83-100	I	4.98	0.19	16	48	42	6	20	<10	
	P	4.91	0.18	16	78	36	17	10	<10	
84-100	I	4.93	0.19	30	57	46	7	40	20	
	P	5.02	0.18	12	95	24	12	20	20	
Final Specification Limits		4.7 to 5.25	0.03 to 0.20	150 Max.	150 Max.	150 Max.	-	100 Max.	50 Max.	

(1) I - Ingot Analyses
P - Rolled Sheet or Plate Analyses

TABLE 5

EXTRUSION DATA FOR Cr-5W ALLOY INGOTS

Heat No.	Extrusion Temp. (°C)	Breakthrough Force (Tons)	"Steady-State" Force (Tons)	"K" Factor[1] (PSI)
47-100	1427	991	750	55,200
56-100	1430	1015	768	58,200
57-100	1434	1228+	905	70,500[2]
58-100	1425	844	556	48,100
64-100	1434	942	694	53,700
67-100	1423	1040	678	59,900
70-100	1410	1030	675	59,000
83-100	1426	1020	629	59,700
84-100	1430	1101	700	64,500

(1) From the equation

$$P = K \ln R$$

where:
- P = extrusion pressure (PSI)
- K = extrusion constant (PSI)
- R = extrusion ratio

(2) Billet extruded in tandem with a steel nose block heated to 850°C

TABLE 6

ROLLING AND FINISHING PROCESS FOR Cr-5W ALLOY SHEET AND PLATE

A. PREPARATION FOR ROLLING

1. Cut extrusion transversely into 2 in. to 4 in. wide sections.
2. Wet sand and electroetch cut faces and inspect for cracks.
3. Clad in loose, box section jackets of 0.125 in. thick mild steel.

B. ROLLING

1. Furnace heat in hydrogen atmosphere to 1050°C
2. Roll per the following schedule at 150 sfpm.

No. of Passes	Temp. °C	% Reduction per Pass	Reheat Time per Pass (min.)	Finish Alloy Thickness (in.)
1 thru 4	1050	20	7	-
5 thru 7	950	20	7	0.250
8 thru 12	900	20	7	0.062

3. Furnace cool to 300°C in hydrogen atmosphere, then cool to room temperature from bubbled alumina preheated to 200°C.

C. CLEANING

1. Remove steel and molybdenum cladding by pickling in a solution of 60 v/o H_2O, 15 v/o HNO_3 and 15 v/o H_2SO_4.
2. Electrolytically etch Cr-5W alloy strip or plate in a saturated oxalic acid solution using a D.C. current density of 1 to 2 amps/in.^{2} at 11 volts. Reduce strip thickness by at least 0.001 in.
3. Cut to finish length and width with wet abrasive saw.

D. INSPECTION AND EVALUATION

1. Inspect each piece for length, width, thickness and surface finish (RMS).
2. From each heat recrystallize sections of 0.062 in. rolled strip at 1050°C for 1 hr. Obtain knoop hardness traverses as an inspection for surface contamination.
3. From each heat, determine DBBTT transverse to the rolling direction on 0.062 in. rolled strip in accordance with the procedure of MAB 192M, paragraph 5.
4. Obtain complete chemical analysis for all required elements on samples of rolled alloy from each heat.

TABLE 7

SUMMARY OF DUCTILE-BRITTLE BEND TRANSITION TEMPERATURE(1)

Cr-5W ALLOY

Heat No.	Longitudinal to Rolling Direction °C	Transverse to Rolling Direction °C	Average °C	△ T (T-L) °C
47-100	-	475	-	-
56-100	300	530	415	230
57-100	200	450	325	250
58-100	475	575	525	100
64-100	225	250	237	25
67-100	150	325	237	175
70-100	350	425	387	75
83-100	100	500(2)	300	400
84-100	200	440	320	240
83-100 (Recrystallized 1 hr. @ 1050°C)	100	-	-	-
83-100 (0.090" thick)	350	-	-	-
84-100 (0.027" thick)	275	>600	-	-

(1) Minimum temperature at which a full 90° bend is obtained.

All material nominally 0.062 in. thick and warm worked unless otherwise noted.

Tests performed per MAB-192-M, with a bend radius of 4t, a bend span of 1.25 in., a specimen 0.500 in. wide x 2.0 in. long and a ram speed of 1 in./min.

(2) Based on only one successful bend test. See Appendix, Table VIII

TABLE 8

RECAPITULATION OF PROCESS YIELDS FOR Cr-5W ALLOY

Heat No.	Cast Ingot Weight (lbs.)	Extruded Net Weight (lbs.)	Finished Sheet and Plate Weight (lbs.)	
47-100	94.6	54.3	8.67	
56-100	93.3	55.6	0.00	Rejectable for chemistry
57-100	93.4	54.4	30.33	
58-100	91.8	58.9	24.81	
64-100	91.1	51.6	27.17	
67-100	92.75	49.9	24.50	
70-100	90.5	55.5	21.50	
74-100	90.1	0.00	0.00	Remelted scrap
83-100	-	54.3	28.14	
84-100	-	54.3	29.03	
TOTAL 10 HEATS			194.15	

Percent yield for all 10-100 lbs. heats	- 19.4%
Percent yield excluding heat No. 74-100	- 21.6%
Percent yield excluding heat Nos. 56-100 and 70-100	- 24.2%
Typical percent yield for representative heats (excludes 74-100, 56-100, and 74-100)	- 26.4%

TABLE 9

TYPICAL INGOT PROCESSING YIELDS

Cr-5W ALLOY(1)

Operation	Source of Loss	Loss (lbs.)	Yield (lbs.)
1. Charge		0	100
2. Melt	Crucible slag	8	92
3. Analysis	Analytical slice	1	91
4. Cut hot top	Ingot pipe	22	69
5. Machine ingot	Cold shuts and nose taper	15	54
6. Cut extrusion	Extrusion ends and saw kerf	4	50
7. Rolling	Edge and end trim	24	26

(1) Excludes ingot losses from rejectable chemistry

TABLE 10

TENSILE STRENGTH WARM WORKED 0.062 IN. Cr-5W ALLOY[(1)]

VERSUS TEMPERATURE

Test Temp. °C	0.2% Yield Str.Psi.	U.T.S. Psi	% Sample Elong. in 1 in.	Remarks
20	91,400	115,600	2.5	
	82,800	113,900	-	Piece missing after break
	98,100	108,700	1.0	
100	96,000	113,600	8.0	
200	95,500	105,300	10.0	
	93,000	105,300	10.0	
300	92,500	104,100	12.0	
	52,000	101,900	12.0	
	78,300	93,900	10.0	
400	54,600	93,800	10.0	
	85,700	97,300	10.0	
500	80,000	90,800	10.0	
	81,700	89,800	10.0	
600	80,700	82,300	10.0	
	69,600	82,800	10.0	
700	74,100	77,900	8.0	
	74,400	78,400	10.0	
800	67,300	72,700	10.0	
	68,400	75,700	11.0	
900	52,100	63,800	12.0	
	50,700	62,200	12.0	
1000	32,800	48,400	20.0	
	31,600	51,600	19.0	
1100	22,500	41,500	24.0	
	19,700	33,000	28.0	

(1) Heat No. 83-100, 0.062 in. thick, tested parallel to rolling direction

TABLE 11

TENSILE STRENGTH RECRYSTALLIZED 0.062 IN. Cr-5W ALLOY[1]

VERSUS TEMPERATURE

Test Temp. °C	0.2% Yield Str.Psi	U.T.S. Psi	% Sample Elong. in 1 in.	Remarks
20	12,300	61,900	8.0	
	33,800	59,000	4.0	
100	34,200	55,800	4.0	
	33,200	60,000	6.0	
200	29,200	60,800	36.0	
	28,600	61,200	19.0	
300	23,800	57,300	34.0	
	25,600	59,600	38.0	
400	23,400	53,900	38.0	
	20,700	54,100	36.0	
500	21,400	50,200	36.0	
	20,900	49,800	38.0	
600	21,300	45,500	34.0	
	20,100	45,500	34.0	
700	18,600	42,000	34.0	
	18,800	43,000	34.0	
800	16,400	39,100	36.0	
	17,300	38,800	36.0	
900	17,000	33,700	40.0	
	16,100	33,100	38.0	
1000	14,000	26,700	46.0	
	13,800	28,700	44.0	
1100	12,300	22,900	42.0	
	12,600	22,900	40.0	

(1) Heat No. 83-100, 0.062 in. thick, warm worked and recrystallized 1 hr. @ 1150°C. Approximate grain size after recrystallization ASTM 7-1/2 (approximately 1500 gr/mm^2). Tested parallel to rolling direction.

TABLE 12

TENSILE TRANSITION TEMPERATURE WARM WORKED Cr-5W ALLOY(1)

% Reduction and Thickness	Test Temp. °C	0.2% Yield Str. Psi.	U.T.S. Psi	% Elong.(2) in 1 in.	Remarks
30%/0.630 in.	20	-	62,900	0	
	200	60,600	61,100	0.25	
	250	59,500	67,700	1.45	
	300	57,000	64,400	1.60	
	400	52,000	60,700	7.3	
50%/0.448 in.	20	-	70,500	0.05	
	200	65,700	68,800	0.53	
	250	64,000	69,300	0.92	
	300	63,300	66,200	0.66	
	400	63,300	73,100	9.81	
70%/0.270 in.	20	-	85,300	0.14	
	125	80,400	80,400	0.19	
	175	80,000	89,000	2.08	
	200	77,200	86,300	7.14	
	300	71,900	80,600	7.13	
90%/0.090 in.	20	42,400	50,300	1.5	
	100	43,400	70,000	2.0	Broke in head.
	150	25,700	105,300	10.0	
93%/0.062 in.	20	91,400	115,600	2.5	
	20	82,800	113,900	-	Piece missing after break.
	20	98,100	108,700	1.0	
	100	96,000	113,600	8.0	
	200	95,500	105,300	10.0	
	200	93,000	105,300	10.0	
97%/0.027 (Heat No. 84-100)	20	113,600	119,400	1.0	
	100	-	84,400	1.0	
	200	84,500	103,400	4.0	
	300	77,600	101,000	4.0	
	400	78,000	90,100	4.0	

(1) All material from heat No. 83-100 except as noted.

(2) For 90, 93, and 97% reductions, % elongation measured from sample. For 30, 50, and 70% reductions, % elongation determined from tensile test chart.

TABLE 13

AVERAGE HARDNESS Cr-5W ALLOY VERSUS ANNEALING TEMPERATURE

ANNEAL TEMPERATURE °C

% Reduction	Warm Rolled	950	1000	1025	1050	1075	1100	1125	1150	1175	1200
30%	38.6	37.0	37.0	-	36.6	-	36.3	35.7	35.9	30.3	26.7
50%	39.9	39.4	40.4	-	39.3	-	37.8	38.0	32.4	26.5	-
70%	43.4	41.9	41.3	-	40.6	39.7	39.4	32.4	28.5	-	-
90%	45.1	42.9	41.9	42.1	40.4	38.5	33.7	27.6	-	-	-
93%	45.5	44.7	42.5	41.9	40.7	36.5	30.2	-	-	-	-
97%	47.7	45.6	44.1	41.7	40.9	34.9	25.0	-	-	-	-

NOTE: All hardness readings Rockwell 30N scale, "Brake" Indenter.
Average of 6 to 8 measurements for each condition.
Time at indicated anneal temperature - 15 minutes.
All test material obtained from heat No. 83-100 except for 97% reduced material obtained from heat No. 84-100.

TABLE 14

1000°F (540°C) TENSILE STRENGTH FOR EIGHT HEATS OF WARM WORKED 0.062 IN. Cr-5W ALLOY AND FOR ONE HEAT OF 0.027 IN. Cr-5W ALLOY

Heat No.	0.2% Yield Str.Psi.	U.T.S. Psi	% Sample Elong.in 1 in.	Remarks
56-100	82,700	88,200	12.0	
	83,500	91,700	8.0	
57-100	84,800	92,100	10.0	
	75,700	93,700	12.0	Yield irregular due to clamp slippage
58-100	81,300	90,900	11.0	Specimen undersize on width
	86,900	95,000	12.0	
64-100	80,100	85,500	11.0	
	73,500	86,900	10.0	
67-100	77,400	82,400	12.0	
	76,100	88,100	10.0	
70-100	88,500	95,900	10.0	
	-	-	-	Specimen damaged in testing
83-100	78,000	82,600	12.0	
	68,800	86,100	14.0	
84-100	79,300	87,500	-	Piece missing after break
	71,400	87,900	10.0	
AVERAGE	79,200	88,900	11.0	
RANGE	68,800-88,500	82,400-95,900	8.0-14.0	
84-100 (0.027 in. thick)	76,200	87,300	5.0	
	67,200	88,200	4.0	

TABLE 15

CORRELATION BETWEEN TENSILE YIELD STRENGTH(1) AND CARBON AND OXYGEN ANALYSES FOR EIGHT HEATS OF Cr-5W ALLOY

Heat No.	0.2% Yield Strength (Psi)	Product Analyses (PPM) Carbon	Oxygen	Carbon + Oxygen
67-100	77,400	<10	41	51
83-100	78,000	16	78	94
84-100	79,300	12	95	107
64-100	80,100	80	49	129
56-100(2)	83,500	134	342	476
57-100	84,800	58	94	152
58-100	86,900	79	82	161
70-100	88,500	42	119	161
INDEX OF(3) CORRELATION	-	37%	21%	76%(4)

(1) For 0.062 in. thick wrought specimens tested at 540°C (1000°F).

(2) Excluded from least squares fit, but listed here for information purposes.

(3) For linear regression of the form Y = A + BX where Y = yield strength and X = chemical analysis.

(4) For Y = A + BX where A = 70,000 Psi and B = 0.1.

TABLE 16

CORRELATION BETWEEN BEND TRANSITION TEMPERATURE AND OXYGEN ANALYSES FOR EIGHT HEATS OF Cr-5W ALLOY

Heat No.	Average DBBTT (°C)	Longitudinal DBBTT (°C)	Transverse DBBTT (°C)	Product Oxygen (ppm)
67-100	237	150	325	41
64-100	237	225	250	49
83-100	300	100	500	78
84-100	320	200	440	95
57-100	325	200	450	94
70-100	387	350	425	119
56-100	415	300	530	342
58-100	525	475	575	82

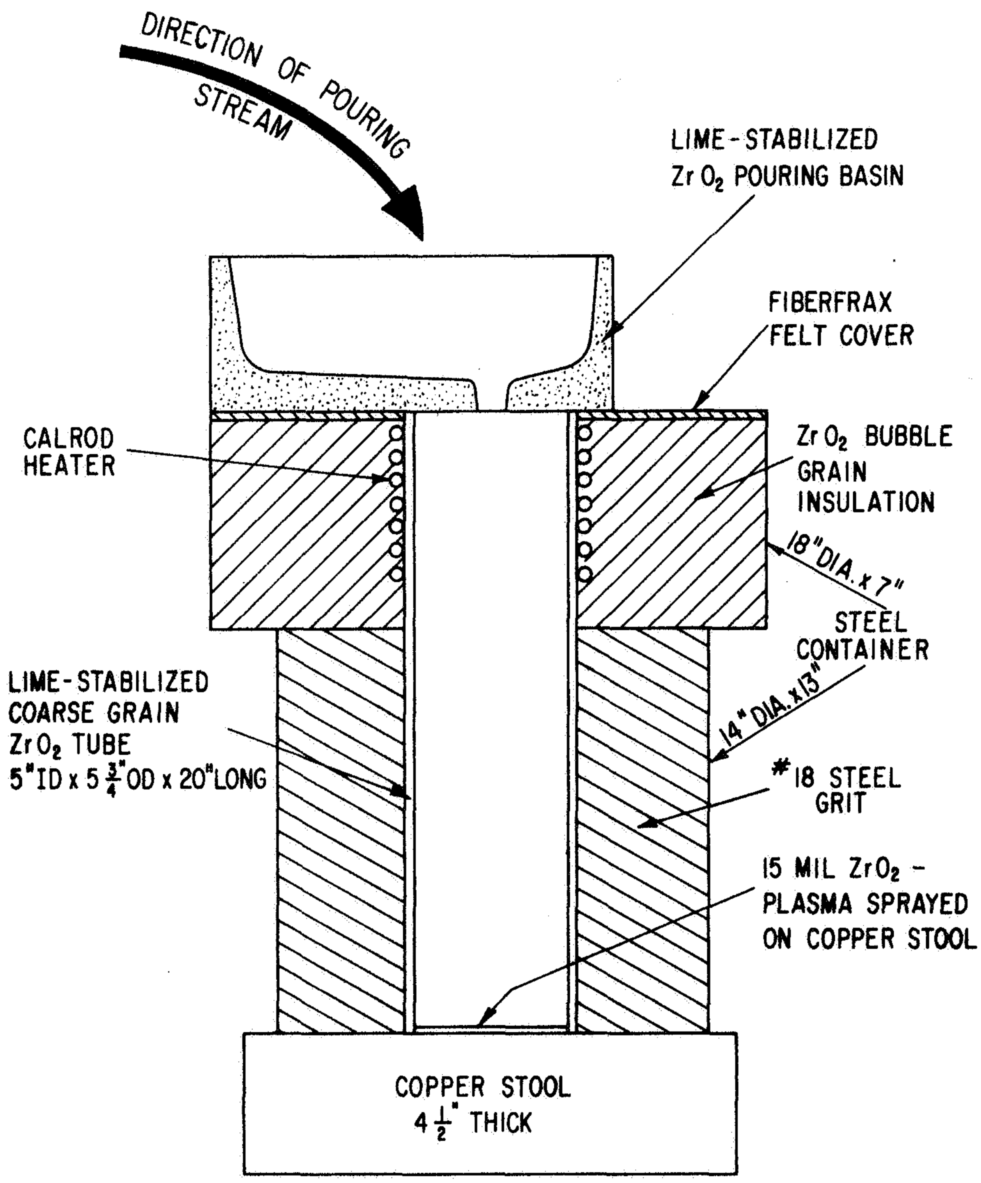


Figure 1: Schematic diagram of mold for casting 100 lb. ingots of chromium base alloy

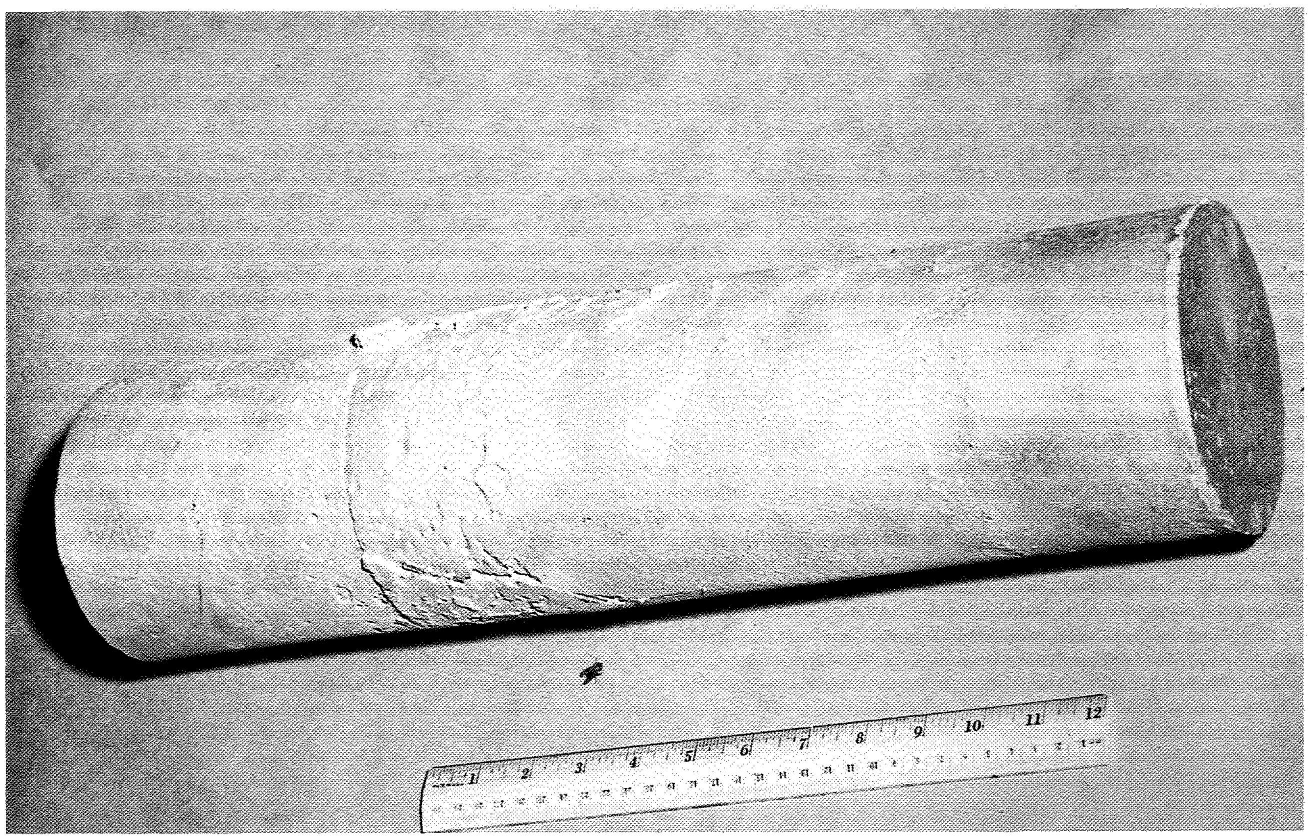


Figure 2: Typical 100 lb ingot of Cr-5W alloy in the cast and sandblasted condition

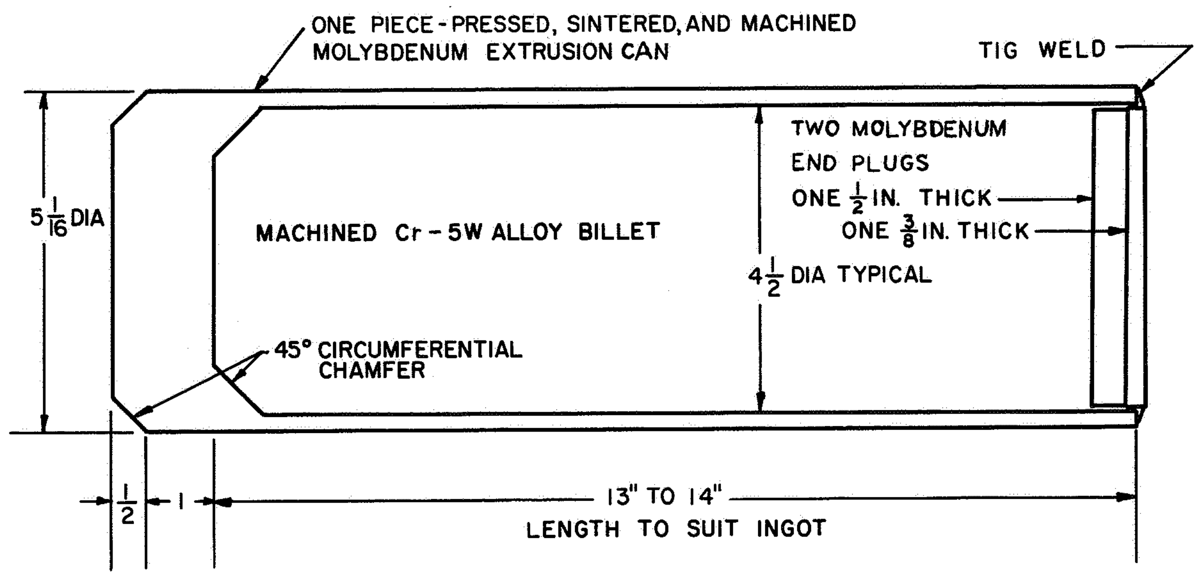


FIGURE 3 SCHEMATIC DIAGRAM OF TYPICAL EXTRUSION CAN AND BILLET ASSEMBLY

Figure 4: Transverse section of Cr-5W alloy extrusion showing molybdenum cladding. Electroetched in saturated oxalic acid solution.

Figure 5: Section of Cr-5W alloy extrusion and section of Cr-5W alloy extrusion reclad in open mild steel can.

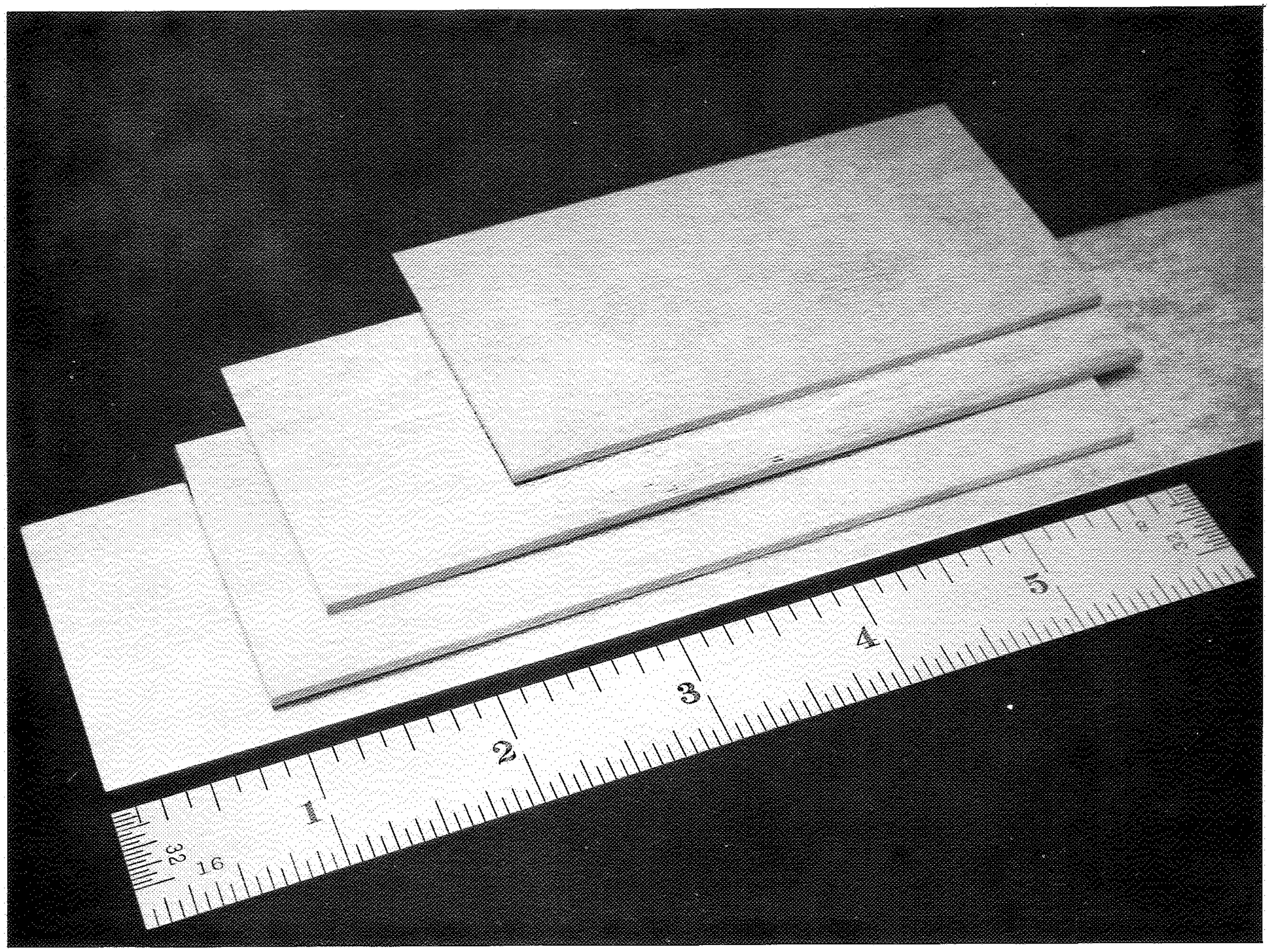


Figure 6: Typical Cr-5W alloy sheet product at 0.062 in. thick

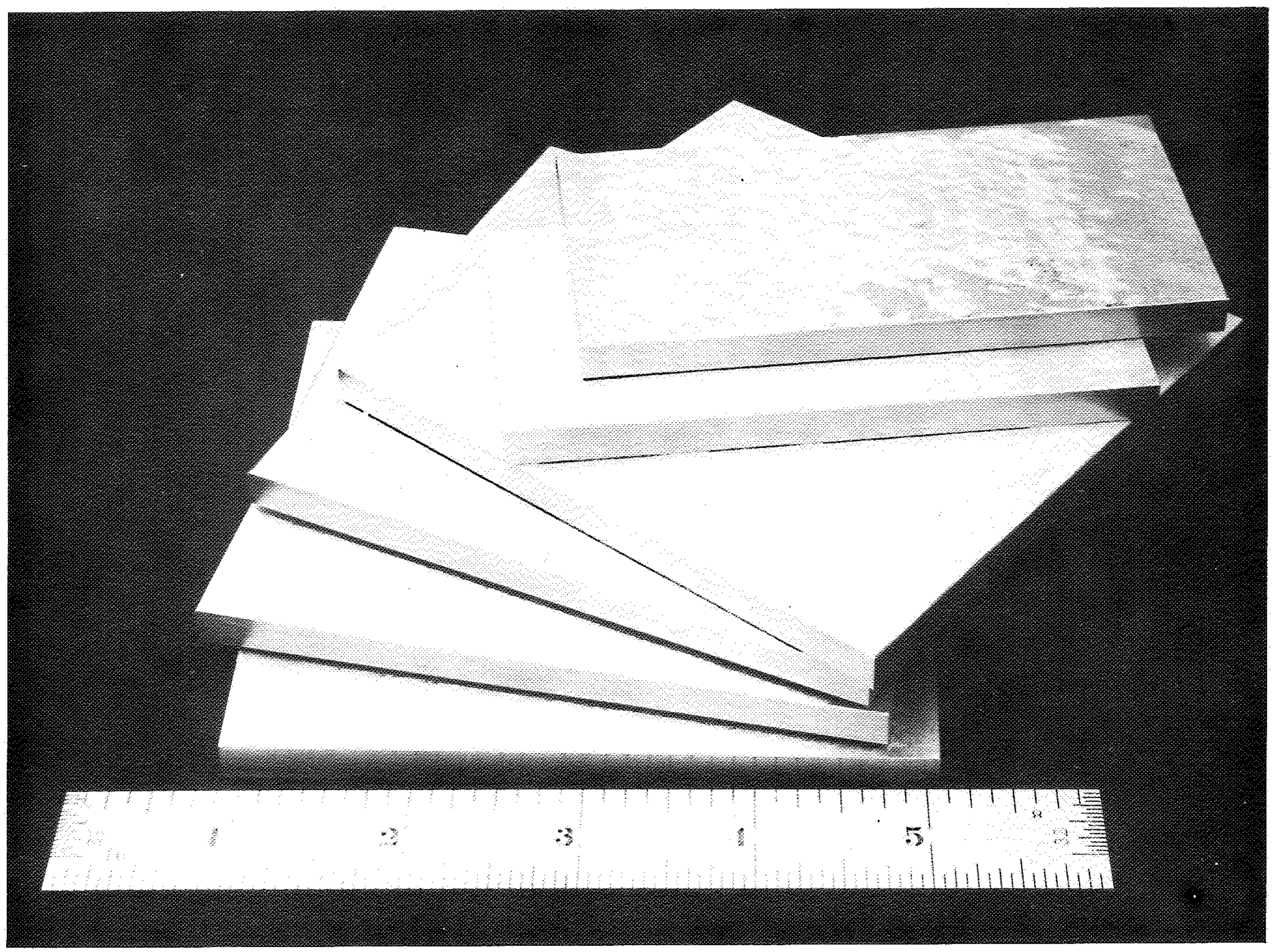


Figure 7: Typical Cr-5W alloy plate product at 0.250 in. thick

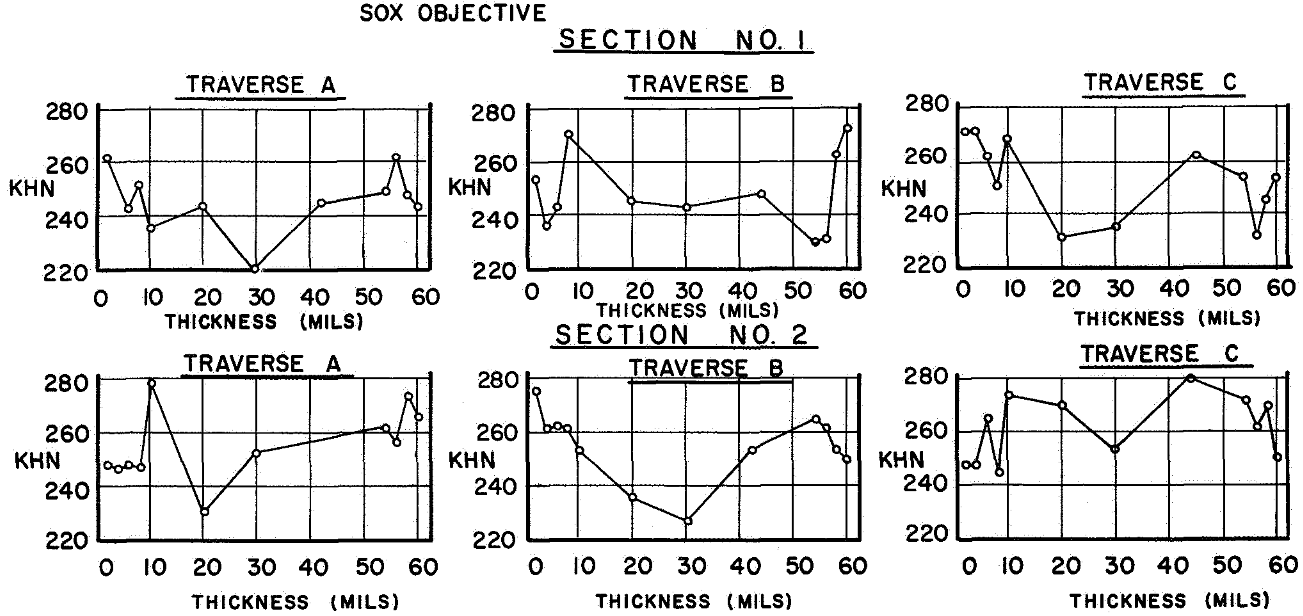
FIGURE 8

TYPICAL KNOOP HARDNESS TRAVERSE FOR RECRYSTALLIZED 0.062 IN Cr-5W ALLOY

MATERIAL: WROUGHT 0.062 IN. THICK Cr-5W ALLOY HEAT NO. 83-100 RECRYSTALLIZED 1 HR. AT 1050° C.

MATERIAL PREPARATION: TWO SECTIONS CUT PERPENDICULAR TO THE ROLLING DIRECTION. THREE HARDNESS TRAVERSES PER SECTION ACROSS THICKNESS AT $\frac{1}{2}$ IN. INTERVALS

HARDNESS TEST: KNOOP INDENTER. 0.1 KG LOAD SOX OBJECTIVE



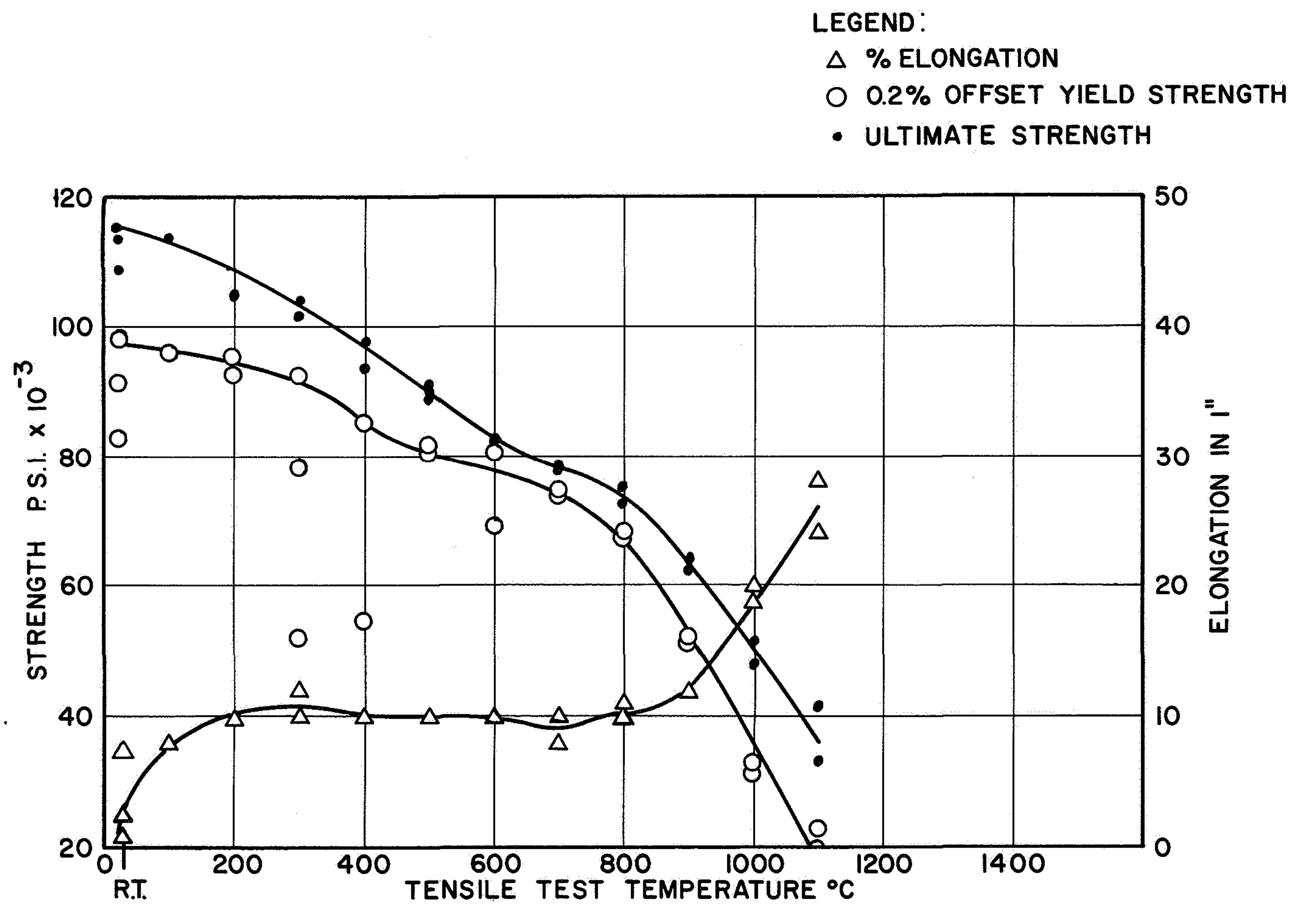


FIGURE 9 ELEVATED TEMPERATURE TENSILE PROPERTIES OF WARM WORKED Cr -5W ALLOY

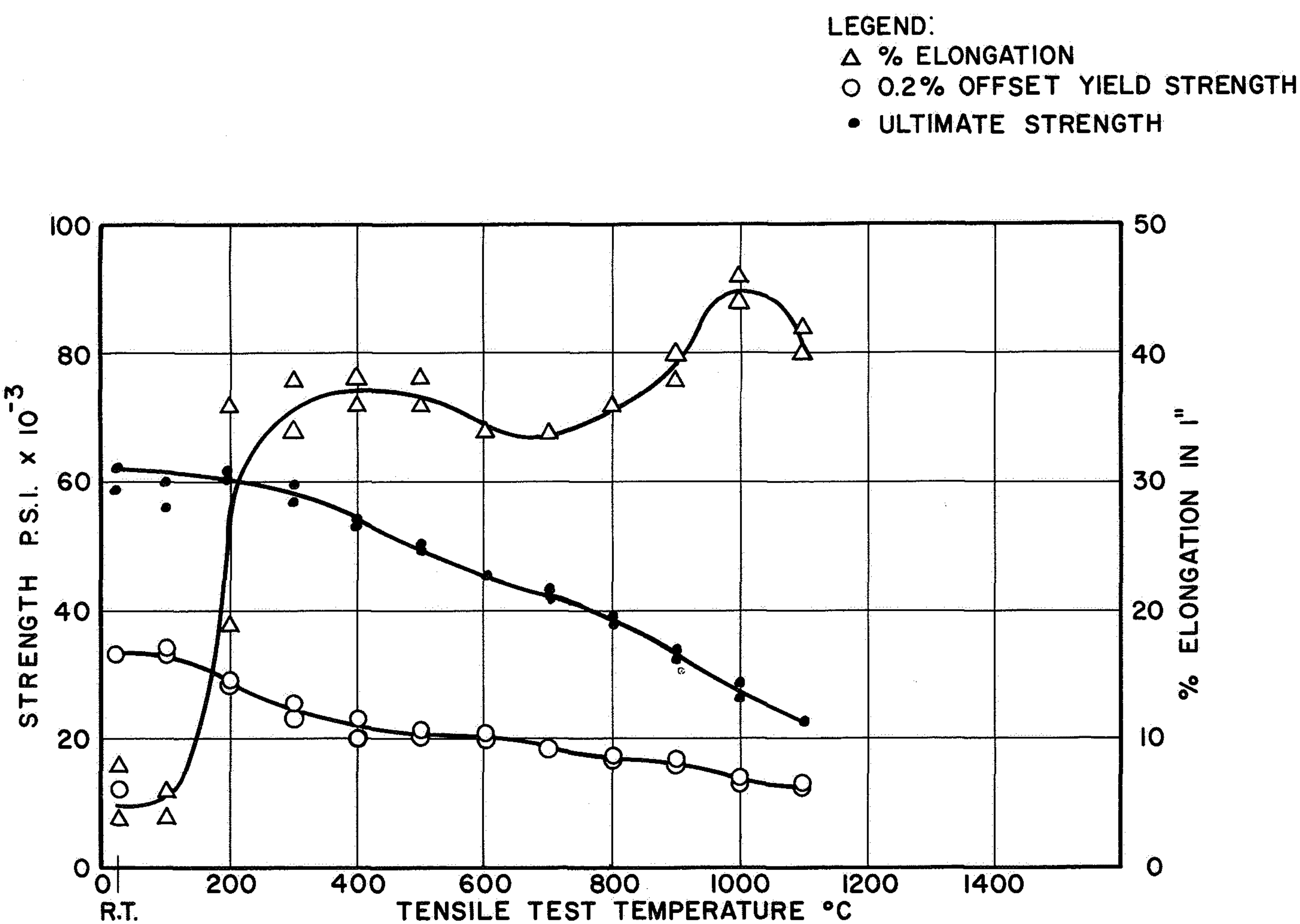
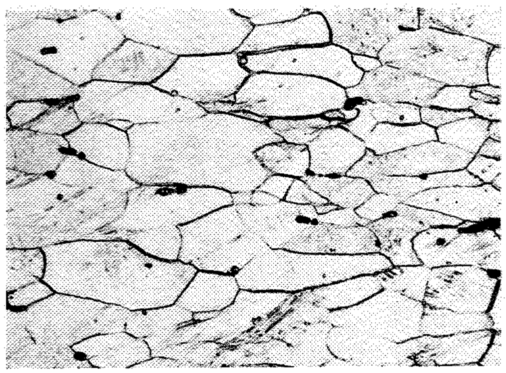
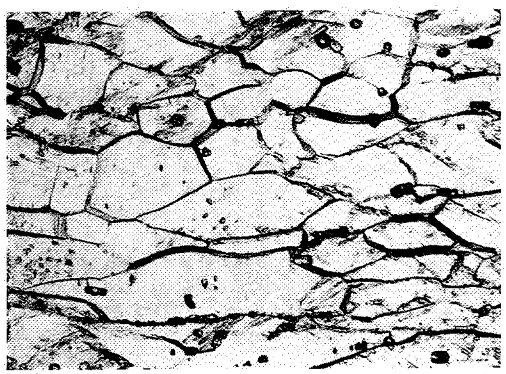


FIGURE 10 ELEVATED TEMPERATURE TENSILE PROPERTIES OF RECRYSTALLIZED Cr - 5W ALLOY

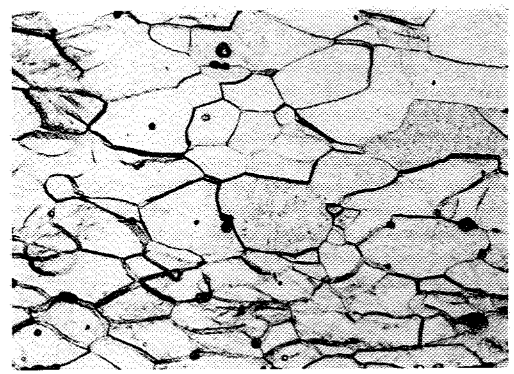


30% Reduction 200X
Wrought

30% Reduction 200X
950°C

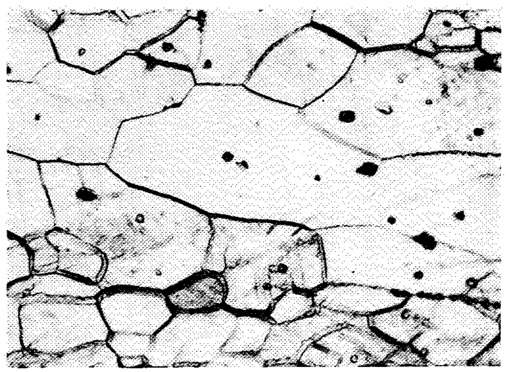


30% Reduction 200X
1000°C

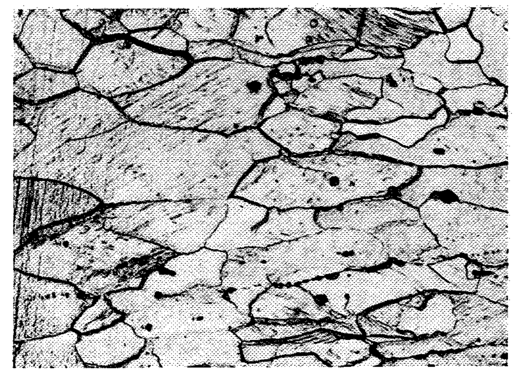


30% Reduction 200X
1050°C

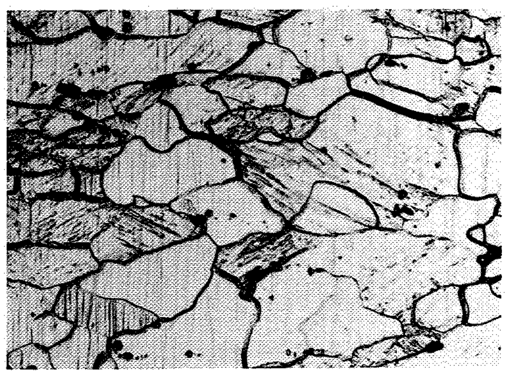
FIGURE 11 - Microstructure of 30% Warm Worked and Annealed Cr-5W Alloy



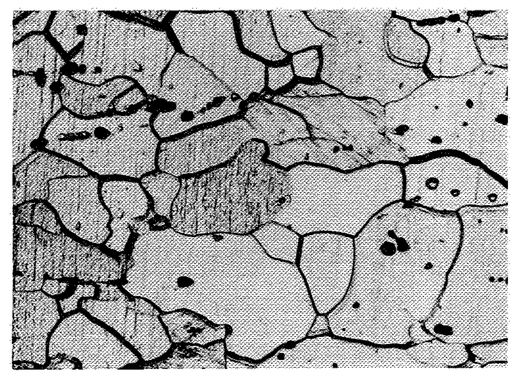
30% Reduction 200X
1100°C



30% Reduction 200X
1150°C

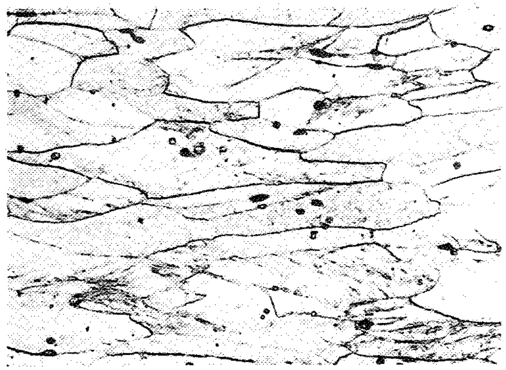


30% Reduction 200X
1175°C

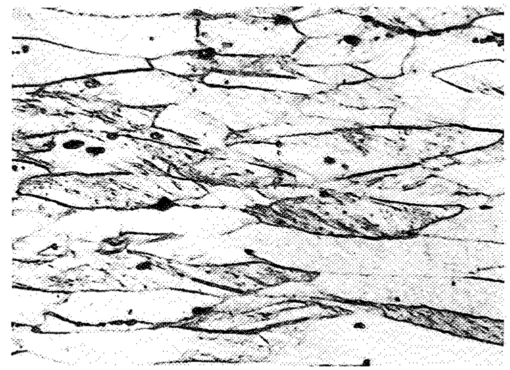


30% Reduction 200X
1200°C

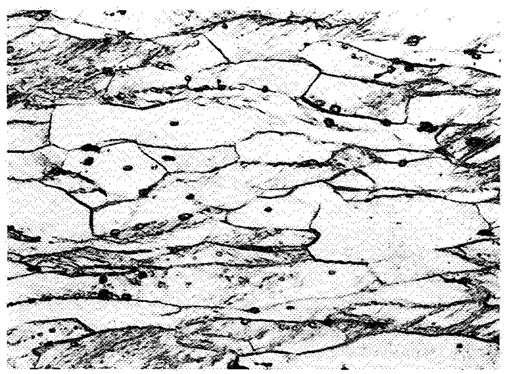
FIGURE 12 - Microstructure of 30% Warm Worked and Annealed Cr-5W Alloy



50% Reduction 200X
Wrought



50% Reduction 200X
950°C

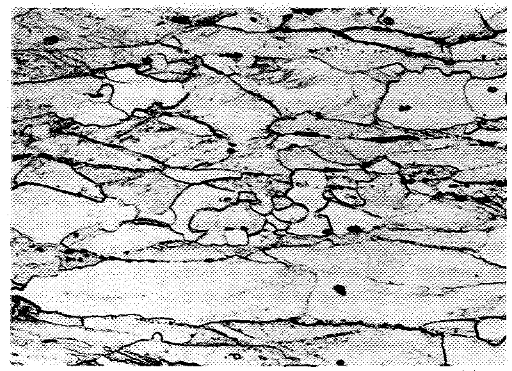


50% Reduction 200X
1000°C

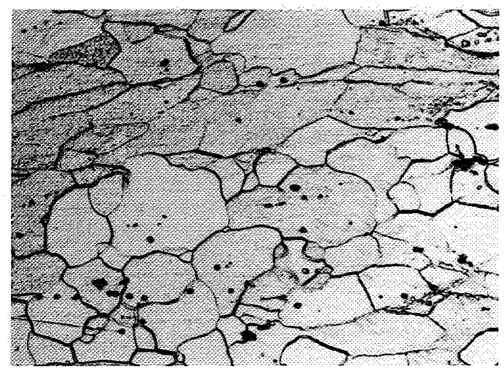
50% Reduction 200X
1050°C

FIGURE 13 - Microstructure of 50% Warm Worked and Annealed Cr-5W Alloy

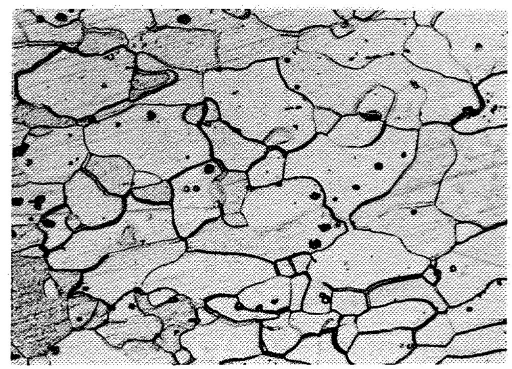
50% Reduction 200X
1100°C



50% Reduction 200X
1125°C

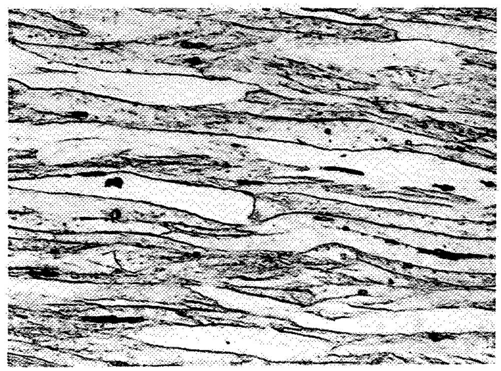


50% Reduction 200X
1150°C

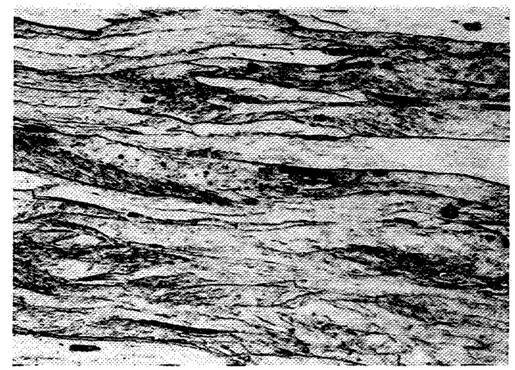


50% Reduction 200X
1175°C

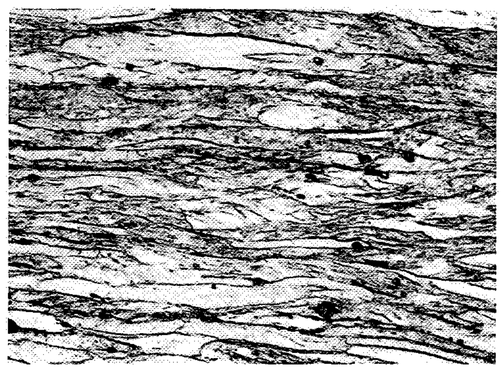
FIGURE 14 - Microstructure of 50% Warm Worked and Annealed Cr-5W Alloy



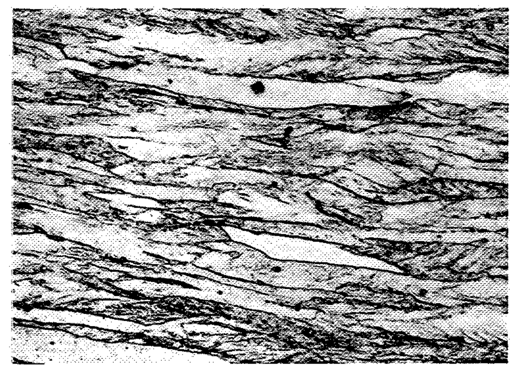
70% Reduction 200X
Wrought



70% Reduction 200X
950°C



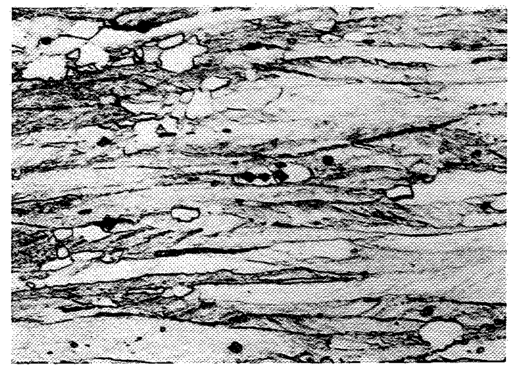
70% Reduction 200X
1000°C



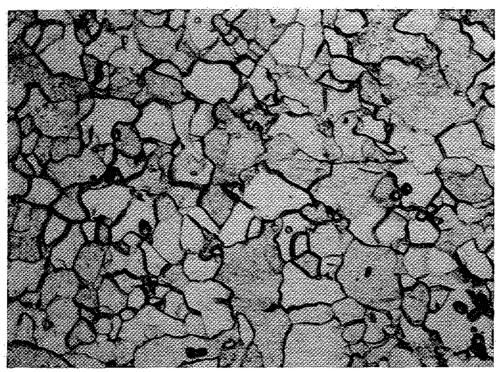
70% Reduction 200X
1050°C

FIGURE 15 - Microstructure of 70% Warm Worked and Annealed Cr-5W Alloy

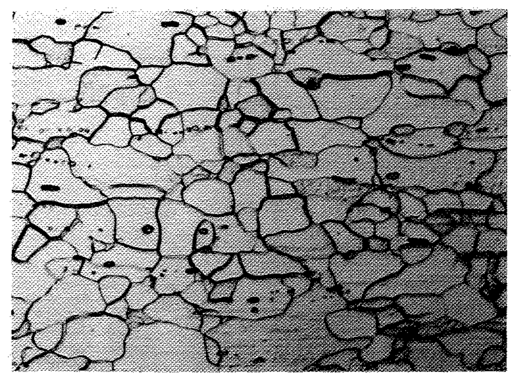
70% Reduction 200X
1075°C



70% Reduction 200X
1100°C

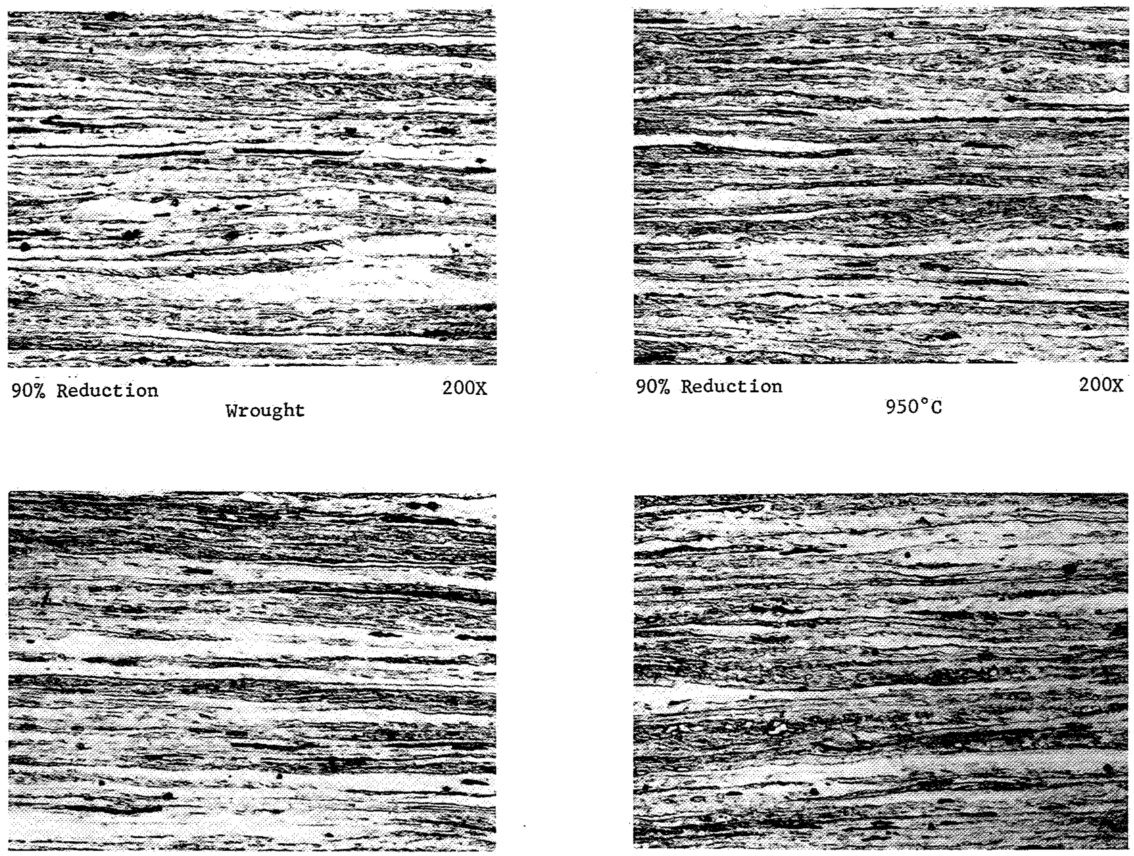


70% Reduction 200X
1125°C



70% Reduction 200X
1150°C

FIGURE 16 - Microstructure of 70% Warm Worked and Annealed Cr-5W Alloy



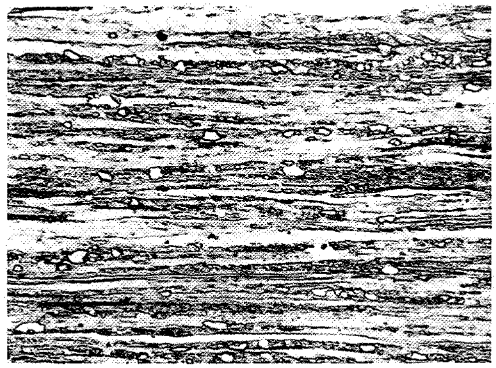
90% Reduction 200X
Wrought

90% Reduction 200X
950°C

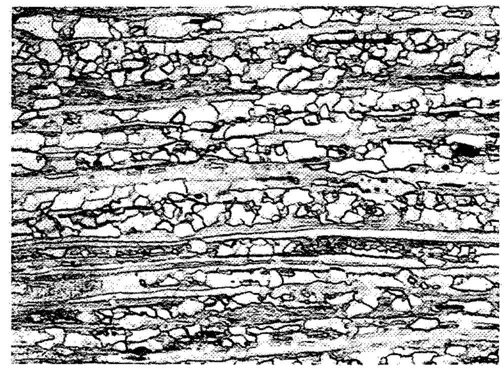
90% Reduction 200X
1000°C

90% Reduction 200X
1025°C

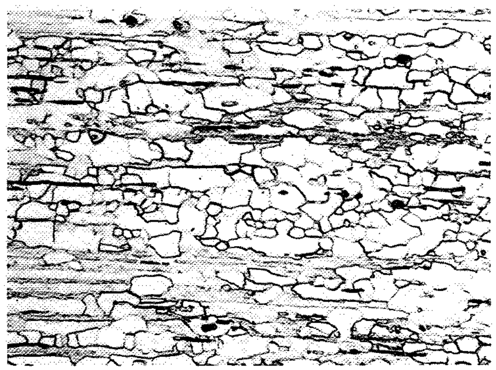
FIGURE 17 - Microstructure of 90% Warm Worked and Annealed Cr-5W Alloy



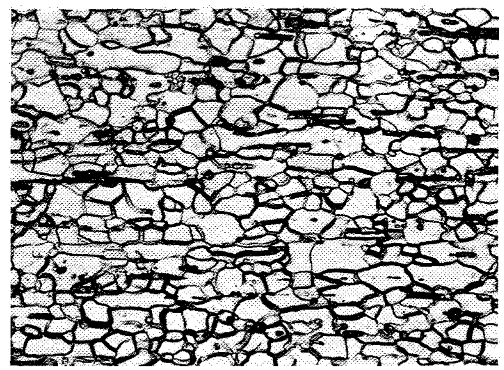
90% Reduction 200X
1050°C



90% Reduction 200x
1075°C

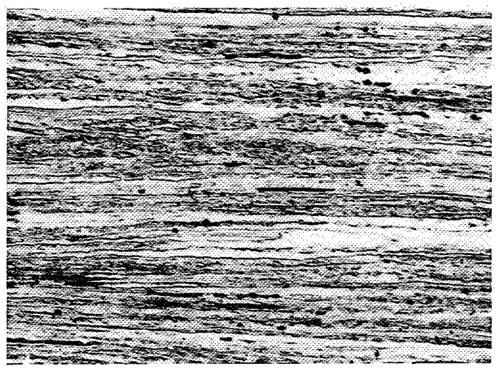


90% Reduction 200X
1100°C

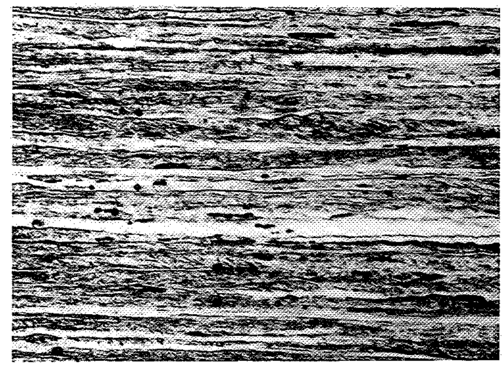


90% Reduction 200X
1125°C

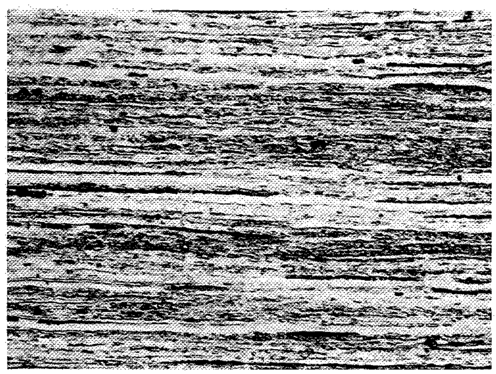
FIGURE 18 - Microstructure of 90% Warm Worked and Annealed Cr-5W Alloy



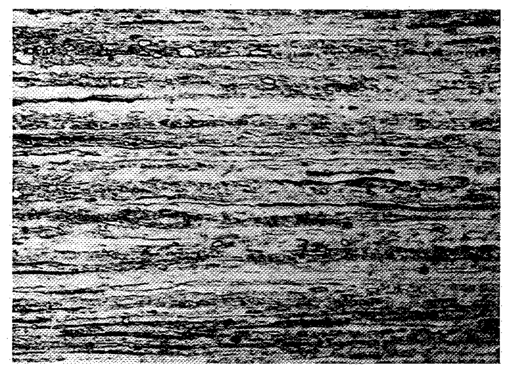
93% Reduction Wrought 200X



93% Reduction 950°C 200X

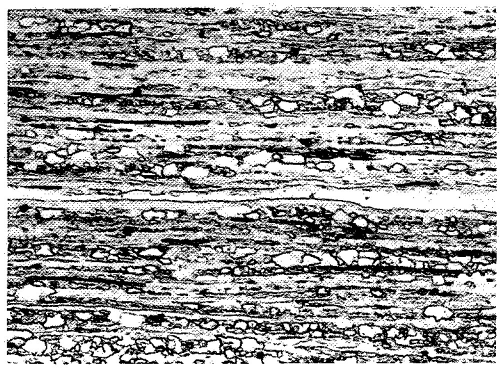


93% Reduction 1000°C 200X



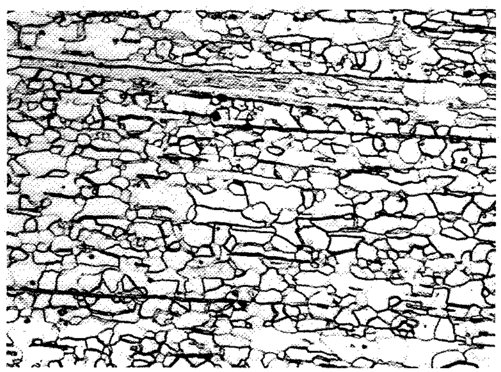
93% Reduction 1025°C 200X

FIGURE 19 - Microstructure of 93% Warm Worked and Annealed Cr-5W Alloy



93% Reduction 1050°C 200X

93% Reduction 1075°C 200X

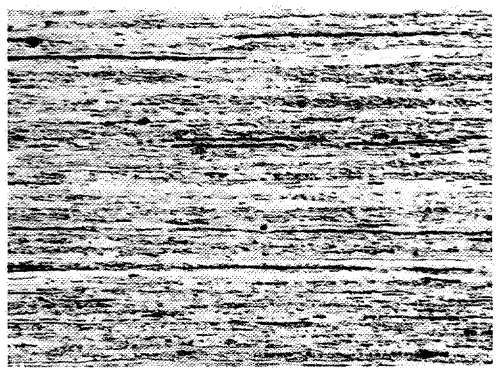


93% Reduction 1100°C 200X

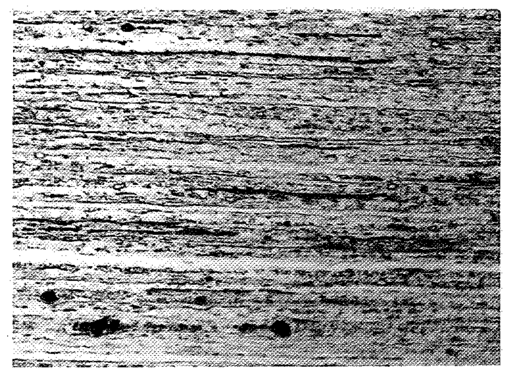
FIGURE 20 - Microstructure of 93% Warm Worked and Annealed Cr-5W Alloy

97% Reduction Wrought 200X

97% Reduction 950°C 200X

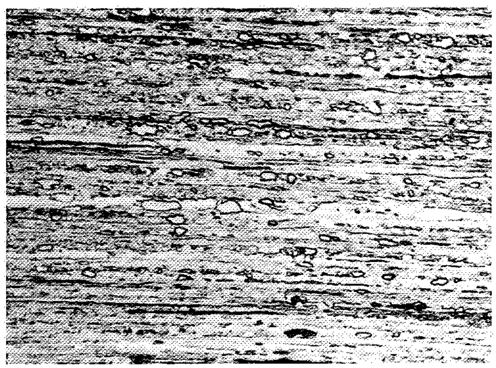


97% Reduction 1000°C 200X

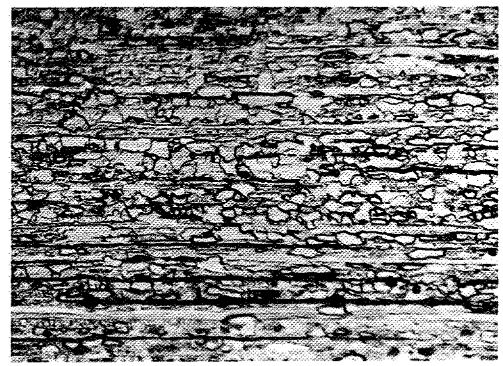


97% Reduction 1025°C 200X

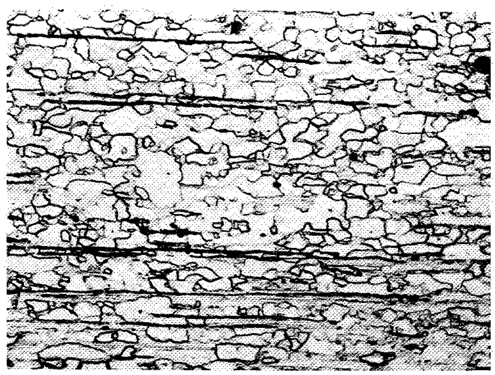
FIGURE 21 - Microstructure of 97% Warm Worked and Annealed Cr-5W Alloy



97% Reduction 1050°C 200X



97% Reduction 1075°C 200X



97% Reduction 1100°C 200X

FIGURE 22 - Microstructure of 97% Warm Worked and Annealed Cr-5W Alloy

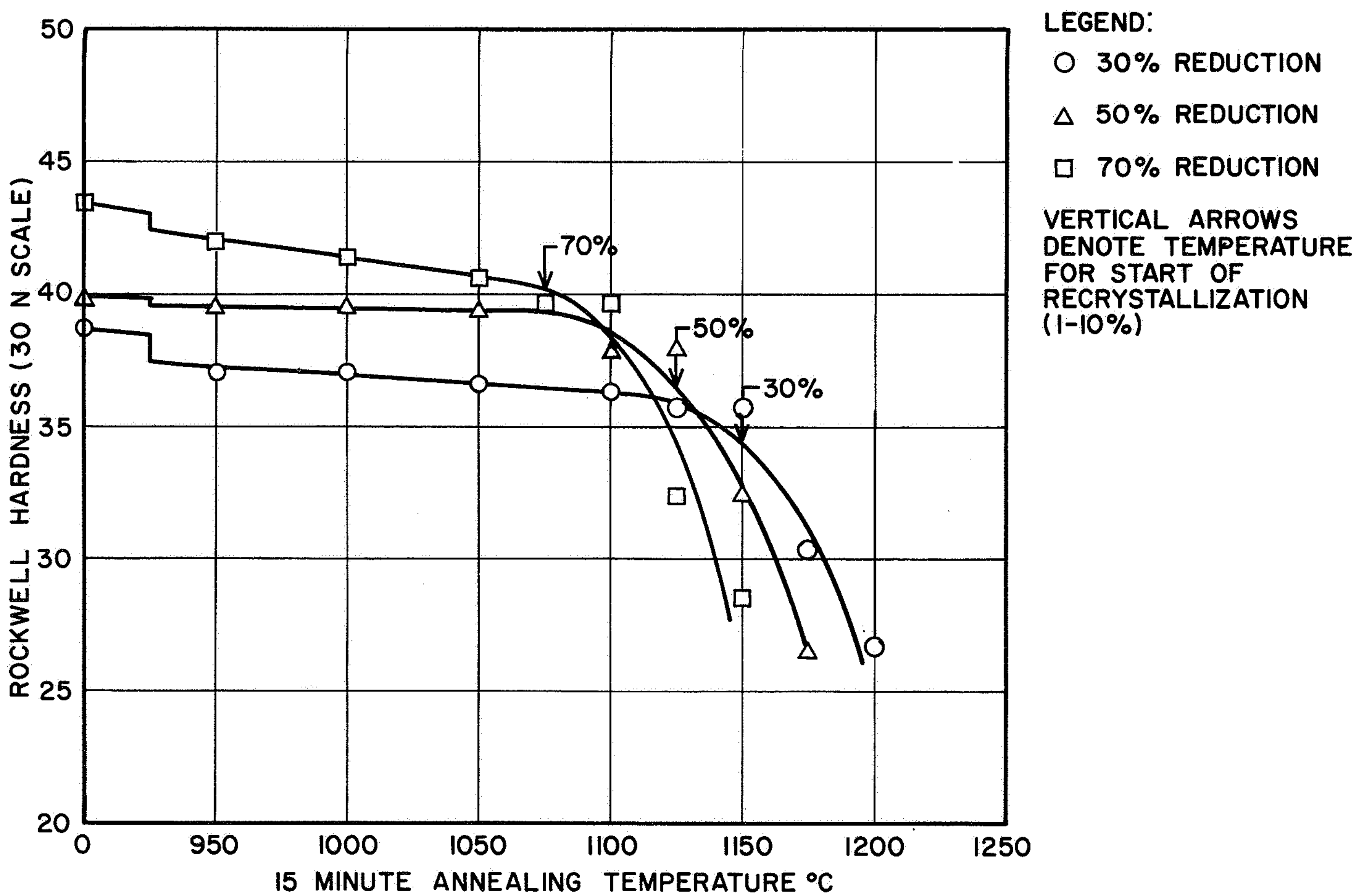


FIGURE 23 ANNEALING OF WARM WORKED Cr - 5W ALLOY - 30% TO 70% REDUCTIONS

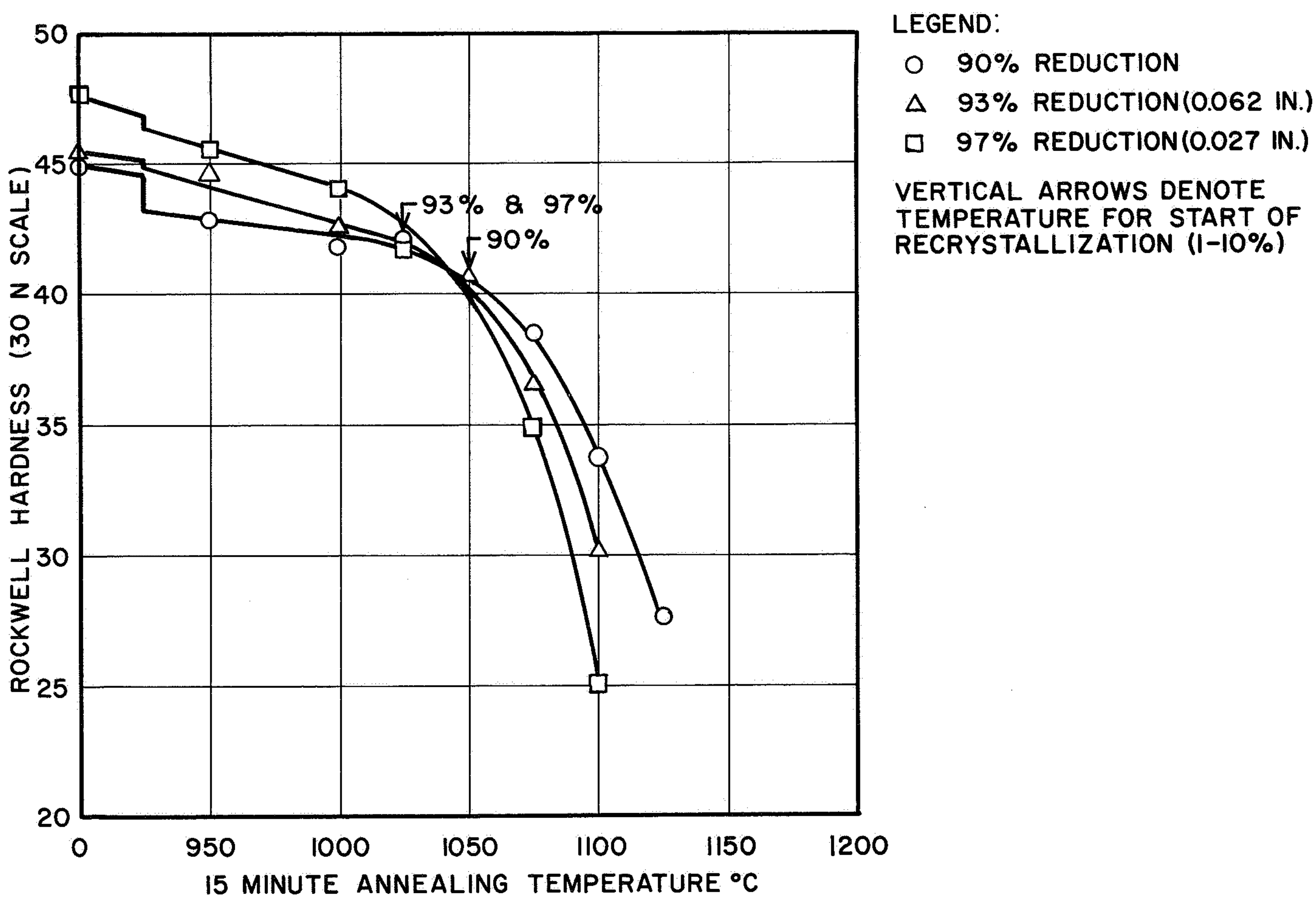


FIGURE 24 ANNEALING OF WARM WORKED Cr-5W ALLOY - 90% TO 97% REDUCTIONS

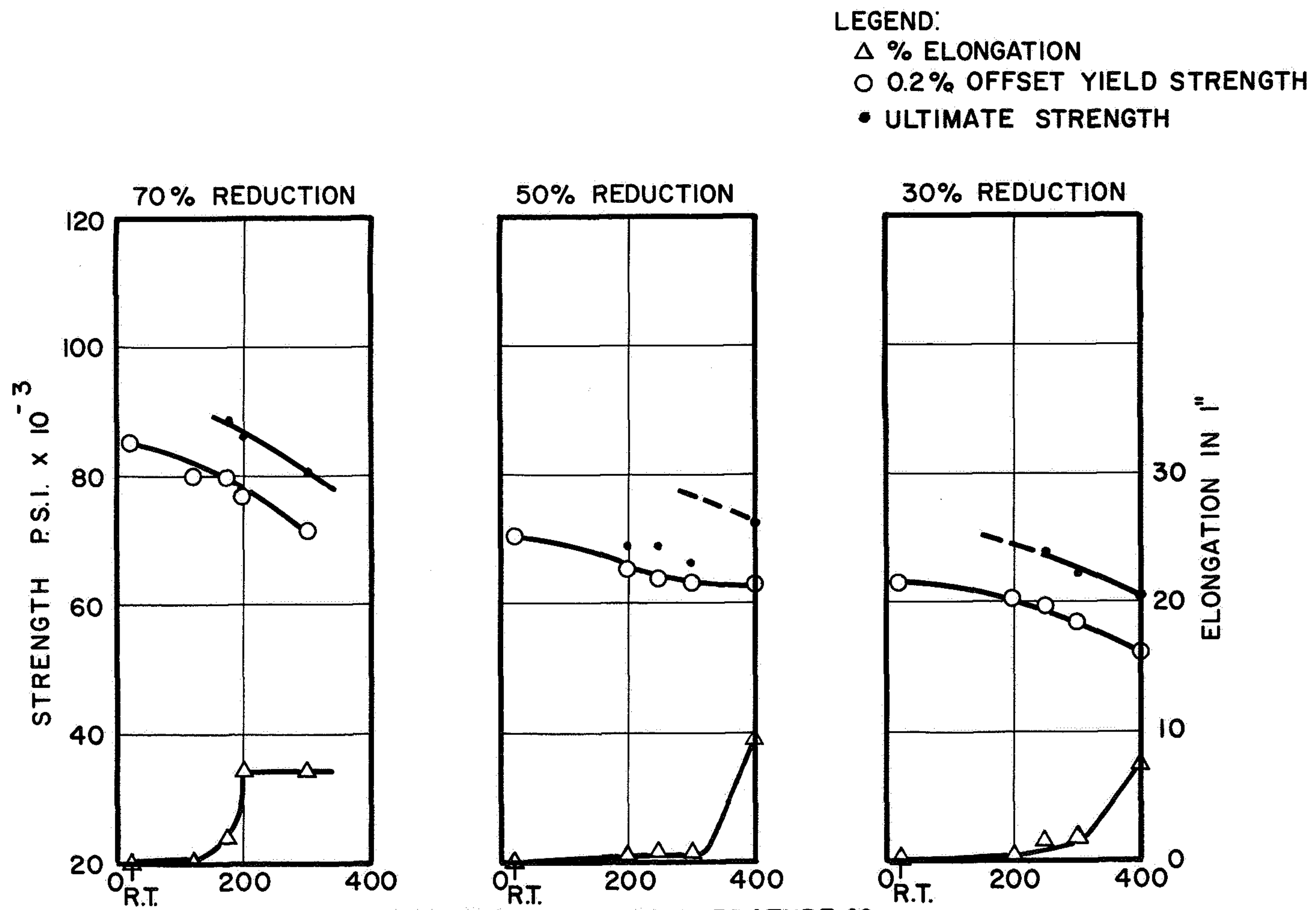


FIGURE 25 TENSILE PROPERTIES OF WARM WORKED Cr - 5W ALLOY 30% TO 70% REDUCTIONS

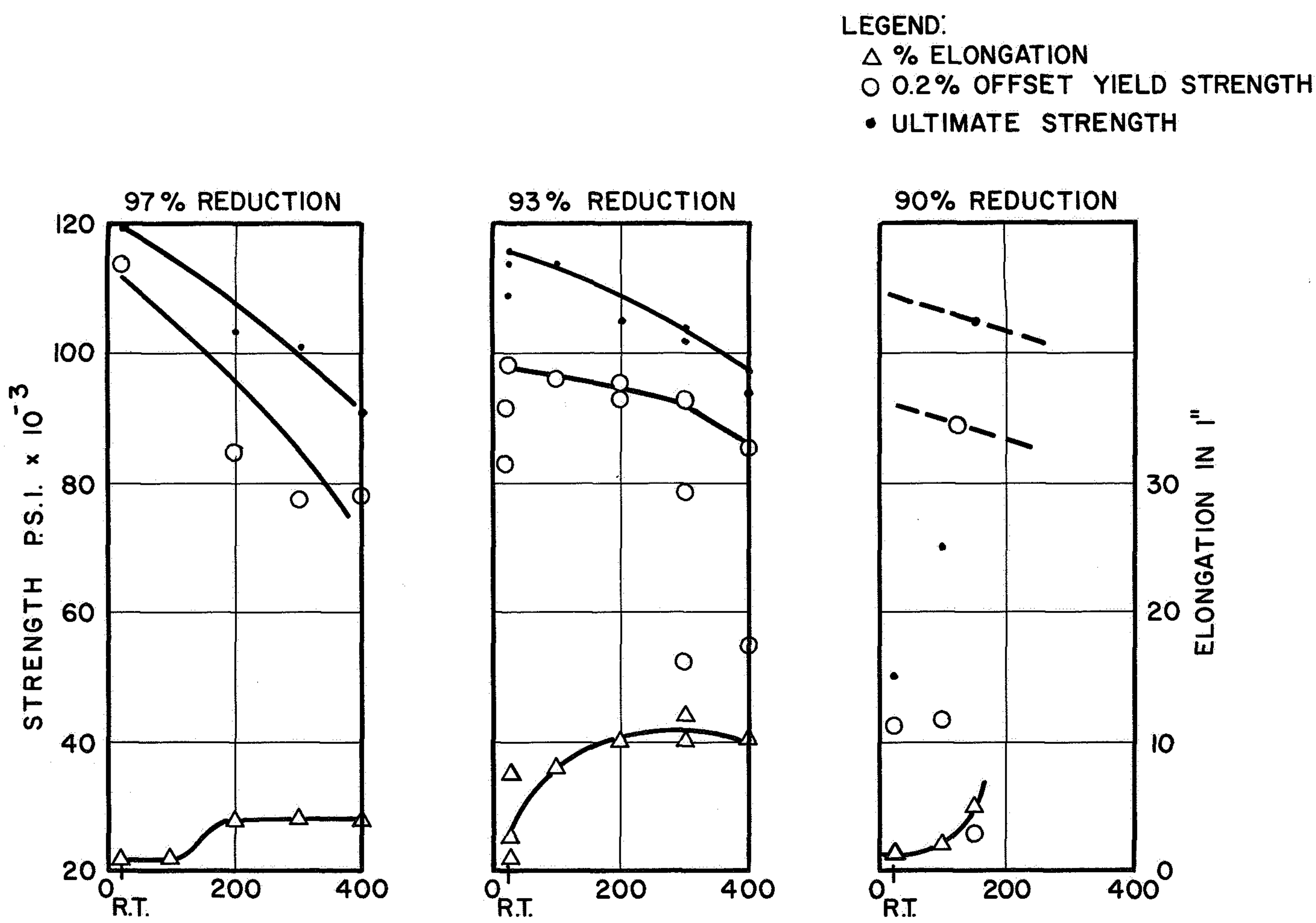


FIGURE 26 TENSILE PROPERTIES OF WARM WORKED Cr-5W ALLOY 90% TO 97% REDUCTIONS

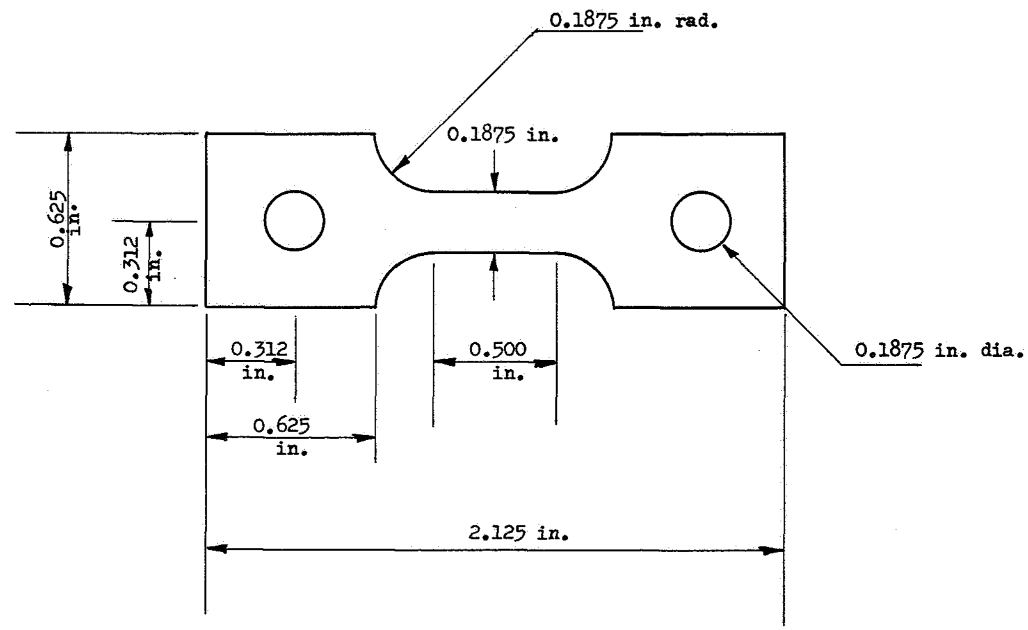


Figure 27: Tensile test specimen for alloy less than 0.225 in. thick

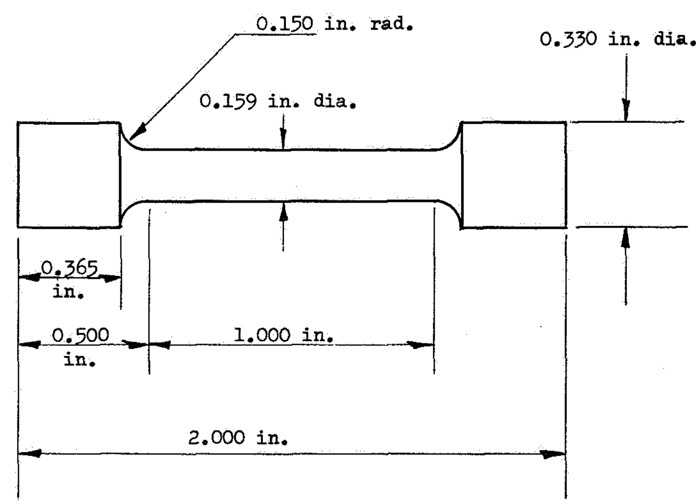


Figure 28: Tensile test specimen for alloy greater than 0.225 in. thick

APPENDIX

DUCTILE-BRITTLE BEND TRANSITION TEMPERATURE TEST DATA

TABLE I

DBBTT DATA FOR HEAT NO. 47-100

Direction Relative to Rolling Direction	Test Temp. °C	Excluded Angle of Bend
Transverse	100	0
	200	5
	300	10
	400	5
	425	25
	450	60
	475	90
	500	90

TABLE II

DBBTT DATA FOR HEAT NO. 56-100

Direction Relative to Rolling Direction	Test Temp. °C	Excluded Angle of Bend °
Longitudinal	200	22
	250	5
	275	40
	300	33
	300	50
	300	90
	325	90
Transverse	515	15
	530	90
	550	100

TABLE III

DBBTT DATA FOR HEAT NO. 57-100

Direction Relative to Rolling Direction	Test Temp. °C	Excluded Angle of Bend °
Longitudinal	155	10
	175	20
	185	45
	200	90
	200	90
	205	90
Transverse	400	10
	425	60
	450	90
	490	95
	530	91

TABLE IV

DBBTT DATA FOR HEAT NO. 58-100

Direction Relative to Rolling Direction	Test Temp. °C	Excluded Angle of Bend °
Longitudinal	200	5
	300	5
	425	60
	430	5
	450	40
	450	58
	475	90
	475	90
	510	90
Transverse	520	35
	550	38
	550	45
	550	90
	575	90
	600	95

TABLE V

DBBTT DATA FOR HEAT NO. 64-100

Direction Relative to Rolling Direction	Test Temp. °C	Excluded Angle of Bend °
Longitudinal	205	27
	205	80
	225	90
	225	90
	250	56
	250	90
	250	90
	275	90
	275	90
	300	90
Transverse	150	0
	200	0
	225	0
	250	95
	325	95
	400	95
	450	95
	525	95
	550	100

TABLE VI

DBBTT DATA FOR HEAT NO. 67-100

Direction Relative to Rolling Direction	Test Temp. °C	Excluded Angle of Bend °
Longitudinal	125	80
	150	90
	150	90
	175	90
	200	90
	205	22
	225	90
	250	90
	300	90
Transverse	250	45
	300	65
	300	85
	325	30
	325	90
	350	90
	400	90

TABLE VII

DBBTT DATA FOR HEAT NO. 70-100

Direction Relative to Rolling Direction	Test Temp. °C	Excluded Angle of Bend °
Longitudinal	100	5
	200	10
	225	5
	320	5
	325	10
	350	90
	350	90
	375	90
	420	90
Transverse	400	30
	400	45
	425	90
	430	90
	450	90
	450	90
	500	90

TABLE VIII

DBBTT DATA FOR HEAT NO. 83-100

Direction Relative to Rolling Direction	Test Temp, °C	Excluded Angle of Bend °
Longitudinal	66	30
	75	10
	75	30
	75	60
	100	45
	100	90
	100	90
	100	90
	120	90
	124	90
	125	90
	125	90
	200	90
	300	90
	400	90
	550	80
Transverse	400	40
	450	55
	500	55
	500	90
	525	55
	550	50
	570	55
	575	35
	604	55
	650	55
	700	60

TABLE IX

DBBTT DATA FOR HEAT NO. 83-100
(RECRYSTALLIZED 1 HR.
@ 1050°C TO 1100°C)

Direction Relative to Rolling Direction	Test Temp. °C	Excluded Angle of Bend °
Longitudinal	45	30
	75	37
	100	42
	100	90
	135	90
	140	90
	200	90
	300	90
	400	90

TABLE X

DBBTT DATA FOR HEAT NO. 83-100
(WROUGHT 0.090 IN. THICK)

Direction Relative to Rolling Direction	Test Temp. °C	Excluded Angle of Bend °
Longitudinal	100	0
	300	5
	350	10
	350	90
	375	90
	375	90
	400	90
	500	90

TABLE XI

DBBTT DATA FOR HEAT NO. 84-100

Direction Relative to Rolling Direction	Test Temp. °C	Excluded Angle of Bend °
Longitudinal	90	15
	125	20
	150	40
	175	60
	200	90
	200	90
	300	90
	400	90
Transverse	350	60
	400	60
	425	60
	425	80
	440	90
	445	90
	445	90

TABLE XII

DBBTT DATA FOR HEAT NO. 84-100
(WROUGHT 0.027 IN. THICK)

Direction Relative to Rolling Direction	Test Temp. °C	Excluded Angle of Bend °
Longitudinal	95	0
	200	20
	250	50
	275	35
	275	38
	275	90
	300	90
	300	90
Transverse	150	10
	200	30
	300	45
	400	20
	500	10
	600	10

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Metals & Controls, Inc.
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