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METALLURGY AND CERAMICS

PROGRESS REPORT

on

METALLURGY OF TUBALLOY

UNIVERSITY OF CHICAGO

Price \$ 0.40

Available from the
Office of Technical Services
Department of Commerce
Washington 25, D. C.

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DATE MAR 15 1957

For The Atomic Energy Commission

H. F. Canale

Chief, Declassification Branch

May 1, 1944

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PROGRESS REPORT

on

METALLURGY OF TUBALLOY

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Hugh K. Kinsman / OSTI
Authorizing Official

Date: 6/27/07

May 1, 1944

SUMMARY

Further dilatometric studies of gamma extruded metal indicate the advisability of heating extruded bars into the beta region for a brief period during the early part of the commercial outgassing process. It is believed that this treatment will iron out much of the dimensional instability that is observed on present outgassed metal.

Hot-rolled bars show anomalous dilatation behavior that requires further study.

New work on the nitrogen system indicates that a higher nitride, with a composition approximating M_3N_5 , is stable at about 500°C. under 1 atmosphere of nitrogen. The composition varies continuously with temperature and pressure in a way that suggests the mutual solubility of this nitride with a lower nitride.

Hydrogen analyses of eight slugs from hot-rolled bars gave values ranging from 0.6 to 1.0 p.p.m. If these are representative of all hot-rolled material, outgassing would not be necessary.

A single alpha extruded bar contained 1.1 p.p.m. of hydrogen.

Precipitation studies of carbide dissolved in gamma at 1000°C. and retained in solution by quenching indicate that carbide solubility in alpha is

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less than .01% and in beta is in the range of .01 to .05%.

Hot hardness measurements and compression tests performed at high alpha temperatures and by heating into beta and cooling to high alpha temperatures do not confirm the suggestion of beta retention as a cause of failure to extrude under the latter heating conditions. Likewise, compression tests under similar conditions show no lack of plasticity. Further work on laboratory extrusion is to be undertaken in studying this further.

Promising bonding by diffusion of Al-Si and Al has been obtained. Copper and zinc appear to aid this diffusion.

Beryllium end caps by hot-pressing beryllium slugs and beryllium powder do not appear promising. The powder oxidizes too much to produce strong compacts, and the metal slugs are not sufficiently plastic. Powder metallurgy with cold compacting is still of interest.

A standard condition for tuballoy consisting of annealing through the alpha-beta transition, followed by slow cooling, or possibly outgassing, is proposed. Tensile tests on a gamma extruded and outgassed bar before and after such treatment show little change, and the elastic constants in both conditions compare favorably with those previously reported. A grain refinement was obtained. It is believed that such a treatment will be particularly beneficial in decreasing dimensional changes in alpha worked metal.

Low superheat and pouring temperatures are shown to produce fine grain in 3% Al-Be castings in sizes from 1/4" to 1-3/8" diameter bars.

EXPERIMENTAL DATA

Dilatometric Studies

The thermal expansion studies described in the last two reports, pp. 21-25 and 39-51, have been continued. Particular emphasis has been placed on studies of gamma extruded material in an effort to determine some practical

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method of obtaining dimensional stability. Hot-rolled material has also been studied but not so extensively.

Work described in the last report showed that gamma extruded and out-gassed material is not in a stable state as regards dimensional changes induced by heating into the beta region and subsequently cooling to room temperature. A single heating-cooling cycle results in permanent longitudinal contraction and transverse expansion. Most of this permanent change in dimensions can be accounted for by changes in the mean coefficients of linear expansion in the alpha region. The coefficient in the longitudinal direction increases as a result of the alpha-beta-alpha transformations, while the transverse coefficient of expansion decreases. In addition to the dimensional changes resulting from changes in expansion coefficient, some evidence was obtained to indicate that the metal may experience an isothermal deformation in the high alpha region. Although this phenomenon was not observed with gamma extruded metal, it was thought important to look for it more carefully than had been done heretofore.

The experiments described below have been carried out to check these dimensional changes more extensively and to lay the ground work for setting up a "normalizing" heat treatment to insure dimensional stability in commercial slugs prior to jacketing. The curves of Figures 55 to 57, for gamma extruded material, and Figures 58 to 60, for hot-rolled bars, were all made according to a more or less standardized procedure as follows: The dimensions of the new specimens were carefully determined with a micrometer. The initial heating in the dilatometer was at a rate (called "fast") that required about two hours to heat from room temperature to 720°C., and from seven to nine minutes were required for the alpha-to-beta transformation. The specimen was then furnace-cooled through the transformation, with no heating current on the furnace, at a rate which required about 30 minutes to cool from 720°C. to

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630°C. and four to five minutes spent in the beta-to-alpha transformation. The specimens were then held at 633[±]4°C. overnight (17 to 20 hours) to search for isothermal dilatations. They were then allowed to furnace cool, generally to about 485°C., to determine the alpha coefficient of expansion before heating in the final cycle. The second and last heating-cooling cycle was carried out more slowly, but the time in transformation varied rather widely because of the difficulty of control when specimens overheat and undercool on entering the transformation. The time in transformation is indicated on each curve. The specimens were then furnace-cooled to room temperature overnight and were again measured with micrometers to check the permanent dimensional changes shown by the dilatometer.

Results on Gamma Extruded Metal

The results on three representative specimens are shown in Figures 55 to 57. No isothermal dilatations were observed in 17 to 20 hours at 633°C. The final permanent changes in specimen length were checked in all cases by micrometer readings.

The first cycle heating and cooling rates roughly correspond to those that might be used in a "normalizing" heat treatment during outgassing, if extruded rods were first heated into the beta and then cooled to the high alpha for the outgassing soak. Two questions then arise: (1) How much change has been produced by the alpha-beta-alpha cycle, followed by several hours' soak in the high alpha region? (2) Is the material now dimensionally stable?

Neither of these questions can be answered precisely nor with certainty. However, the experiments summarized in Figures 55 to 57, together with others whose curves are not reproduced, furnish the basis for tentative opinions.

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Figure 55. Gamma extruded, longitudinal specimen. This specimen shows the typical permanent contraction and the reduction of the coefficient of expansion in the alpha after a single cycle. After the first (fast) cycle, the specimen would have shown a permanent contraction of about .006 inch per inch of which about .004 inch is attributable to an increase in coefficient of expansion and .002 inch to a net contraction in the first cycle transformations. In the second cycle transformations, there was a net growth of .003 inch per inch to give a final net contraction of .003 inch per inch.

This specimen was 3-3/8 inches long, whereas all others were about 1 inch long. The increased specimen mass in this case accounts for the increased length of time required for completing the transformations.

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FIG.55

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Figure 56. Gamma extruded, transverse specimen. These curves are fairly typical, except that the coefficients of expansion in the alpha are lower than usual for transverse specimens. The coefficient of expansion after the first cycle has dropped from about 20×10^{-6} to 16.8×10^{-6} per °C. and there is no further change after the second cycle. The permanent growth of about .004 inch per inch is accounted for principally by the growth during transformation in the second (slow) cycle. After the first cycle, the specimen would probably have shown a permanent growth of only about .001 inch per inch.

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FIG. 56

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Figure 57. Gamma extruded, transverse specimen. This shows the permanent gain in length and an unusually large reduction in the coefficient of expansion in the alpha. Of the .0075 inch per inch permanent growth, approximately .0055 inch per inch apparently took place in the first (fast) cycle as a result of the change in the alpha expansion coefficient. The other .002 inch was the result of growth during the transformations in the second cycle.

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FIG. 57

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Longitudinal specimens are always shorter after a single heating-cooling cycle, and soaking in the high alpha region has no effect. Alpha coefficients of expansion always increase to about $20 \pm 2 \times 10^{-6}$ per °C. for the range 25 to 660°C. Observed decreases in length measured at room temperature have fallen in the range .002 to .006 inch per inch, but these values are, in each case, lower limits of the actual contractions because surface roughening and slight changes in shape tend to make the observed length after heating larger than the average length. When estimated corrections for this error are made, the observed contractions of longitudinal specimens fall in the range .004 to .007 inch per inch.

Transverse specimens are always longer after a single heating-cooling cycle, with no dimensional change as a result of soaking in the high alpha region. When corrections for roughening and distortion are applied, the observed expansions all fall in the range .001 to .004 inch per inch.

Since there are no detectable changes in density, the specific longitudinal contraction should be twice the specific transverse expansion, or

$$\frac{\Delta L}{L} + 2 \frac{\Delta D}{D} = 0$$

This condition is met satisfactorily within the widely scattered values observed.

In considering the question of stability after a single alpha-beta-alpha cycle, followed by a soak in the high alpha, it should be recalled that, in practice, the metal is not subjected to a slow alpha-beta-alpha cycle after outgassing. Consequently, any dimensional changes induced by the second (slow) cycles in the present experiments must be carefully examined to determine whether or not they are the result of the slowness of the transformations.

It will be noted in Figures 55 to 57 that the alpha expansion coefficients do not change appreciably after the first cycle. The principal

dimensional change induced by the second cycle is an increase in length resulting from a net growth in the two transformations. There is considerable evidence to support the belief that this growth is a function of the rate of heating and cooling through the transformations. Fast heating and cooling rates produce little effect. Consequently, it is unlikely that the fast heating and cooling during canning operations will produce much effect.

On the basis of results obtained to date, therefore, it would seem advisable to "normalize" gamma extruded metal during the outgassing by initially heating the charge into the beta, furnace-cooling to the usual outgassing temperature, and completing the outgassing operation with the usual soaking period. Further test will be required to determine how effective this treatment actually is. In particular, the effect of long periods in the medium and low alpha regions should be investigated.

One other aspect of the problem should be mentioned. Although a single heating-cooling cycle seems to produce most of the changes that are likely to occur, the "stabilized" coefficients of expansion may vary rather widely from sample to sample. The extreme example of this variation that has been observed in gamma extruded metal is the very low coefficient, about 10×10^{-6} per $^{\circ}\text{C}.$, shown in Figure 57 as compared with the more usual values of 17 to 20×10^{-6} per $^{\circ}\text{C}.$, shown in Figure 56 and in Figure 33 of the last report. These variations are unexplained, but they suggest the possible necessity of more careful control of extrusion and quenching temperatures.

Results on Hot-Rolled Metal

Figures 58 to 60 summarize the results on hot-rolled material. It is impossible to draw general conclusions on the basis of these three experiments, particularly in view of the widely differing results obtained on the two

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Figure 58. Hot-rolled, longitudinal specimen. Neither this specimen nor the similar longitudinal specimen shown in Figure 59 behaved as gamma extruded longitudinal specimens. The final alpha coefficient of expansion, 5×10^{-6} per °C., is very much lower than that observed after the first cycle. There is no appreciable growth or contraction in the transformation as has been observed with other specimens. The first (fast) cycle would have produced a permanent growth of about .002 inch per inch as a result of a reduction in the alpha coefficient of expansion.

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FIG. 58

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Figure 59. Hot-rolled, longitudinal specimen. The specimen starts with the lowest coefficient of expansion yet observed, 4×10^{-6} per $^{\circ}\text{C}$. After the first (fast) cycle, the coefficient has increased to about 18×10^{-6} per $^{\circ}\text{C}$., so that the specimen would show a permanent contraction of about .009 inch per inch. After the second cycle, the coefficient had increased to 21×10^{-6} , but a net growth of about .003 inch per inch during the second cycle transformations reduced the net permanent contraction to .008 inch per inch. This is, however, the largest permanent contraction that has been observed.

The dilatation phenomena in this specimen are exactly opposite to those shown in Figure 58 in a similar specimen. It is apparent, therefore, that no generalizations can be made regarding the dilatation behavior of rolled rods until more study has been given this material.

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FIG. 59

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Figure 60. Hot-rolled, transverse specimen. The general behavior of this specimen is much like that of gamma extruded transverse specimens. The coefficient of expansion starts high, 26×10^{-6} per °C., and ends at 12.7×10^{-6} per °C. after the two cycles. Most of this change had taken place at the end of the first cycle. The permanent growth of .012 inch per inch consists of about .004 inch per inch net growth during the second cycle (slow) transformations. Consequently, had the specimen been cooled to room temperature after the first cycle, a permanent growth of about .008 inch per inch would have been observed.

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FIG.60

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Effect of Pressure on the Alpha-Beta Transformation Temperature

$$\frac{dT}{dp} = 4 \times 10^{-3} \text{ } ^\circ\text{C./atmosphere}$$

To explain the failure to complete the alpha-to-beta transformation below about 672°C., some portion of the metal must be under a pressure of about 40,000 p.s.i.

Work has recently been resumed on the nitrogen system. The results are incomplete, and interpretation must await further study.

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About 100 equilibrium points have been obtained at eight temperatures, ranging from 492°C. to 1048°C. These points were approached from both high and low compositions and in all cases showed complete reversibility. One hour was the maximum length of time required to reach equilibrium for the 658°C. to 1048°C. isotherms. For the 492°C. isotherm, as much as two days were necessary to establish individual equilibrium points.

For this range of temperatures, the nitrogen concentrations decreased with decreasing pressures and decreased with increasing temperatures. The maximum concentration observed corresponded to a composition $MN_{1.68}$ at 492°C. and 825 mm. of Hg pressure. At 50 mm. of Hg and this same temperature, the concentration had decreased to $MN_{1.65}$. At 1048°C., the concentrations were $MN_{1.56}$ and $MN_{1.52}$ at 825 mm. of Hg and 50 mm. of Hg, respectively. The general shape of the isotherms resembled Langmuir adsorption isotherms in the range of pressures from 50 mm. to 825 mm. of Hg. Below 50 mm. the concentration decreased very rapidly with small decreases of pressure. Isobaric curves plotted from data supplied by the isotherms could be approximated exceedingly well by straight lines over this range of temperatures and pressures.

However, when the same specimen was heated to 1200°C., a new effect occurred. The solubility decreased as was expected, but equilibrium was not reached in short lengths of time. Gas was evolved from the sample at an extremely slow rate and after five hours was still being evolved. When the specimen was then cooled to temperatures below 1048°C., corresponding to isotherms previously determined, new "equilibrium" points were obtained. When the specimen was again heated to 1150 to 1200°C. and then cooled to the same isotherms, still other points were obtained. The data that were previously reproducible could no longer be reproduced.

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A Toepler pump was inserted in the system, and gas was removed at 1150°C. When no more gas was being evolved, the specimen was heated to 1200°C. and pumped on again with the Toepler pump. No further gas was removed after several hours of pumping. It appears that this nitride phase of lower composition formed slowly from the higher nitride phase at temperatures above 1048°C. Previous work indicates that this lower nitride phase is stable up to 1700°C.

Work is in progress to investigate further the properties of this lower nitride with emphasis on equilibrium in possible systems composed of two nitride phases and a gaseous phase of nitrogen.

Hydrogen Content of Rolled Rods

Eight slugs from rolled rods were received for hydrogen analyses. Results were as follows:

<u>Slug Number</u>	<u>H₂ Content in P.P.M.</u>
D-15	0.6
D-8	0.7
A-9	0.6
A-12	0.7
B-10	1.0
C-18	0.7
E-21	0.9
H-1	1.0

These values are comparable to those observed on outgassed, extruded rods. If these samples are representative all rolled material, there would seem to be no necessity for outgassing. Before making a definite decision on the necessity for outgassing, it would be desirable to analyze more rolled

material and, in particular, to search for a possible variation in hydrogen content along a rolled bar.

Hydrogen Content of an Alpha Extruded Bar

A bar, 3/4" in diameter by about 9-1/2" long, that had been alpha extruded by Creutz was found to contain 1.1 p.p.m. hydrogen.

Carbide Solubility

Micrographs showing solution of carbide in tuballoy on heating up to 96 hours at 1000°C. and quenching were given in Report #15. Tests to indicate the solubility of the carbide in the alpha and beta phases have been made and are discussed in the following.

Biscuit metal .01% C shows no change in the amount of carbide upon heating at 600°C. in the as-received condition up to 48 hours, as shown in Figures 61, 62, 63, and 64. Reheating a specimen of biscuit metal, which had a solution treatment of 48 hours at 1000°C. followed by quenching, at 600°C. shows precipitation of carbide after 6 hours with little additional in 48 hours (Figures 65, 66, 67, and 68). This indicates that solubility of carbide in alpha is substantially under .01%, since not all carbides were in solution after the 48-hour treatment. Biscuit metal, solution-treated 96 hours at 1000°C. and quenched, when reheated at 725°C. for periods of 6, 10, and 48 hours shows no precipitation of carbide, as indicated in Figures 69, 70, 71, and 72. Retention of the carbides in solution at 725°C. indicates a carbide solubility in beta of at least .01% C.

Similar tests were made on specimens of Bar Al088, .09% C. Some precipitation occurred on heating the as-extruded metal six hours at 600°C.,

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200X

27360

Figure 61. As received.

200X

29777

Figure 62. Heated 6 hours at 600°C., quenched.

200X

29778

Figure 63. Heated 10 hours at 600°C., quenched.

200X

29779

Figure 64. Heated 48 hours at 600°C., quenched.

Biscuit Metal, .01 C, Heat-Treated as Shown

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200X 27361
Figure 65. Heated 48 hours at
1000°C., quenched.

200X 29780
Figure 66. Heated 48 hours at
1000°C., quenched.
Heated 6 hours at 600°C., quenched.

200X 29781
Figure 67. Heated 48 hours at
1000°C., quenched.
Heated 10 hours at 600°C., quenched.

200X 29782
Figure 68. Heated 48 hours at
1000°C., quenched.
Heated 48 hours at 600°C., quenched.

Biscuit Metal, .01 C, Heat-Treated as Shown

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200X 27362
Figure 69. Heated 96 hours at 1000°C., quenched.

200X 29783
Figure 70. Heated 96 hours at 1000°C., quenched.
Heated 6 hours at 725°C., quenched.

200X 29784
Figure 71. Heated 96 hours at 1000°C., quenched.
Heated 10 hours at 725°C., quenched.

200X 29785
Figure 72. Heated 96 hours at 1000°C., quenched.
Heated 48 hours at 725°C., quenched.

Biscuit Metal, .01 C, Heat-Treated as Shown

274 274

Alpha Extrusion

The results of these tests are shown in Tables 12 and 13 and graphically in Figure 85. At low temperatures, some difference in hardness is noticeable between cast, alpha extruded, and gamma extruded metal. This difference becomes negligible as the alpha-beta transition is approached, except for the cast metal. The specimens of cast metal were from a large extrusion billet

200X

27372

Figure 73. As received.

200X

29786

Figure 74. Heated 6 hours at 600°C., quenched.

200X

29787

Figure 75. Heated 10 hours at 600°C., quenched.

200X

29788

Figure 76. Heated 48 hours at 600°C., quenched.

Extruded Bar 1088, .09 C, Heat-Treated as Shown

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TABLE 12: HOT HARDNESS WHEN HEATED TO TEST
TEMPERATURE AND HELD 15 MINUTES

Temperature	Alpha Extruded Metal	Gamma Extruded Metal	Cast MIT Metal
19.9°C.	251.7 ^(x)	--	--
200°C.	143.7 ^(o)	--	--
400°C.	66.1 ⁽⁺⁾	--	--
500°C.	34.7 ⁽⁺⁾ , 33.2	24.9	50.2
600°C.	19.0	16.2	28.0
604°C.*		19.0	--
650°C.	13.3	14.2	22.4
654°C.*		25.0	--
670°C.	(a)	36.0 ⁽⁺⁾ , 35.5	30.9
690°C.	35.6 ⁽⁺⁾	31.5, 30.1	52.1
700°C.	32.4	47.4	--
702°C.*		44	--
753°C.*		16	--
772°C.*		0	--

*Data from Report No. 5, MIT metal, gamma extruded.

(x) 3000 kg. load

(o) 2000 kg. load

(+) 1000 kg. load

(a) No impression. One specimen deformed, other no deformation⁽⁺⁾.

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TABLE 13. HOT HARDNESS OF ALPHA EXTRUDED METAL
HEATED TO BETA AND COOLED TO TEST
TEMPERATURE

Heat Temperature	Time at Temp., Minutes	Hot Hardness Temp.	Time to Cool to H.H. Temp.	Time at H.H. Temp.	B.H.N.
700°C.	15	600°C.	25	0	19.1
735°C.	30	600°C.	35	0	18.2
735°C.	30	650°C.	16	0	21.0
735°C.	30	660°C.	18	0	23.8
700°C.	15	670°C.	7	15	49.1
735°C.	30	600°C.	32	reheated in 27 minutes to 735°C. and repeat cycle - 0 minutes at hot hardness temp.	15.6

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FIG. 85

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from MIT which is believed to be remelted scrap of fairly low purity, and the hardness difference may result from the impurities present. Quite variable results are obtained on the hardness in the beta phase, the only consistency being that the values are all substantially greater than in the high alpha region. On alpha extruded metal no satisfactory readings were obtained at 670°C. With 500 kg. loading, no measureable deformation occurred, while at 1000 kg. one specimen was badly deformed and the other remained round. The cast specimens appear to be only partially transformed at 670°C., since a much higher hardness was found at 690°C. Gamma extruded specimens gave lower values at 690 than at 670°C., but gave a value higher than either at 700°C. It would appear that the values obtained between 662° and 700°C. are affected by the completeness of the transformation, temperatures as high as 676° having been found for the end of the alpha to beta transformation at these rates of heating. A new series of tests, in which the specimens are allowed a longer time at temperature before testing, seems indicated.

In the tests where the specimens were heated into the beta region and cooled to the test temperature, there was no evidence of higher hardness in the high alpha region, 600 to 660°C., although slight increases at 650 and 660° over that at 600°C. were evident. The hardness at 670°C. was much higher than at 660°C. These data do not indicate any explanation for failure to extrude metal treated in this manner.

It was considered that the size of specimen used for the hot-hardness tests may have been a factor which caused the results not to represent the effect observed on extrusion. Compression tests were made at 600°C. and after heating to 735° and cooling to 600°C. at 10,000-pound load on specimens 1-1/8" in diameter by 2-1/4" long. The specimen heated into beta and cooled

35
showed 29.8% decrease in length, while the other showed 11.5% decrease. Thus, when there is no restraint on the sides, the metal appeared to be plastic when cooled from the beta. The effect of section size tested does not appear to be a factor.

The effect of pressure on the alpha-beta transformation has been considered and is discussed in the section on dilatometer work. Since alpha is the more dense phase, application of pressure to metal in the beta phase, or containing beta, tends to raise the temperature of transformation or transform beta to alpha, thus favoring the presence of the plastic alpha phase during extrusion. Metal over the die opening would not be under pressure and could conceivably, under certain conditions, be the nonplastic beta phase.

Failure of alpha extrusion at Revere now appears to be lack of sufficient capacity in the press. A later trial on a few billets, which were not allowed to heat over 600°C., also failed. Since the laboratory tests indicated that alpha extrusion would require nearly the maximum capacity of the press, the increase in load caused by the greater frictional load on the commercial billets may be sufficient to exceed the press capacity. The failure to extrude on billets cooled from the beta on the laboratory press now requires further confirmation, and further laboratory tests are planned.

Coating Investigation

Diffusion-Bonding Aluminum

The bonding of the aluminum jacket to the Al-Si bonding alloy below the melting point of the latter appears to offer possibilities for minimizing attack of the jacket. Some diffusion tests have been undertaken to investigate this possibility.

36

The "sandwich technique" previously described was used, alternate samples of 2S aluminum and Al-Si eutectic being bolted together after suitably cleaning the surfaces and inserting bonding agents where desired. Heating was performed in a tube furnace with a hydrogen atmosphere at 1000°F.

Figures 86, 87, 88, and 89 show the effect of time at 1000°F. (538°C.) on diffusion at the interface between 2S Al and Al-Si eutectic. Apparently, a considerable amount of time is required at this temperature to effect good bonding, and tests at a somewhat higher temperature should be tried.

Figures 90 and 91 show the effect of introducing a copper film at the interface. The presence of copper appears to increase the rate of diffusion. Zinc and brass at the interface also increase the rate of diffusion and may introduce problems of control, since zinc tends to diffuse rapidly at localized points (Figures 92 and 93).

Further tests of this procedure are planned to show the effect of variations in time and temperature.

Hot-Pressing of Beryllium

Attempts to press end caps to finished dimensions in one operation were made by hot-pressing of beryllium powder and by hot-pressing of solid slugs. All such attempts were unsuccessful.

Beryllium powder (+80 mesh) was heated to 760°C. (1400°F.) in 4-3/4 hours under a hydrogen atmosphere in a hot-pressing die. This powder was then hot-pressed at 10,000 lbs./sq. in. for five minutes. The slug which was removed showed very poor sintering at the edges and excessive oxidation. In order to minimize the oxidation obtained during the heating operation, several tries were made to add powder under a controlled atmosphere to the hot die

38

250X
Figure 90. Heated 7 hours at
1000°F.

29799

250X
Figure 91. Heated 24 hours at
1000°F.

29800

Al-Cu-AlSi Diffusion Sample
Unetched

250X
Figure 92. Heated 24 hours at
1000°F.

29801

250X
Figure 93. Heated 24 hours at
1000°F.

29802

Al-Zn-AlSi Diffusion Sample

Al-Brass-AlSi Diffusion Sample

Unetched

39

(760°C.) with subsequent pressing at 10,000 lbs. per sq. in. The compacts so produced showed poor sintering and excessive oxidation.

Hot-pressing operations were tried with solid beryllium slugs. The temperatures used were 760°C. (1400°F.) to 787°C. (1450°F.) with pressures up to 10,000 lbs./sq. in. Little deformation of the beryllium could be accomplished at this pressure and temperatures. The temperature was the maximum obtainable with the equipment. Higher pressures could not be used, since difficulty was experienced with galling at the pressures used.

Studies of sintering compacts made by cold pressing will be continued.

Annealing Tuballoy

A considerable amount of metal has been used in the gamma extruded, quenched, and outgassed condition. Subsequent canning operations have been done at temperatures where heating above the alpha-beta transition is required. The dilatometer studies given in Report #16 show that anisotropy exists in gamma extruded and outgassed metal, but that coefficients of expansion of alpha on longitudinal and transverse samples approach the same value upon being heated and cooled through the alpha-beta transition.

In work on steel, and other metallic materials, the sample is commonly placed in a "standard condition", such as annealed or normalized, for comparison of properties. These treatments are such that the properties are reasonably independent of the previous condition of the metal. A great many tests have been made on tuballoy, nearly every one of which has been performed on metal of varying history, and, while these tests give comparisons of the data between different conditions of the metal, no basis is provided for comparisons of the effect of composition on these properties. In the case of the

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canning referred to above, it would seem highly desirable to have subjected this metal to heating through the alpha-beta transition prior to canning to minimize dimensional changes, which would otherwise occur during canning, especially since it has been shown that recrystallization occurs when so heated.

Alpha rolling or extruding appears to introduce a considerable amount of "cold" work, as indicated by hardness and tensile properties. Heating to 600°C. appears to relieve some of the stress, but has not been observed to cause recrystallization. It would not be surprising to find gradual relief of stress accompanied by dimensional changes or warping on alpha rolled metal when reheated to moderate temperatures. Such effects could probably be greatly reduced by a recrystallizing anneal before use.

With these thoughts in mind, some preliminary tests were made on annealing gamma extruded and outgassed metal, through the alpha-beta transition, followed by slow cooling. It is proposed to follow these tests by annealing other lots similarly, which are in other states of heat treatment or fabrication, to determine if such a treatment will bring all metal to a standard condition.

Figure 94 shows the change in structure found upon annealing Heat #4160 gamma extruded and outgassed bar stock at 725°C. for one hour and slow cooling.

Determination of the elastic constants and tensile properties were made on two specimens in the outgassed condition and two after the annealing treatment, as shown in Table 14.

The elastic constants compare favorably with those reported previously. The tensile properties were determined on the same specimens used for the elastic properties.

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41

2X

29518

2X

29519

25X

29803

As extruded

25X

29804

Heated 1 hour at 725°C.,
slow cooled.

Figure 94. Effect of heating gamma extruded and outgassed Heat #4160 into beta and slow cooling on microstructure.

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TABLE 14. EFFECT OF ANNEALING AT 725°C. ON TENSILE PROPERTIES

Condition of Specimen	Yield Strength, p.s.i.	Tensile Strength, p.s.i.	Elongation, % in 2"	Mod. of Elasticity*	Poisson's Ratio*
Extruded, outgassed, and annealed	12500	64000	6.0	23.2×10^6	.35
	7750	61000	5.0	25×10^6	.32
As-extruded and outgassed	12000	68400	1.5	25.5×10^6	.35
	10500	63550	1.5	25.3×10^6	.38

* Calculated at 5000 p.s.i. stress

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In making these tests, after the yield was reached the test pieces showed slip bands, and, before fracture occurred, the surface of the samples had become so rough from slip that it was not possible to get an accurate measure of the small reduction of area that occurred. During the pulling of the test, in the region between yield and complete fracture a number of sharp clicks were heard, which sounded as if some internal fractures were occurring before general fracture occurred. This was observed on all samples.

In the case of this material, the annealing treatment caused very little change in tensile properties. It is anticipated that much greater changes will occur on alpha rolled or extruded metal.

Effect of Casting Conditions on
Grain Size of 3% Al-Be Alloys

During the work on casting beryllium tubes, one tube was noted to have a fine grain size. It was considered that fine-grained metal would possibly be more desirable for the production of rolled strip to be used for end caps, and that production of a fine grain in cast tubes rather than the large radial grains ordinarily obtained would improve the properties of the tube. Accordingly, the following experiments were conducted in order to determine the factors affecting grain size of the 3% Al-Be alloy.

Every attempt made to measure the temperature of the melt failed. Beryllia, graphite, and McDaniel thermocouple protection tubes were tried. The beryllia tube would not stand the shock of being inserted into the metal even when preheated in a gas burner. The second attempt to use the beryllia tube failed in that the temperatures indicated obviously were not correct. The graphite tube reacted at relatively low temperatures and the McDaniel tube was attacked before any temperatures could be obtained.

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In the absence of temperature measurements, the heats were heated for 16, 9, and 5 minutes after they were completely melted. The successive castings were poured after waiting a prescribed interval between castings. In Heat 87, the castings were cast at intervals of 30 seconds, in Heat 88 at intervals of 15 seconds, and in Heat 93 at intervals of 8 seconds.

The first two heats, 87 and 88, were made from virgin beryllium with the aluminum being added shortly before the bath was entirely melted. Heat 93 was made from scrap alloy which was coarse grained. Scrap was used on this heat as a graphite protection tube was to be tried in it, and in case the heat was lost, the virgin metal supply would not be depleted. While this heat does not conform to the standard melting practice, it does appear to be in line. This heat differs from the first two principally in that its aluminum content may be slightly higher.

Three to four castings were made from each heat at decreasing pouring temperatures. The last pour of each heat was at so low a temperature that a skull was left in the furnace. Each casting consisted of the following sections one above the other starting with the smallest section at the bottom:

- 1-3/8 inches in diameter, 4 inches long
- 1 inch in diameter, 3 inches long
- 11/16 inch in diameter, 2 inches long
- 1/2 inch in diameter, 1-1/2 inches long
- 3/8 inch in diameter, 1-1/8 inches long
- 1/4 inch in diameter, 3/4 inch long

The data obtained on these heats are as follows:

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Heat No.	Time Superheated	Time Between Castings	Grain Size of Castings			
			1	2	3	4
87	16 min.	30 sec.	coarse radial	coarse radial	coarse radial	fine equiaxed
88	9 min.	15 sec.	coarse radial	coarse radial	coarse radial	fine equiaxed
93	5 min.	8 sec.	fine equiaxed	fine equiaxed	fine equiaxed	

The grain size of the different sections was about the same, indicating that the rate of cooling had little effect. All the coarse radial grains were about the same size, and the equiaxed grains of Heats 87 and 88 were about the same. The fine equiaxed grains of Heat 93 were a little finer than those of Heats 87 and 88.

In addition to grain size, the fractures showed dull gray areas which appeared to be intercrystalline fractures, while the bright areas appeared to be transcrystalline fractures. It is possible that the dull gray areas represent microporosity which was caused by microshrinkage or dissolved gases. These dull gray areas were only noticeable in the radial-grained fractures; however, if they existed in the equiaxed fracture, they would be difficult to distinguish.

From the results of three heats, the following conclusions are indicated:

1. Casting section sizes of $3/8$ to $1-3/8$ inches in diameter in a chill mold has very little effect on grain size.
2. Superheating above a low critical temperature is detrimental to grain size.
3. If the alloy is poured at a high temperature after superheating, the grains will be the coarse radial type, but if poured at a very low temperature after

high superheat the grains will be fairly fine and equiaxed.

4. If the alloy is not heated above the critical superheat temperature which is believed to be about 2450°F., the grains will be fine and equiaxed regardless of whether they are poured at this temperature or at a lower temperature.

The initial temperature to which this alloy can be heated and retain the fine grain may appear to limit its usefulness by not permitting sufficient superheating to be able to cast the alloy. The first casting of Heat 93 was poured in a split graphite mold which was not properly fitted together at the bottom. This permitted the alloy to flow into this crack and form a fin. On the 1/4-inch section, a fin 1.5 inches long and 0.085-inch thick was formed. Total head of metal on this fin was about 12 inches; however, the effective head must have been considerably less, as it most likely was formed before the casting was completely filled. The ability to flow this fin indicates considerable fluidity.

Two photomicrographs (Figures 95 and 96) are included showing the typical grain sizes of the superheated metal and the metal which was not heated above the critical temperature. These photomicrographs were taken of samples cut from 1/2-inch diameter sections.

The work included in this section appears to answer the original question of how to produce fine-grained alloy. However, there are a number of related questions which are not answered and which may need to be investigated:

1. In the last heat, No. 93, which produced the finest grains, a graphite protection tube reacted

47

20X

29184

Figure 95. 3% Al-Be alloy, Heat 87, Casting No. 1 -- etched in HF.
Metal superheated and poured at superheat temperature.

20X

29185

Figure 96. 3% Al-Be alloy, Heat 93, Casting No. 1 -- etched in HF.
Metal heated under critical temperature and poured immediately.

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- with the metal. It may be that the presence of carbides has an effect on the grain size. This possibility is not great, but it may bear checking.
2. It may be desirable to determine whether this low-temperature melting practice will be effective for producing fine-grained pure beryllium. However, this possibility seems fairly remote.
 3. As Heat No. 93 was not melted from virgin metal, it may be desirable to rerun this test to check to results obtained.
 4. Present data do not indicate that sulphur has any effect on the grain size, but there is no proof.
 5. It would be desirable to continue the work on a method of determining the temperature of the molten alloy and determine the critical heating temperature for virgin alloys and remelted alloys.

FUTURE WORK

The dilatometer studies are being continued.

Some further work on the nitrogen-metal system is in progress.

The work on carbide solubility is being continued.

Additional work on alpha extrusion in the laboratory is to be undertaken to determine causes for previous failures under certain conditions.

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Diffusion bonding of jackets is being studied.

Tests to confirm that heating above the alpha-beta transition produces a standard condition will be made.

HWR:HRN:LRJ:LHG:JRK:EK:HAS:ECK/LM
May 11, 1944

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