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PROGRESS REPORT

on

METALLURGY OF TUBALLOY

to

UNIVERSITY OF CHICAGO

from

BATTELLE MEMORIAL INSTITUTE

CLASSIFICATION CANCELLED

DATE FEB 18 1957 *fw*

For The Atomic Energy Commission

H. F. Cannel
Chief, Declassification Branch

Photostat Price \$ 4.80

Microfilm Price \$ 2.70

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June 10, 1943

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SUMMARY

The method for determination of hydrogen by solid extraction has been the subject of further study and it now appears that reported values are high and subject to a blank correction. It is pointed out that the method gives values which are proportional to the true hydrogen content but cannot be considered absolute values. Test results of a modification of the test method and an estimate of the blank correction are given.

Of three bars of Class 2 metal obtained for outgassing studies, one which was already low in hydrogen was, unfortunately, selected for tests. It was found that heating in hydrogen at 600°C. for periods up to 96 hours, heating in argon at 600°C. for 6 hours following heating at 600°C. in hydrogen for 120 hours, and heating in argon at 900°C. for 3 hours showed only minor variations in hydrogen content, indicating that the hydrogen content of this bar was already at a minimum value. Samples

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of this bar charged with hydrogen were outgassed in hydrogen and natural gas at 600°C. for 6 hours. The natural gas atmosphere gave lower values. However, while the hydrogen content was reduced to nearly half the original value of about .002 wt. %, it was still high enough to produce gas swells on the basis of previous tests.

The other two bars showing hydrogen contents in the neighborhood of .001 wt. % were outgassed in natural gas at 600°C. for 6 hours and, while showing some decrease in hydrogen content, the values were higher than anticipated. Four inch slugs were machined from these outgassed bars and the balance of the low hydrogen bar was forwarded to the Aluminum Company of America for jacketing.

Since the natural gas atmosphere gave such poor results on the tests above, vacuum treatment at 600°C. was recommended for commercial outgassing, with a helium or argon atmosphere as second choice.

Tests on jacketed slugs were continued. The outgassed slugs tested at 550°C. have now run 69 days with no visible signs of failure. The test at 300°C. of metal not outgassed shows satisfactory results at 38 days. Tests at 200°C. have run as long as 75 days and are continuing. Other tests have shown many poor welds which causes some uncertainty in the type of failures obtained.

Micrographs of structures found in satisfactory and poor machining bars are shown. Verification of the lamellar structure by different polishing and etching procedures was obtained. A high iron bar shows lamellar structure in the as extruded condition, indicating the possibility of a relation between the iron content and the lamellar structure.

Tensile tests at room temperature, 150 and 600°C. on a silicon alloy show substantially higher values than for the cast MH scrap metal previously tested.

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The present operation of commercial extrusion is described.

Further study of the metal-hydrogen equilibria at atmospheric and other pressures is in progress. The relation of the hydrogen analysis by solid extraction to absolute values is being investigated. Specimens are being analyzed to follow the hydrogen content through the extrusion process.

EXPERIMENTAL DATA

Hydrogen-Metal Studies

Discussion of Errors in the Hydrogen Analyses

A large amount of data on the hydrogen content of extruded bars has been obtained by the method of hot extraction at 750°C. This method, described in the last report, page 153, gives a total hydrogen value that is obtained by extrapolation and has been called the "extrapolated total hydrogen". While this analytical method does not yield absolute values, there has been reason to believe that it does give reproducible hydrogen values that are proportional to and somewhat greater than the true values.

During the period since the last report, a number of experiments have been performed to test the hot extraction method in an attempt to relate the results to some absolute basis. These experiments have been of two types: (1) The analysis of slugs that were supposed to contain a known amount of hydrogen and (2) a comparison of the hot extraction results with results obtained by a combustion method of hydrogen analysis. Unfortunately, however, the conclusions of this work are not completely satisfactory because of the belated discovery of a source of error in the hot extraction method and the suspicion of possible incomplete hydrogen

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recoveries in the combustion method.

Hydrogen "Blanks" in the Hot Extraction Method

The "blank" hydrogen obtained from the hot extraction apparatus was determined some time ago and found to be completely negligible. The walls of the silica furnace tube were always kept quite cool since the specimen, heated by induction, was suspended in the furnace by means of the thermocouple wires. It was reasoned, therefore, that the negligible blanks obtained with the cold furnace and cold specimen were representative.

Recently, however, it has been discovered that moisture adsorbed in the system is slowly evolved during the extraction, and the water vapor is reduced by the hot specimen to give extraneous hydrogen which may be considered a "blank" for the method. Unfortunately, there is no completely satisfactory means of determining this blank for each run, and the procedure at present is to minimize the amount of adsorbed water vapor by keeping the system as dry as possible when it is opened to the atmosphere and by exhausting the system for several hours before the specimen is heated. There is every reason to believe that these precautions reduce the water vapor blank to negligible amounts.

Since most of the results reported in this report were obtained by the old method, it is necessary to make some estimate of the size of the blank which must be applied to them. Table 29 gives a comparison of results showing that the old method gives hydrogen contents that range from .00005 to .00013 % higher than the new method gives on specimens that have had identical treatments. Actually the blank appears to amount to 3 to 9 cc. of hydrogen with the most likely value about 5 cc. On specimens of the usual size, this amounts to an error of .00007 wt. %.

TABLE 29. COMPARISON OF RESULTS OBTAINED ON SIMILAR SPECIMENS BY THE OLD AND BY THE NEW HOT EXTRACTION METHODS OF HYDROGEN ANALYSIS.

Sample	Previous Treatment	Extrapolated Total H ₂ , Wt. %	
		Old Method	New Method
AE-61-H2	Heated in H ₂ 46 hrs. at 800° C.		.00195
AE-61-B2	" " " " " " " "	.00200	
AE-64-K	Unknown*		.00075
AE-64-DD	Nat. Gas, 6 hrs. at 600° C.	.00080	
AE-64-FF	As extruded	.00082	
AE-64-JJ	As extruded	.00088	
AE-61-C	Heated in Argon 3 hrs. at 900° C.		.00029
AE-61-M	" " " " " " " "	.00034	

* After this specimen was run it was discovered that there was no adequate record of its previous history. It is certain, however, that it was either "as extruded" material similar to specimens FF and JJ or had been outgassed in natural gas similar to specimen DD.

Unless otherwise indicated, all the hydrogen analyses given in this report were obtained by the old method and a correction has been applied for the estimated hydrogen blank.

It should be emphasized again, however, that both the corrected results and the results obtained by the new method cannot be considered true hydrogen analyses. As pointed out in the last report, theoretical considerations indicate that the extrapolated total hydrogen values are higher than the true values by a factor of about 1.2. In any case, the extrapolated totals may be considered approximately proportional to the true hydrogen contents.

Study of Outgassing Methods

Three extruded bars of Class 2 material were received for a study of outgassing methods. The plan was to use a small amount of this material in laboratory tests which, it was hoped, would determine a satisfactory method of commercial outgassing. The remainder was to be given such an outgassing treatment and then cut into 4 inch slugs for canning tests.

Since all three bars were extruded on the same day, it was assumed that their hydrogen contents would be similar and that samples from any one bar could be used for the laboratory tests. Slugs 2-1/2" long were cut from the leading half of Bar AE-61, and were labelled A, B, C, etc., in the order of position. Piece A, from the leading end, was not used.

The results of hydrogen analyses on some of this material are given in Table 30. The material, as extruded, contained surprisingly little hydrogen, considerably less than half the hydrogen found in previous analyses of extruded bars. Since, however, previous work had

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TABLE 30. HOT EXTRACTION HYDROGEN ANALYSES OF SLUGS FROM BAR AE-61

Sample	Treatment	Extrapolated Total Wt. % Hydrogen	
		Uncor- rected	Corrected
AE-61-K	As extruded	.00041	.00035
AE-61-B	" "	.00038	.00032
AE-61-H	In H ₂ 600° C. 24 hrs.	.00046	.00040
AE-61-J	In H ₂ 600° C. 44 hrs.	.00044	.00038
AE-61-R	In H ₂ 600° C. 96 hrs.	.00041	.00035
AE-61-D	In H ₂ 600° C. 120 hrs. then Argon 600° C. 6 hrs.	.00039	.00033
AE-61-M	In Argon 900° C. 3 hrs.	.00034	.00027
AE-61-B2	In H ₂ 800° C. 46 hrs.	.00200	.00194
AE-61-H2	In H ₂ 800° C. 46 hrs.	.00195	.00195*
AE-61-D2	In H ₂ 800° C. 46 hrs., then in H ₂ 600° C. 6 hrs.	.00171	.00164
AE-61-J2	In H ₂ 800° C. 46 hrs. then Nat. Gas 600° C. 6 hrs.	.00125	.00118

* This analysis was run by the new method, and no correction has been applied. All others, run by the old method, have been corrected by deducting an estimated hydrogen blank of 5 c.c. from the extrapolated total gas.

all been done on reprocessed scrap, it was assumed that low hydrogen was characteristic of the Class 2 material. Later analyses of the other two bars of Class 2 material show that this assumption was not justified.

An attempt was made to obtain a base line for the hot extraction analytical method by analyzing slugs that had been held for long periods of time at 600°C. in hydrogen at atmospheric pressure. The hydrogen solubility curve, Figure 72, page 147 of the last report, shows that at least .00027% hydrogen by weight should be dissolved in the metal when equilibrium is reached under these conditions. Accordingly, several slugs were heated in purified hydrogen for various lengths of time. All specimens were started at the same time and heated up over 500°C. in argon to minimize hydride formation. The furnace tube was opened periodically, after a brief purging with argon, to remove a specimen which was allowed to cool in air. The specimens were cleaned of scale by sandpapering.

The results of the analyses of these specimens, H, J and R, are given in Table 30. At first sight, it appears that the treatment first added hydrogen and then slowly removed it until the hydrogen content approached that of the original material. This unlikely conclusion is not justified because (1) the values for the hydrogen treated and the as extruded material all lie within .00008% which may fall within the experimental error of the method and (2) surface contamination may have increased the apparent hydrogen content of the hydrogen treated specimens.* The only safe conclusion is that the hydrogen treatment did not produce a

*A note in the original record states that H was imperfectly cleaned of scale, J was cleaned somewhat better, and R was very well cleaned. At the time, careful removal of all traces of scale seemed unimportant since it was assumed to be oxide. It seems possible, however, that the scale and metal immediately below it may have contained traces of hydride.

significant change in the hydrogen content of the material.

The apparent hydrogen contents of the specimens lie appreciably above the .00027% predicted by the solubility curve. If, however, we recall that the solubility data represent lower limits of hydrogen solubility and that theoretical considerations indicate that the extrapolated total hydrogen is too high by a factor of 1.2, the discrepancy is found to be small. Applying this latter factor, the .00035% obtained with Specimen R becomes .00029%.

Specimen D, after heating in hydrogen at 600°C. for 120 hours, was then held in argon at 600°C. for 6 hours, and Specimen M, as extruded, was held in argon at 900°C. for 3 hours. These two treatments appeared to produce a small, but probably not significant, reduction in hydrogen content. See Table 30.

These results obtained on samples of Bar AE-61 showed that this bar of Class 2 material was very low in hydrogen and that it did not need outgassing. Treatments in argon had no appreciable effect, and the hydrogen content was not appreciably greater than the minimum solubility. Consequently, hydrogen could never be evolved to cause a gas swell of a jacketed slug.

Unfortunately, these conclusions, valid enough for Bar AE-61, were later found to be invalid for the material as a class. It turned out that the other two bars contained two to three times as much hydrogen as Bar AE-61, and that the choice of the latter for experimental outgassing tests had been extremely unfortunate.

In order to conserve material, further outgassing tests were conducted on four used slugs of Bar AE-61. These were charged with hydrogen by heating at 800°C. in hydrogen at atmospheric pressure. Accord-

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ing to the solubility curve, this treatment would be expected to bring the hydrogen content to at least .0013%. New and more accurate solubility data, as yet incomplete, indicate that this figure is considerably higher and is approximately .00165%.

The specimens were placed in the cold furnace tube, heated above 600°C. in argon, purified hydrogen was then run in and the specimens held in a slow stream of hydrogen for 46 hours at 800°C. The furnace was then cooled to 700°C., rapidly flushed with argon and the specimens quenched in water from 650° to 700°C. All specimens had one or two longitudinal cracks after this treatment.*

The results of analyses on these specimens are given in Table 30. The charged specimens, B2 and H2, analyzed .00195%. If this figure is divided by 1.2, the value .00162% is obtained in surprisingly good agreement with the best value from recent solubility data. Specimens D2 and J2, after outgassing treatments in hydrogen and natural gas, showed some reduction in hydrogen content but not enough to be satisfactory in 6 hours treatment time. As expected, natural gas is more effective than hydrogen.

Outgassing Treatment of Metal for Jacketing Tests

Tests described in the preceding section showed that Bar AE-61 was so low in hydrogen that it did not need outgassing. Accordingly, this bar was given no outgassing treatment. Analyses of pieces from Bars AE-64 and A-1318, however, showed them to be relatively high in hydrogen (see Table 31) and they were given an outgassing treatment in natural gas 600°C. for 6 hours. The outgassing treatment consisted of

*The first samples used in the study of hydrogen contents were from bars that were cracked in a similar manner. They were fairly high in hydrogen and had been quenched immediately after extrusion from about the same temperature.

TABLE 31. HOT EXTRACTION HYDROGEN ANALYSES OF VARIOUS MATERIAL

Sample	Description or Treatment	Extrapolated Total Wt. % Hydrogen	
		Uncor- rected	Corrected*
AE-64-FF	As extruded	.00082	.00074
AE-64-JJ	" "	.00088	.00080
AE-64-DD	In Nat. Gas 600° C. 6 hrs.	.00080	.00068
A-1318-CC	As extruded	.00089	.00080
A-1318-JJ	" "	.00118	.00106
A-1318-D	In Nat. Gas 600° C. 6 hrs.	.00068	.00056
Chipman #3	Rod, hot rolled 1100-1200° F.	.00038	.00032
#4	" " " " " "	.00050	.00044
#5	" " " " " "	.00035	.00029

* Differences between the uncorrected and corrected values depend upon the mass of the specimens which varied considerably in this group of tests.

placing the cold bars in a furnace at 600°C. in a natural gas atmosphere, as this appeared to be the most desirable method of outgassing at the time. The bars attained temperature in 20 to 30 minutes but passed through the temperature range of the hydride formation in the natural gas atmosphere which may have contained a considerable proportion of hydrogen since the gas was in contact with a hot metallic muffle. Total time in the furnace was 6 hours and the bars were air cooled. The reduction in hydrogen was not so large as was hoped and expected.* Whether the hydrogen is sufficiently low is uncertain.

Hydrogen Content of Hot Rolled Rod

Three specimens of material hot rolled at 1100-1200°F. were sent in for analysis at the request of Dr. Chipman. The results, given in Table 31, show this material to be as low in hydrogen as any that have been analyzed.

Recommendations for Commercial Outgassing

Treatment in natural gas at 600°C. does not appear to be an effective outgassing procedure for reasons that are not known. Possibly the hydrogen partial pressure at the surface of the metal is too high. Possibly the layer of hydride that forms on heating up decomposes to diffuse in as well as out of the metal and thereby effectively slows down the rate of diffusion. In any case, the only safe procedure at the moment is to use an atmosphere relatively low in hydrogen. Vacuum, helium, or argon were recommended for commercial outgassing. When experiments, now in progress, on the pressure dependence of hydrogen solubility are

*The natural gas treatment of Specimen AE-61-J2, described in the previous section, had not been completed at the time Bars AE-64 and A-1318 were treated.

completed, it will be possible to fix the acceptable limits of hydrogen partial pressures more precisely.

Tests on Aluminum Jacketed Slugs

The heating of jacketed metal, which was under test, was continued and Table 32 shows the present state of these tests. Only a few new tests were undertaken. Several of the later tests resulted in failures attributed to poor welding. Torch welding of the assemblies used is rather difficult and it was necessary to have a new welder complete the closure on some of the later jackets.

It will be noted that the two assemblies outgassed 45 hours at 600°C. in argon have now been on test 69 days with no outward signs of failure. Slugs 19K and 19J showed oxidation failures after 37 days which are attributed to poor welds. The two comparative slugs which received a one hour outgassing treatment appear satisfactory at 50 days. Slugs of AE-64 and A-1318 which were outgassed in bar form were jacketed and placed on test as a check on the amount of H_2 permissible. As noted in the table, AE-64 showed an oxide swell in two days while A-1318 appears satisfactory after 5 days. Tests were run on AE-61, a bar which showed low H_2 as received, and these slugs appear to be satisfactory after 15 days. The two slugs of Bar #9 at 300°C. show no changes after 38 days indicating that the H_2 pressure is too low at this temperature to cause failure.

Jackets classed as permitting oxide swells must be interpreted as uncertain in regard to gas swells since a leak sufficient to allow oxidation would allow gas to escape. It may be possible that gas pressure

TABLE 32. TESTS ON ALUMINUM JACKETED SLUGS IN AIR

Specimen or Test No.	Pretreatment	Type of Closure	Metal	Temp. of Test	Date Started	Time on Test	Type of Failure
11-1	45 hrs. at 600°C. in A	Welded	Bar 9	550°	4/2	69 days	one
11-2	" " " " " "	"	"	"	"	"	"
19V	1 hr. at 600°C. in A	"	Bar 19	"	4/21	50	None
K	2 " " " " " "	"	"	"	"	37	Oxide swell-leak
2	1 " at 600°C. in Vac	"	"	"	"	50	None
J	2 " " " " " "	"	"	"	"	37	Oxide swell-leak
AE61G	None		AE61	550	5/26/43	15	None
AE61M	"		"	"	"	15	None
AE64GG	6 hrs. in furnace, 600°C., Nat. Gas	Welded	AE64	"	6/3	2	Oxide swell-leak
Al318-K	" " " " " " " "	"	Al318	"	6/3	5	None
9-3	None	"	3/8" rod	"	4/1	71	None
300-1	"	"	Bar 9	300	5/3	38	None
300-2	"	"	"	"	"	38	"
2	"	"	"	"	3/27	75	None
7	"	"	"	"	3/29	73	"
13	45 hrs. at 600°C. in A	Brazed	"	"	4/6	65	"
8-2	None	Welded	3/8" rod	"	3/30	72	"
8-3	"	"	"	"	"	72	"

caused the leak but this is not considered probable in view of the known poor welding conditions and the appearance of the jackets. However, it is possible that sufficient gas was present to cause a gas swell had the jacket been free from leaks.

These tests indicate that outgassing in argon at 600°C. for 45 hours on Bar #9 metal, which produced a gas swell in the as received condition, is an effective outgassing treatment. Tests on Bar #19 as extruded failed to produce a gas swell at 550°C. because of a poor weld but a specimen overheated in the 300°C. test did fail by gas swelling and hydrogen analysis indicated about the same H₂ content as Bar #9. Outgassing of samples of this bar in lead, argon and vacuum for 1 and 2 hour periods at 600°C. has given uncertain results, probably caused by poor welding, for treatment in lead and 2 hour treatments in argon and vacuum. However, the 1 hour treatments in argon and vacuum appear to give satisfactory results up to 50 days which agrees with results obtained on the gas analysis, and indicates that a treatment period as short as one hour may effectively outgas the metal. The outgassing treatment for Bars AE-64 and A-1318 is still to be verified by tests on slugs jacketed by ACA.

Metallography

In studying the causes for variations in machinability, a number of micrographs were prepared showing the size and distribution of the carbide inclusions. For this purpose, the mechanical polishing technique previously described was used and the specimens photographed in the as polished condition.

Figure 79 shows the inclusions in a bar which was one of a group of bars showing poor machining properties but which may have been better than the group average. The inclusions are quite small and fairly well distributed. Figure 80 shows the inclusions in a bar which gave good machinability. The inclusions are somewhat larger and more numerous in line with the higher carbon content. The inclusions in Bar #A-1089, which showed poor machinability, seem more numerous than in Figure 79 at about the same carbon content. Bar #A-1025, Figure 82, which showed good machinability and carbon at .07%, shows larger inclusions but they are not so numerous as in A-1088 with .09 carbon.

In view of some doubts expressed by other investigators as well as some difficulties in this work in regard to the polishing and etching treatments used, some further work was undertaken in trying other techniques. It was suggested that the lamellar structure, previously reported, may result from over-etching or perhaps have been caused by a flowed surface layer. Since other investigators seem to prefer electropolishing, specimens were prepared by mechanical polishing through 400 "wet or dry" paper followed by 5 to 10 seconds electropolish in the perchloric acid and acetic anhydride bath previously described. The electropolishing treatment permitted observation of large anisotropic grains under polarized light and all specimens were polished to this surface before etching. Following this, on the same specimens used for the mechanically polished and etched structures, the specimens were etched in the 1:1 nitric-acetic acid, and repolished and re-etched electrolytically in oxalic acid.

Figures 83, 84, and 85 show micrographs obtained under the three sets of conditions. The lamellar structure persists in all three. This particular specimen was from a bar which showed poor machinability and was

Figure 79

21813

Bar No. A1065, as polished
Carbon 0.06%
200 X

Figure 80

21814

Bar No. A1088, as polished
Carbon 0.09%
200 X

Figure 81

21815

Bar No. A1089, as polished
Carbon 0.05%
200 X

Figure 82

21816

Bar No. A1025, as polished
Carbon 0.07%
200 X

Size and distribution of carbide particles in various bars of stock.
Specimens mechanically polished by procedure previously described.

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Figure 83

21817

Polished mechanically - Etched in
Nitric and Acetic Acids

Figure 84

21818

Electropolished - Etched in Nitric and
Acetic Acids

Figure 85

21819

Electropolished - Etched in Oxalic
Acid electrolytically

Effect of varying the
metallographic procedure
on the structure of a
specimen of Bar No. A1089
furnace cooled from 1700°
F. This bar showed poor
machinability. Hardness
after furnace cooling
85R_B.

500 X

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given a furnace cool from 1700°F to determine the structure and hardness after this treatment.

Figures 86 and 87 show the structures obtained on a bar showing good machining properties after a similar furnace cooling treatment. The lamellar structure was not obtained but a structure showing grain boundary precipitation resulted. A similar structure was obtained on Bar A-1025 which also showed good machinability, shown in Figure 88.

The structures shown in Figures 89 and 90 were obtained from a furnace cooled specimen of a bar from a group of four which were classed as poor machining. These structures are similar to those of the bars of good machinability above and the hardness was similar, indicating the possibility that this bar may have been confused with the other three bars in the group.

The last four micrographs are all from the same bar which appeared to have regions of different structure in the as extruded condition. These samples were mechanically polished and etched in nitric and acetic acids. The variable structure indicates that the bar was close to the lower transition when quenched after extrusion, part of the metal cooling sufficiently rapidly to suppress the transformation partially. The development of the lamellar structure in most of the section seems to indicate that cooling was slow enough to allow formation of the lamellar structure. This bar was extruded from Ingot W8 known to be contaminated with iron and which also exhibited the poorest machinability of any bars tested. The polygonal grains shown in Figure 94 have previously been shown in metal contaminated with Fe by Highrite and Lilliendahl in Trans. AIME, vol. 117, p. 381, 1935.

Present information indicates that mechanical polishing with

Figure 86

21820

Bar No. A1088 - Electropolished and etched in nitric and acetic acids

500 X

Figure 87

21821

As in Figure 86 but etched electrolytically in oxalic acid

500 X

Figure 88

21822

Bar No. A1025, electropolished and etched in nitric and acetic acids

500 X

Structures of two bars showing good machinability taken on specimens after furnace cooling from 1700° F. Both etches reveal similar structures for Bar No. A1088. These structures are very similar to that of Bar No. 1065. Hardness of both bars after furnace cooling 95RB.

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200 X

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Figure 89

Electropolished and
etched in nitric and acetic
acids

200 X

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Figure 90

Electropolished and
etched in oxalic acid
electrolytically

Specimen of Bar No. A1065, furnace cooled from 1700° F. One of a group of bars classed as poor machining.

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Figure 91

21825

Section near surface

500 X

Figure 92

21826

Section in intermediate zone

500 X

Figure 93

21827

Central zone

500 X

Figure 94

21828

Same as Figure 93

200 X

Bar W8 as extruded containing 2320 p.p.m. Fe which showed variable structure in the specimen examined. Etched in nitric and acetic acids.

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the nitric acetic acid etch will reveal satisfactory structures but an electropolish provides a more rapid method of attaining the final polish. Mechanical polishing permits more satisfactory examination of inclusions since the electropolishing method tends to attack the inclusions. The nitric-acetic acid etch seems more satisfactory than electrolytic oxalic acid because of greater ease of manipulation.

In line with the hardness values previously given, the microstructures of furnace cooled specimens appear to indicate differences in structure which relate to machinability although this picture is not clear cut and requires further confirmation. Present studies indicate that poor machining bars give a lamellar structure on furnace cooled specimens. The structure of the metal high in iron may indicate that iron produces the lamellar structure although more study is required to establish this relation.

High Temperature Strength of Silicon Alloy

Dr. Creutz forwarded three silicon alloy rods $3/4$ " dia. x $3-1/2$ " under shipping memorandum of May 19, 1943, for tests of the high temperature tensile properties. These bars were assumed to be in the as cast condition although some machining or grinding had been done on parts of the surface. In one tensile bar, a blow hole was encountered in the threads which necessitated machining this bar to a shorter length than standard. This specimen was tested at room temperature as shown in Table 33. The specimens were standard tensile test bars for $1/4$ " reduced section.

TABLE 33. HIGH TEMPERATURE STRENGTH OF Si ALLOY

Material	Temp. °C.	Tensile Strength p.s.i.	Yield Strength 0.2% set p.s.i.	Per Cent Elongation	Per Cent Reduction Area
Si alloy	room	73,500	67,350	0	nil
" "	150°	74,800	66,800	0	0.31
" "	600	25,300	19,800	9.1	6.9
MH metal cast	room	52,500	43,000	1.8	0.3
	150	27,130	brittle	1.7	none
	600	11,720	8,900	17.9	31.3

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The data previously obtained on cast MH scrap metal are included for comparison. The silicon alloy shows some superiority in strength at all three temperatures, being less affected at 150°C. than the MH scrap metal. The ductility of the silicon alloy at all temperatures was lower than the comparison metal.

Commercial Extrusion

Six billets for experimental study have been extruded since the report summarizing the development of the commercial extrusion process. Three of these billets were mentioned in the previous report in connection with the Tantung hard-faced die. One of the six billets was a high iron billet, 2320 p.p.m., (W8) supplied by Ames.

This billet extruded very poorly, yielding only 51 pounds of scabby rod from a 92 pound billet. Billets removed from the furnace before and after this billet were satisfactory so that it is believed that the temperature was in the proper range for extrusion. It, therefore, appears that iron contents in this range are too high for satisfactory extrusion.

This high iron bar had the poorest machinability of any of the metal tested. It was necessary to reduce the spindle speed from 229 to 68 r.p.m. and even at that speed it was barely possible to obtain one piece per grind.

While a new billet heating furnace with a single muffle was suggested, it was concluded that its construction could not be justified. The present furnace was then operated until a leak in the water cooling jacket of the atmosphere lead-in pipe occurred. At this time, the muffles

were rebuilt using silicon carbide for the side walls and increasing the width to 12" in order to accommodate longer billets. New doors were installed which open by horizontal movement on rollers. The furnace was placed in operation again June 9th and, except for the roof falling in, has been running more satisfactorily than before, heating billets at the rate of about 28 per hour.

Runs of 200 extrusions per die are common. This appears to be satisfactory performance. Six dummy blocks are used in rotation during a day's run and can be used for more than one day's operation. The original cylinder liner failed by enlarging at the die end, allowing metal to flow between the dummy block and liner.

The bars are rolled while hot to straighten but are quenched at somewhat higher temperature than formerly. Quenching too hot causes more warpage and the bars are allowed to reach a more or less uniform cherry red color before quenching.

FUTURE WORK

A comparison of hydrogen analyses by the hot extraction method and by a combustion method is in progress. A more accurately determined hydrogen solubility curve is being obtained. Preliminary results agree well with the old curve except at temperatures above 750°C. The pressure dependence of hydrogen solubility is also being studied.

Specimens from cast billets and the rods extruded therefrom are being analyzed to follow the hydrogen content through the extrusion process.

The pressures and temperatures necessary to produce swelling of aluminum jackets and the cause of swelling in the temperature range 300 to

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450°C. are to be studied.

The relation of microstructure to machinability is still under study and further work on metallographic techniques is anticipated.

An investigation of extrusion and centrifugal casting of beryllium has been started.

June 10, 1943

HWR: LHG: HRN:va

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