UNCLASSIFIED**PROGRESS REPORT**

on

METALLURGY OF TUBALLOY

to

UNIVERSITY OF CHICAGO

from

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May 10, 1943

SUMMARY

A study of the hydrogen-hydrocarbon equilibria indicates the temperature range of 450 to 625 °C at minimum hydrogen content and, therefore, most suitable for outgassing.

A similar experiment with nitrogen shows that this gas is absorbed above 450°C and that excessive pressures are probably not attained below this temperature.

Specimens outgassed at 600°C for 1 to 3 hours in argon and a "commercial" vacuum are showing satisfactory behavior when tested at 550°C after jacketing and show that the initial hydrogen content is reduced to about half the original when subjected to the analytical procedure used. Two aluminum jacketed slugs which were outgassed at 600°C for 45 hours have now been on test at 550°C for 38 days with satisfactory results. Two similar specimens, not outgassed, are satisfactory after 7 days at 300°C.

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Differences in machinability in bars of Class 1 metal have been observed and attempts to trace the differences are described. The hardness of furnace cooled metal appears to provide a means of separating bars which are poor machining.

Tests on the effect of boron additions to cast iron indicate the desirability of annealing to obtain machinability and that such castings may be readily produced.

Preliminary tests on flash welding indicate that good welds can be produced on existing commercial welding machines.

Hot hardness tests in the beta region confirm the drop hammer tests in showing some forgability below the upper transition.

Some notes on extrusion die materials and metallography are included. *end*

Work in progress includes the establishing of a suitable outgassing procedure and determining the causes of poor machinability. In connection with the latter, the metallographic work is being continued.

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EXPERIMENTAL DATA

Gas-Metal Studies

The preliminary investigation of hydrogen in extruded bars, as presented in the last report, indicated that the evolution of hydrogen might be the cause of gas swells in jacketed slugs. Extruded bars were found to contain about .001 wt. % hydrogen or a volume of gas, at atmospheric pressure and room temperature, about twice that of the metal. If all, or even a moderate portion, of this gas were evolved from a jacketed slug, the pressure would be sufficient to cause swelling. The actual amount of hydrogen that would be evolved at various temperatures depends upon the amount present and on the solubility. Consequently, experiments were undertaken to determine the amount of hydrogen contained in the metal in equilibrium with hydrogen at one atmosphere pressure at various temperatures.

Hydrogen-Metal Equilibria

The equilibria studies were made with the apparatus shown schematically in Figure 71. The manometer and connecting tubing were made of capillary glass. For some of the low temperature work, the specimen tube and its connecting capillary were made of Pyrex, but, for the higher temperatures, a fused silica specimen tube was used and was connected to the glass system by a short length of rubber tubing. By means of the three-way stopcock, the system could be evacuated, or hydrogen could be added or subtracted from the system through a burette. Ordinary tank hydrogen was used without purification except drying.

Calibration experiments were performed to determine the "hot

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FIG.71 APPARATUS USED IN HYDRIDE — METAL AND SOLUBILITY STUDIES. ALL CONNECTING TUBING IS SMALL BORE CAPILLARY.

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volume" and the "cold volume" of the system. The capillary manometer provided a sensitive means of determining whether the metal was evolving or absorbing hydrogen at a given temperature, and a calibration of the capillary provided a measure of small changes in the volume of gas in the system. With these calibration data and a record of the volume of gas added or subtracted through the burette, it was possible to determine the amount of hydrogen contained in the metal at any temperature when the metal was surrounded by hydrogen at atmospheric pressure. This "hydrogen content" was measured from an arbitrary base content which may or may not have been very close to zero.

The specimen was heated in a furnace designed to maintain very uniform temperature. Thermocouples fastened to the outside of the specimen tube were connected to a potentiometer controller which maintained the temperature constant to $\pm 1^{\circ}\text{C}$ and to a semi-precision potentiometer for reading the temperatures.

Some preliminary experiments were made with a metal specimen in the form of a solid rod. Although these tests provided information regarding trends, the shape of the specimen was unsatisfactory for attaining equilibrium in a reasonable length of time. Subsequent experiments were made with metal (B-1) in the form of rolled strips approximately .05 inch thick.

Hydride- With increasing temperature, the metal starts to absorb hydrogen rapidly to form the hydride at 290°C and decomposition of the hydride against a hydrogen pressure of approximately one atmosphere (actually 736 mm. Hg.) starts at about 429°C . These conclusions are represented graphically in the upper portion of Figure 72. The amount of hydrogen necessary to convert the 10 gm. sample of metal to the hydride

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FIG. 72 - SOLUBILITY OF HYDROGEN IN THE METAL IN EQUILIBRIUM WITH HYDROGEN AT BAROMETRIC PRESSURE. (741 TO 746 mm Hg)

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was not measured. The value of 1.06% hydrogen for the hydride was obtained by allowing the hydride to decompose above 450°C against one atmosphere hydrogen pressure and measuring the volume of gas evolved. Since 1.06% hydrogen represents a composition of about $MH_{2.5}$, it is doubtful that all the gas was evolved under these conditions.

The mercury manometer was not sufficiently sensitive to permit a determination of the decomposition pressure of the hydride in the stable range below the decomposition temperature. Apparently, below about 420°C, the hydride is stable at hydrogen pressures greater than 1 mm Hg.

Hydrogen Solubility in the Range 450° to 950°C- The lower portion of Figure 72 gives the results of measurements of the solubility of hydrogen in the metal at higher temperatures. The silica specimen tube was filled with small pieces of the B-1 metal. The system was evacuated while the metal was heated to about 500°C. After a period of pumping on the system, measured volumes of hydrogen were admitted to the system through the burette to maintain the local barometric pressure which varied, during the course of the measurements, from 741 mm to 746 mm. Each circled point on the curve represents an approximate equilibrium attained by holding the temperature constant until there was no appreciable change in volume at constant pressure. The weight per cent of dissolved hydrogen was computed from the difference between the total mass of gas admitted to the system and the mass that remained in the gas phase at each equilibrium point. The points in the range from 775°C to 950°C were taken both on the way up and on the way down. All others were taken on the way down. The crosses in the region 400° to 450°C were non-equilibrium values taken while the sample was absorbing hydrogen at a rather rapid rate. The three points lying below the main curve between 500° and 600°C

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were taken in an independent run in which the system was re-evacuated and a measured amount of hydrogen again added to bring the system to equilibrium.

The curve undoubtedly shows the approximate shape of the true solubility curve. Since, however, there is no assurance that the specimen started with zero hydrogen content, there is no reason to believe that the hydrogen contents are correct, but the values given do represent lower limits. In other words, the true solubility curve may lie above the one given in Figure B, but it cannot be lower.

The curve has steep slopes in the neighborhood of 760° to 770°C and between 640° and 650°C . These are probably associated with the allotropic transformations known to occur near these temperatures. The lower transformation seems to have been suppressed despite the moderately slow rate of cooling.

The solubility curve has a broad minimum with very little change from 450° to 630°C . On reducing the temperature, there was no appreciable absorption of hydrogen until the temperature approached 400°C when the hydride seemed to form with considerable rapidity. Once this absorption started, it was not checked by raising the temperature to 440°C .

Practical Implications of the Solubility Curve- If a jacketed metal slug contains no more hydrogen than corresponds to the minimum of the solubility curve, hydrogen evolution alone cannot cause a gas swell because hydrogen cannot be evolved to build up a partial pressure greater than atmospheric. If, however, the metal contains appreciably more than this minimum hydrogen content, hydrogen evolution may cause a gas swell when the slug is heated in the range 450° to 650°C . When slugs are heated

in the range 280° to 450°C, hydride formation will prevent the evolution of hydrogen even though the gas may have been present in very large quantities.

When surface layers are machined off before jacketing, hydride films left from previous heat treatments will be removed. Consequently, the most reasonable source of hydrogen in amounts sufficient to cause a gas swell is solution in the metal at temperatures above 650°C. Heating in natural gas at temperatures near 1000°C, as is done in heating billets for extrusion, is undoubtedly a source of appreciable hydrogen solution. This conclusion is supported by analyses reported in the previous report.

The solubility curve suggests methods by which the hydrogen content of the metal may be reduced. Heating in the range 450°C to 625°C in any inert atmosphere, even pure hydrogen, will be expected to reduce the hydrogen content to harmless values. Lowering the hydrogen partial pressure of the furnace atmosphere will increase the rate of evolution, but the diffusion rate at these temperatures may be the principal limitation in attaining rapid outgassing. Higher rates of outgassing can be obtained at higher temperatures, provided a sufficiently low hydrogen partial pressure is maintained. It is uncertain how low this hydrogen pressure must be, because, thus far, the pressure dependence of the solubility curve has not been determined.

Absorption of Nitrogen

Although hydrogen may not evolve to sufficient pressure to cause a gas swell, there is still the possibility that other gases enclosed in the can may expand with rising temperatures to help produce a swell. Oxygen will probably react with the metal at moderate temperatures with a resulting lowering of the pressure. The behavior of nitrogen appeared

less certain, and it seemed desirable to investigate it briefly.

The hydrogen solubility apparatus containing metal in the form of thin strips was exhausted at room temperature, filled with nitrogen to barometric pressure and closed so that a fixed amount of nitrogen was used in the subsequent experiment. The metal specimen was heated, and the pressure in the system was followed as the temperature rose continuously. No attempt was made to determine equilibrium conditions.

The resulting data are contained in Figure 73 in which the temperature of the specimen and the mass of nitrogen absorbed by the specimen are plotted against the time. No absorption was observed until the temperature reached 450°C after which the gas was rapidly absorbed until the pressure in the system was reduced to about 10 mm Hg. at 900°C. When the system was pumped down at 940°C and then shut off from the pump, the pressure rapidly rose to about 10 mm, a value which probably represents the decomposition pressure of the nitride at this temperature. The apparent minimum in the curve in the neighborhood of 450°C is the result of differential expansion of the metal and silica tube, an error for which correction was not made.

This experiment shows that above 450°C, nitrogen is absorbed and cannot, therefore, build up dangerous pressures. Below this temperature, nitrogen partial pressures considerably greater than one atmosphere might be maintained. In any case, the maximum pressure to which entrapped air could build up on heating from room temperature to 450°C would be about 23 lbs. per sq. in. gage pressure, and this would probably be considerably relieved by reaction of the metal with oxygen.

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Figure 73.

Nitrogen Absorption as a Function of Time and Temperature. The mass of gas absorbed is given in arbitrary units and computed from the expression:

$$\text{Mass Absorbed} = 0.182 \cdot P \left(\frac{V_H}{T_H} + \frac{V_C}{T_C} \right)$$

Where P is pressure in cm.Hg, V_H and V_C are the hot and cold volumes in cc, and T_H and T_C are the hot and cold temperatures in °K.

Study of Hydrogen Degassing Methods

Samples of Bar 19 were machined into 1.1 in. diameter slugs about $2\frac{1}{2}$ in. long. These were given various treatments to determine possible methods of reducing the hydrogen content of extruded rods. After these outgassing treatments, representative samples were analyzed by the solid-evolution-in-vacuum method.

This analytical method, briefly described in the last report, consists of heating the specimen by high frequency induction in the evacuated silica tube of a vacuum fusion apparatus. The specimen is suspended in the tube by two chromel-alumel thermocouples peened into the ends. The specimen is raised from room temperature to 750°C in 2 to 3 minutes and then held at this temperature for $2\frac{1}{2}$ to 3 hours. During this period, the evolved gas is pumped from the furnace tube by a high speed diffusion pump and collected in test tubes over mercury. The gas evolved in various periods of time, usually 30 min. increments, is analyzed volumetrically. In practice, it has been found unnecessary to analyze each 30 min. sample because the evolved gas is almost entirely hydrogen and blank corrections are very small.

If the total hydrogen collected at various times is plotted against the reciprocal of the square root of the time, it is found that the points, after the first 1 to $1\frac{1}{2}$ hours, lie very nearly on a straight line as shown in Figure 74. The straight line portion of these curves have been extrapolated to infinite time and the "extrapolated total hydrogen" taken as the hydrogen content of the sample. Theoretical considerations indicate that the extrapolated value is proportional to the true value and probably exceeds it by a factor of about 1.2.

Analytical results obtained on some of the outgassed specimens

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are summarized in Figure 74. The sample of Bar 19 as extruded gave an extrapolated total of about .001% hydrogen. Slugs that were held for about an hour at 600°C in argon, in molten lead, or in a rather poor vacuum gave approximately half this value. A bar of metal that had been hot-rolled at 1100° to 1200°F and that was sent in by Dr. Chipman gave about .0003% hydrogen.

From these results, it may be concluded that moderate periods of heating at 600°C in argon or a "commercial vacuum" will reduce the hydrogen contents to the neighborhood of .0005%. Whether this is low enough to be satisfactory is uncertain at present. Heating in lead is unsatisfactory because the results depend upon the depth of immersion and also because the molten lead appears to attack the metal locally.

Tests on Aluminum Jacketed Metal

The tests on aluminum jacketed slugs in progress were continued. Of these Tests 11-1 and 11-2 at 550°C in air in Table 24 have now run 38 days without visible failure. Those under Tests 12-1 and 12-2 show failure although the metal was subjected to a similar outgassing treatment. These failures are attributed to leaks in the joints as the welder had difficulty in using the brazing material. These tests seem to indicate that the treatment of 45 hours at 600°C in argon prevented gas swells.

A series of samples from Bar 19 were treated at 600°C in lead, argon, and vacuum. Those treated in argon and vacuum appear to be satisfactory after 19 days at 550°C (Tests 19V, 19K, 19Z, 19J). The untreated bar and those treated in lead gave failures of uncertain character. The lead treatment at 600°C caused some difficulty because of alloying. A similar slug from Bar 19 at an intended temperature of 300°F showed a gas

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FIG. 74 - RESULTS OF HYDROGEN ANALYSES BY VACUUM EXTRACTION AT 750°F

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TABLE 24. TESTS IN AIR ON ALUMINUM JACKETED SLUGS

Test No.	Pretreatment	Type of Closure	Metal	Temperature Of Test °C.	Date Started	Time On Test	Type of Failure
11-1	45 hrs. at 600° in	Welded	Bar #9	550°	4/2	38 days	No failure
11-2	Argon		"	"	"	"	"
12-1	"	Brazed	"	"	4/6	9 days	Oxide swell - leak
12-1	"	"	"	"	4/6	20 days	Oxide swell - leak
19D	None	Welded	Bar 19	"	4/22	4 days	Uncertain
19U	1 hr. at 600°C. Lead	"	"	"	4/21	3 days	"
191	2 " " " " "	"	"	"	"	1 day	Specimen dropped causing leak
19V	1 hr. at 600°C., A	"	"	"	"	19 days	No failure
19K	2 " " " " "	"	"	"	"	"	"
19Z	1 hr. at 600°C., Vac	"	"	"	"	"	"
19 J	2 " " " " "	"	"	"	"	"	"
9-1	None	Spun and welded	3/8 rod	"	4/1	7	Removed for micro examination
9-2	"	" " "	"	"	"	31	" " " "
9-3	"	" " "	"	"	"	52	None
15	"	Welded	Bar 9	300	4/8	5	OK at 5 days, Furnace over- heated
19G	"	"	Bar 19	300	4/16	3	Gas swell, Specimen overheated
300-1	"	"	Bar 9	300	5/3	7	No failure
300-2	"	"	"	300	"	7	" "
2	"	Welded	Bar 9	200	3/27	44	" "
5	"	"	"	"	3/29	14	Oxide swell, Bad weld
7	"	"	"	"	3/29	42	No failure
13	45 hrs. at 600°C., A	Brazed	"	"	4/6	34	" "
8-1	None	Spun and welded	3/8 rod	"	3/30	13	Oxide swell, Bad tubing
8-2	"	" " "	"	"	"	41	No failure
8-3	"	" " "	"	"	"	41	" "
14	"	Spun only	"	"	3/25	19	Oxide swell, Poor closure

swell when the furnace became over heated. A sample of 3/8" rod in aluminum (Test 9-3) is in satisfactory condition after 52 days at 550°C.

Two new tests were started at 300°C using Bar 9, previously shown to cause gas swells at 550°C, and these specimens show no evidence of gas swells after 7 days on test at the lower temperature even though no outgassing treatment was applied.

Test 5 at 200°C had an obviously poor weld and failed by oxidation in 14 days. Other tests at 200°C were also satisfactory except for closure defects.

These tests indicate no gas swells at temperatures of 300°C and under, and that suitable outgassing treatments can be applied to prevent this type of failure at higher temperatures.

Machinability

Some unexpected variations were found in the machinability of large extruded bars of Class 1 material. There were very distinct but comparatively unimportant differences in the amount of sparks and tendency of the chips to burn. In addition, there were variations of over 4 to 1 in tool life, even on bars of the same heat treatment.

Studies of the analyses, microstructure and heat treating of the materials have explained part of the differences, but as yet the factors which so greatly affect tool wear are not known. Work in progress will, it is hoped, enable the subject to be completely understood.

The differences in material were first experienced during a study of the proper machining schedule for turning 6" x 1.28" cylinders. From the first two bars received (A-1088 and A-981, Table 25), 18 pieces were produced with a single tool in 5 minutes apiece. At the end of the

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TABLE 25. MACHINABILITY DATA

Part No.	Heat Treatment as Machined	No. of Pieces Mach.	No. of Tools Used	Pieces per Grind	Speed r.p.m.	Remarks on Machining	Rockwell 'B' Hardness		Composition							
							As Machined	Furnace Cooled from 1700°F.	C-%	B ppm	Fe ppm	N ppm	Ca ppm	Mg ppm	Mn ppm	Li ppm
1088	Quench after extrusion	8)	1- still good	+18	293	Only a little sparking, tool wear small	98	95	.09	.26	44	18	-50	-10	-5	
881	Quench after extrusion	10)					96	86		.17	40	44	-50	-10	25	
967	Quench after extrusion	11					93	86	.19	30	35	25			20	20
1065	Quench after extrusion	10)	6	5-6	229	Considerable sparking and tendency for fire. Excessively high tool wear	91	94	.06	.26	29	46	-50	-10	-5	
1074	Quench after extrusion	12)					95	86		.23	43	45	-50	-10	-5	
1057	Quench after extrusion	11)					96	84		.21	64	21	-50	-10	-5	
1089	Furnace cooled from 1700°F.	8	2	4		Excessive spark- ing and tool wear	85	85	.05	.26		.50	-50	10	-5	
1025	Furnace cooled from 1700°F.	10	1- Still Good	+10		More sparking than 1089, little tool wear	95	95	.07	.17	33		-50	-10	15	
1023	Air cooled from 1700 to 1500° F. then water quenched	9	1- Still	+8 good		Little sparking, little tool wear	98	91		.14	100		-50	-10	3	
993	Air cooled from 1700 to 1500°F, then water quenched, heated 1 hr at 1110°F.	10	1- Still	+10 good		Little sparking, little tool wear	97	87		.15	43	67	-50	-10	5	
954	Air cooled from 1700 to 1500°F, the water quenched	9	1- Still	+9 good		Excessive sparking, little tool wear	98	92		.20			-50	-10	27	
1097	Air cooled from 1700 to 1000°F, the water quenched	8	1- Still	+8 good		Excessive sparking, little tool wear	98	91		.27	141	38	-50	-10	-5	

series, the tool was still in excellent condition. A water-base coolant was used, and little difficulty was experienced with burning of the chips.

Subsequently, 10 more bars were received and machining started. It was immediately apparent that the machining characteristics of this lot was different for there was so much fire that it was necessary to reduce the turning speed, and the tools lasted for only 5-6 pieces.

It was thought that the differences in machinability might be a result of quenching from different temperatures after extrusion. To determine whether this was the cause, the microstructure of the extruded bars was examined and compared with that of variously heat treated samples. It was found that the first lot of readily machinable bars contained randomly distributed inclusions, that the second lot of difficultly machinable bars contained inclusions grouped along grain boundaries. Heat treating studies showed that when samples were air cooled from 1700°F to various temperatures and then quenched, the inclusions were randomly distributed if the quenching temperature was over that of the upper critical (1422°F, 772°C) and along grain boundaries at lower temperatures.

These results indicated that the structure of readily machinable bars would be obtained if the quenching temperature was about 1500°F (820°C). To find whether the machinability tests would reflect the changes of structure, the remaining six bars were given four different heat treatments and tested for machinability (see Table 25) when it was found that heat treatment was not the only important factor in controlling machinability. For, although the two bars quenched from 1500°F (820°C) machined easily as would have been predicted from their structure, even though one was subsequently drawn at 1110°F (600°C), so did those which were expected to machine poorly-- the bars quenched from 1000°F (540°C).

Only one bar of this latter six machined poorly, this was one

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furnace cooled from 1700°F (925°C). The companion bar with the same heat treatment machined easily.

These results showed that heat treatment alone did not determine whether a bar would machine easily or difficultly. Neither did the hardness as machined, as Table 25 shows. It was evident that there was some other factor inherent in the material which exerted an exceedingly important effect. Work has been started that is intended to isolate this factor, to find its source, its effects and possibly a cure. Though the work is incomplete, some of the results are sufficiently conclusive to be reported.

A method has been found which is at least partly successful in separating inherently easily machinable bars from difficultly machinable ones. This involves furnace cooling of a sample from 1700°F to a comparatively low temperature and measuring hardness when it appears that difficultly machinable bars will be considerably softer than others.

Applying this test to the twelve bars which had been tested for machinability, it was found that they were separated into two distinct groups of six bars each. The hardness of one group ranged from 84 to 87 Rockwell "B" (159 to 170 Brinell by conversion), of the other group, from 91 to 95 Rockwell "B" (187 to 207 Brinell by conversion). Of the six softer bars, four were reported to be poorly machinable. Three of these four were tested as quenched after extrusion and it was evident from their microstructure that the quenching took place at a temperature below the upper critical of 1422°F (772°C). The fourth had been furnace cooled. The other two bars which were soft as furnace cooled had machined well. One of these was machined as quenched after extrusion, and, from its microstructure, it seemed evident that it had been quenched above the upper critical temperature. The other was known to have been quenched from such a temperature, 1500°F, and later reheated at 1110°F to simulate a degassing

treatment. This treatment did not appear to affect machinability.

Five of the six bars which fell in the hard group after furnace cooling machined readily. One of these five was quenched after extrusion, another furnace cooled from 1700°F, one quenched at 1500°F and the other two quenched at 1000°F. The sixth was one tested in a lot of four poorly machinable bars at a time when differences in machinability were not expected. Only incomplete records from that time are available, and the possibility exists that this one bar may have machined satisfactorily.

To recapitulate, twelve bars which had been tested for machinability were furnace cooled from 1700°F (925°C) and checked for hardness, when they were found to fall into two different hardness groups. Four of the six in the softer group were difficult to machine, five of the six in the harder group were easy to machine. Thus, in nine out of twelve cases, the machinability of the bars could have been predicted from their hardness after furnace cooling. Two of the bars which, on the basis above, would have been expected to be difficult to machine actually machined readily. Since both of these were quenched from above the upper critical temperature, one is led to the belief that such a high temperature quench may improve the machinability of bars which otherwise would machine with difficulty.

The following tentative conclusions may be drawn regarding the effects of material and heat treatment on machinability. These are subject to revision as additional data are obtained.

- 1) Certain samples will machine with relative ease regardless of their heat treatment.
- 2) Other samples will machine with ease only if they are quenched before they have air cooled below the upper transformation temperature. Subsequent heating at temperatures below the

lower transformation does not seem to affect machinability.

- 3) If these latter samples are air or furnace cooled to too low a temperature before quenching, they will be extremely difficult to machine.
- 4) The first type of material can be distinguished from the second by furnace cooling from 1700°F (925°C). After this treatment, readily machinable material will be harder than difficultly machinable material.

Investigation of the differences, and the cause for the differences, between the two materials are in progress. It has been found that bad material has a lamellar structure after furnace cooling, whereas good material does not. An earlier report (Battelle, April 10, 1943) indicates that this lamellar structure forms during the transformation from beta to alpha, so it would seem that this transformation may be retarded or entirely absent in one kind of material-- the readily machinable. Work now in progress shows that there is a difference of the type expected between the thermal arrests on heating or cooling in the two types of material. This work is to be amplified and substantiated by dilatometric studies, metallographic study of variously heat treated specimens and X-ray diffraction.

The cause of the difference in bars is not yet known. Analyses for B, Fe, N, Ca, Mg, Mn and Si offer no clues, as Table 25 shows. Four carbon analyses are reported in that table and it is seen that the one sample which was soft after furnace cooling was lowest in carbon by a small amount. Additional analyses are being obtained and may or may not substantiate this clue.

So far, only the important differences in tool life which were obtained with different samples have been discussed. Samples are also

different in another respect, though a comparatively unimportant one. This is their tendency to spark during machining. Metallographic examination has shown that the samples which spark most are those with the largest number of inclusions visible on an as polished specimen. However, the number of inclusions and accordingly the amount of sparking are not related to either the carbon content or the tool life.

Boron Cast Iron

An investigation of the properties of cast iron containing from 0.5 to 1.0% B was requested by Dr. Greninger.

One preliminary heat was made in the induction furnace and two in the gas-air furnace and cast in 2" x 4" x 6" blocks as shown in Table 26 under heat numbers 9611, 9612, and 9613. These heats showed as cast BHN of 444, 338 and 450, respectively, which indicated lack of machinability although Heat 9612 at 338 BHN could be drilled. The subject was discussed with Mr. N. F. Tisdale of the Molybdenum Corporation of America who stated that boron cast irons could be malleablized. A composition was recommended with 2 to 2.5 Si and 2.80 to 3.00 C with Mn below 0.50 because Mn forms a stable boride. No definite malleablizing temperatures were stated but these temperatures were above 1625°F and the annealing time was not unduly long. The iron must be cooled slowly from the malleablizing temperature to avoid hardening by air cooling.

Mr. Tisdale also stated that a high carbon ferro-boron is now being made containing 20-24% B, which is readily soluble in iron and can be added to the spout at the cupola. This grade of ferro-boron could be produced at the rate of 200 tons per month if necessary.

With this information in mind, six additional heats were made in

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TABLE 26. INTENDED AND ACTUAL COMPOSITIONS OF BORON CAST IRONS

Heat No.		Furnace	C	Si	Mn	P	S	B
9611	Intended	Induction	2.00	2.20	0.70/0.90	0.11	0.03	0.50
9612	Intended	Gas - Air	3.30	"	" "	"	"	0.50
	Actual							0.46
9613	Intended	" "	3.30	"	" "	"	"	1.00
	Actual							0.77
9632	Intended	" "	3.00	2.25	0.35	0.10	0.10	1.00
	Actual		2.91	2.00	0.42			.51
9633	Intended	" "	3.00	2.25	0.80/0.90	0.10	0.10	1.00
	Actual		2.95	2.28	0.72			.68
9634	Intended	" "	3.00	2.25	0.35	0.10	0.10	0.50
	Actual		2.98	2.12	0.44			.41
9635	Intended	" "	3.00	1.75	0.35	0.10	0.10	1.00
	Actual		2.94	1.86	0.44			.62
9636	Intended	" "	3.00	2.25	0.35	0.10	0.10	1.00
	Actual		2.98	2.22	0.44			.59
9637	Intended	" "	3.00	2.25	0.35	0.10	0.10	1.00
	Actual		3.24	2.21	0.44			.72

the gas-air furnace. The additions were made at a temperature of 2600-2650°F and the heats were poured at 2450-2500°F three minutes after the additions were made.

Tests were made on the dilatometer at 1600 and 1700°F to follow malleablizing but this method was dropped because of the time required to obtain malleabilization. Small sections were heated at 1700°F for 24 hours and 1800°F for 12 hours and hardness determined before and after, as shown in Table 27.

These treatments show some decrease in hardness at 1700°F and still more at the 1800°F treatment and further tests were made at the higher temperature. Table 27 also shows the change in hardness obtained with these additional treatments. For these tests, the specimens were packed in sand and allowed to cool in the sand in the furnace, except for specimens of three heats, as shown in the table, which were air cooled.

For the compositions tested, the carbon-silicon ratio is the same except for Heat 9635 which was made at somewhat lower Si content. Heat 9636 with 2.22 Si shows minimum hardness after 5 hours anneal while the low Si Heat 9635 required 16 hours to attain the same hardness. Heat 9637, while intended to be of the same composition as Heat 9636, was actually higher in carbon and was so brittle that hardness could not be determined by Brinell but showed somewhat different annealing characteristics. The high Mn Heat 9633 required longer annealing time than a similar low Mn heat but was not greatly different than the low Si heat. One hour anneal on the 0.5% B yielded a lower hardness than was obtained on any of the 1% B heats.

All specimens could be machined on a shaper and could be drilled with a 1" drill after the 1800° treatments. Tool wear might be high because of the presence of carbides which were present in all annealed

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TABLE 27. HARDNESS OF BORON CAST IRONS WITH MALLEABLIZING ANNEALS

Heat No.	Method of Cooling	Brinell Hardness - Held at 1800°F. as shown ⁽¹⁾						Brinell Hardness ⁽²⁾	
		As Cast	1	3	5	8	16	21 hrs.at 1700°F.	12 hrs.at 1800°F.
9632	Furnace	406	352	343	341	335	290*	356	388
	Air	406	-	-	383	383	-	-	311(3)
9633	Furnace	444	383	388	372*	362	357	401	-
	Air	444	-	-	393	377*	-	-	-
9634	Furnace	328	269	277	269	269	270*	297	280
	Air	328	-	-	311	307	-	-	288(3)
9635	Furnace	468	406	395	388	380	352*	415	-
9636	"	412	354	356	345	345	352*	363	-
9637	"	417	352*	327*	352*	352*	297*	341*	-

(1) Specimens were furnace cooled in sand except as indicated.

(2) Small specimens furnace cooled without sand.

(3) These specimens were prequenched from 1700° F.

* Conversion from Rockwell C because of porosity and in the case of Heat 9637 brittleness.

structures.

The irons showed flakes of primary graphite with some annealed samples showing separation of nodular graphite usually adjacent to the flakes. The presence of a considerable amount of fairly stable carbide does not indicate good machining properties.

The casting properties indicate somewhat more shrinkage than gray iron but probably not so much as regular malleable iron. Production of good castings should not be a serious problem.

These tests indicate that, to produce a machinable boron cast iron, annealing of castings would be necessary. An iron with 0.5% B appears preferable in obtaining the most machinable metal and rapid annealing. Finishes obtained on machining were not comparable with those obtained on gray iron. No difficulty should be encountered in making sound machinable castings.

Flash Welding

Samples of 3/8" extruded rod were flash welded on a Taylor-Winfield Model B530KVA machine under the conditions shown in Table 28. The welding conditions were selected to give a preliminary survey of the possibilities of this type of welding. The welds were sectioned and examined macroscopically, Figure 75 showing the condition of the weld in Specimen #1. This shows complete fusion across the 3/8" diameter. Specimens #7 and 8 showed similar characteristics while the remaining specimens were poorly welded, showing cavities and oxide inclusions in the weld zone.

Microscopic examination of the weld showed some probable oxide inclusion in parts of the weld of Specimen #1 along the line visible in

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TABLE 28. FLASH WELDING DATA

Specimen	Transformer Tap	Speed Sec.	Burn Off	Upset	Macro Examination
1	5	0.9	0.2"	0.13"	Complete Weld
2	1	0.9	0.2	0.13	---
3	1	1.5	0.2	0.13	Poor Weld
4	2	1.5	0.2	0.13	Poor Weld
5	7	0.8	0.2	0.15	Complete Weld
6	8	0.8	0.2	0.16	Complete Weld

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Figure 75.

Macrograph of Weld,
Specimen #1, Showing
Complete Fusion.

21580

Figure 76.

Microstructure of
Weld, Section at Left
in Figure 75, Showing
Inclusions. Etched.

500X

21581

Figure 77.

Microstructure of
Weld, Section at
Right in Figure 75.
Weld line is barely
visible and structure
of adjacent metal un-
affected. Etched.

500X

21582

604°C
BHN-19

654°C
BHN-25

702°C
BHN-44

753°C
BHN-16

772°C
BHN ---

Figure 78. Hot Hardness Specimens with Brinell Numbers and Temperature of Test. 2X

by low values for some of the elements shown. The presence of 21.7% Co was somewhat surprising since Co might be expected to form a low melting alloy similar to Ni which would promote early failure of the die. However, the Cr and W contents are probably high enough to account in a large measure for the success of this material.

Metallography

The metallographic work during the past month has been directed in lines to assist in determining the cause of differences found during machining, as discussed in a previous section. Considerable variation in the reaction of various billets to the etching reagent has been observed and, in some samples, difficulty was encountered in developing satisfactory structures.

Previous work has indicated that slow or furnace cooling produces the lamellar structure previously shown. In the present work, it has not as yet been possible to develop the lamellar structure in all furnace cooled samples. This would indicate that some factor is affecting the cooling rate required to produce this structure or that the constituent present which produces this structure is absent. No correlation between the structures and known compositions has been found, except in the case of carbon content with inclusions.

Carbon analyses have been made on sample of metal on which microstructures have been shown in previous reports as follows:

Pure metal	.02% C	Figure 70
B1 metal	.07	Figures 41 to 44
3/8" Extruded rod	.07	Figure 23
M310, L148	.08	Figures 61 to 69
MH scrap (bottom?)	.10	---
MH scrap	.37	Figures 39 & 40
Vacuum Fusion Button	1.77	Figures 37 & 38

These carbon contents appear to correspond to the amount of carbide inclusions found in these micrographs.

Further metallographic work is in progress in attempting to clear the discrepancies found on different lots of metal and in particular relation to machinability.

FUTURE WORK

The hydrogen content of metal treated to give the minimum amount of hydrogen will be determined using the analytical procedure previously described. Work on the development of a commercial outgassing procedure is in progress and is being similarly followed.

Samples are being obtained from an as cast billet, extruded bar, and extrusion butt in order to follow the hydrogen content through the extrusion process.

The causes of variations in machinability are being investigated further by means of microscopic examinations and studies of the thermal arrests on heating and cooling of good and bad material.

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this region as shown in Figure 76. A micrograph of the weld zone where no visible line was found in the macrograph is shown in Figure 77. Only an irregular line of the weld is visible with no apparent effect on the adjacent metal.

It will be noted that the welding conditions favorable to good welds are high current values and high speeds. Some further study of the welding technique should permit obtaining satisfactory welds on commercially available equipment.

Hot Hardness Tests

In view of the results obtained on the drop hammer tests reported last month, the determination of the hardness at elevated temperatures, particularly in the beta region, seemed desirable.

A mutual indentation method for hot hardness determinations of steel is described by O. E. Harder and H. A. Grove, Trans. AIME, vol. 105, 1933, p. 88. In making the tests, two cylinders of the metal to be tested, 1 cm long x 1 cm diameter, are placed in contact in parallel position in suitable compression blocks between the ram and anvil of a standard Brinell hardness testing machine. Heating is accomplished by an electrically heated tubular furnace surrounding the anvils. After the specimens have reached the desired temperature, this temperature is maintained for twenty minutes before applying the load which, in the case of these tests, was 500 kg. for 30 seconds. An argon atmosphere was maintained in the furnace and the specimens quenched in water after indentation.

The hardness number is then calculated by first obtaining the average area of the indented surface. The Brinell hardness number is then equal to $1.52 \times$ the load in kg. divided by the area of indentation in sq. mm.

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The results of these tests are shown in Figure 78. These tests show an increase in hardness as the temperature increased from 604°C to 702°C. The hardness then decreased with increase in temperature until, at 772°C, the specimens actually flowed together into one piece. The tests agree in general with the drop hammer tests. The hot hardness at 753°C appears to be of the same order as at 604°C thus confirming the forgability in the temperature range below the upper transition.

Extrusion Die Materials

Three additional billets have been extruded on the "Tantung" hard faced experimental die making a total of 7 extrusions on this die. The die is still in excellent condition, showing no evidence of use.

Some improvements in hard facing practice with "Stoodite #63" have been obtained although direct comparisons cannot be made since present production is on the smaller rod. This should result in more severe conditions for the die because of greater reduction from the billet size, and the production of longer rods should result in higher die temperatures and possibly somewhat higher pressures. However, with the improvements in hard facing technique, the smaller dies have been showing good service, up to 180 extrusions having been obtained.

An analysis of "Stoodite #63" shows the following:

C	2.48
Cr	10.1
Ni	0.12
Co	21.7
Mn	0.30
B	<.0005
Fe	40.0
W	21.0

This leaves some 4-5% unaccounted for which may be partly caused

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