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PROGRESS REPORT

on

METALLURGY OF TUBALLOY

to

UNIVERSITY OF CHICAGO

from

BATTELLE MEMORIAL INSTITUTE

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SUMMARY

Commercial extrusion is proceeding satisfactorily using dies hard faced with "Stoodite 63". Three dies have been used for extrusion of 248 billets for 1.28" rod.

Drop hammer tests show marked ductility at 1300° F (705°C) and above, but very little deformation occurred at 1220 to 1250° F (660 to 677°C). As the temperature was lowered from this range, deformation increased.

Thermal expansion measurements of alloys in the range of composition of the zinc-aluminum hot dip coating did not provide an explanation for failures of this coating. Micro-examination of the coating revealed circumferential cracks in a layer adjacent to the base metal which appear to be a potential weakness of the coating.

Tests on aluminum jacketed metal are in progress. One test at 550°C gave a gas swell with no oxidation following which tests were started on metal which was out-gassed before final welding. Two such units have been on test for 8 days with no signs of failure. Tests at 300°C and under have not shown evidence of gas swells to date.

Following the gas swell on tests of aluminum jacketed metal, substantial quantities of hydrogen were extracted from the metal which seem to explain the swelling of the jacket at 550°C. These extraction tests show an increase in hydrogen content when heating in natural gas and a decrease when heating in argon at 1000°C. Quenching in water after air cooling to 600°C does not change hydrogen content appreciably.

Additional photomicrographs are given following changes of the microstructure of Billet M310, L128 hot rolled at 920°C and quenched when reheated to 900, 700, and 600°C followed by various cooling rates. The lamellar structure seems to be characteristic of furnace or slow cooled metal, while quenching above the upper transition gives a homogeneous structure except for undissolved carbide.

EXPERIMENTAL DATAHot ExtrusionTests on Extrusion Die Materials

Tests in the experimental extrusion press were made on a number of additional die materials as shown in Table 19. Low carbon ferro-chrome both with and without nitrogen, "Tantung G", and self-hardening "Stoodite" were used as hard facing materials covering the leading edge and bearing of the die. These materials all appeared to be very satisfactory on the experimental press. Further tests were made with the 25 Cr, 7 Mo, 1 Si, 1 C steel and this still appeared promising but there was some evidence of alloying on the leading edge of the die. Tests were also made on dies made from a high alumina spark plug type refractory which appeared to withstand the extrusion satisfactorily but was somewhat susceptible to cracking from thermal shock.

Following up the leads obtained in the laboratory, dies were made for the commercial extrusion press. These production tryouts are described below.

On March 13, extrusions were made on a chromium plated hot die steel and the 25 Cr, 7 Mo, 1 Si, 2 C cast steel. For the former, approximately .005" of chromium was plated on a die of heat treated .30-.40 C, 3-4 Cr, 9-10 W, 0.25 V hot die steel. This die was used for four extrusions and, while the leading edge was worn or alloyed, about one half of the 1/8" bearing was still coated with chromium.

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TABLE 19. TESTS OF DIE MATERIALS ON LABORATORY PRESS

Die Material	Die Design		Initial Die Surface	Die Condition After Extrusion	Rod Surface
	Bearings	Radius			
Cr25,Mo7, Sil, Cl	1/8	1/8	Repolish	Slight alloying	Some cross checking
" " " "	1/8	45° bevel	"	" "	Scored
" " " "	1/4	1/32	"	" "	" " "
" " " "	1/4	0	"	" "	Light scoring
" " " "	1/4	1/32	"	" "	Scored
Spark plug ceramic	1/4	Counter bore			
		1/32	Heated 2 hours at 1500°F.	OK on air cool	Rippled
" " "	"	"	Heated 2 hours at 1500° F.	OK cracked on quenching	Rippled
Low C Ferro-Chrome Hard Facing	"	"	Polished	No change	Cross checks
As above	"	"	"	Good	Rippled
As above plus N ₂	"	"	"	"	Good
Same	"	"	Repolished	"	Good
Tantung G - Hard facing	"	"	Polished	"	"
" " "	"	"	Repolished	"	Rippled
Stoodite Hard facing	"	"	Polished	"	Rippled
" " "	"	"	Repolished	"	Good

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Rods with a smooth surface were obtained. The second die was the chill cast alloy (composition as that on the preceding page) showing 50 Rockwell C hardness. After extruding two rods, this die appeared to be in perfect condition.

A number of dies of the latter material as well as the same composition, except with 1% C, were prepared for the larger scale production extrusion of March 19th. Three dies were used in extruding 14 billets (two were special billets prepared at Chicago with copper plates on the leading end). The first die used was of the chill cast alloy with 2 C which had given good results on the previous run. The design for this die, however, was modified from the 1/8" bearing with 1/32" relief at the back to use a 3/8" bearing with a 7° taper at the back. This die failed miserably on the first extrusion but was used for two billets. The die and rods were both badly scored. The second die was the 1 C chill cast alloy but the design was that used previously, 1/8" bearing with 1/32" relief at back, and was used for 7 billets. Some scoring occurred on the leading edge on extrusion of the first billet. This scoring progressed further with each billet run, and the bearing of the die was nearly worn away after the 7th billet. The third die used was the same type as that used on the first two billets. The two copper faced billets were extruded first and then three regular billets run. This die showed some failure at the leading edge but about 2/3 of the 3/8" bearing was retained. Examination of these three dies later showed many cracks radiating from the die opening. There seemed to be some improvement in die life when using the copper faced billets. It was also considered that this

lot of billets was of poor quality which may have affected die behavior. The cracking of the dies seemed a serious defect even though wear on the bearing could be reduced. It also appeared that the 3/8" bearing was unnecessarily long and offered no advantage over the other design.

Arrangements were made for extrusion of 6 billets on March 31st. For this run, a series of dies with hard facing materials were prepared. These included facing of the bearings, shear edge and back from the shear edge about 1" with a thickness of about 1/32" of self hardening "Stoodite", "Tantung", low carbon ferro-chrome and high carbon ferro-chrome. A "Stoodite" hard faced die was used for the first four extrusions with excellent results. There was only slight evidence of failure of the leading edge and the die material seemed much superior to any of the others tried. The die was changed after the fourth extrusion in order to test a low carbon ferro-chrome faced die. This die failed on the first extrusion reversing the results obtained on the experimental press.

A large production run of rod for the 1.28" size was made on April 3. For this run a die was prepared by B & T by enlarging an old aluminum extrusion die and hard facing with "Stoodite #63". To date, three dies hard faced with "Stoodite" have been used. The first was used for 7 billets on March 31st and is still in good condition. Die #2 was "Stoodite" hard faced over hot die steel which was not heat treated and ran 87 billets on April 3rd and 102 on April 7th when it failed by chipping out of the hard facing. Die #3 was similar to Die #2 and showed the same type of failure after 52 extrusions.

On this later date, 4 MIT billets were extruded in the

smaller size rod using a "Tantung" hard faced die. This die showed no evidence of failure and seemed to be the best of the die materials tried experimentally.

Analysis of the "Stoodite 63" is in progress and, to date, it has been found to contain less than .0005% B. The balance of the analysis will be reported later. On the basis of the production run and from a cost standpoint, the "Stoodite" material seems satisfactory. Die sizes have been selected to allow $1/16$ " of material on a side to remove by machining. This seems a rather large allowance but, with the straightness so far obtained on bars, this allowance appears necessary. A contraction allowance of .035 to .040" on the diameter is allowed on bars 1 to $1\frac{1}{2}$ " diameter. Thus, the die diameter to produce bars for 1.1" finished size is 1.1 plus .125 plus .035, or 1.260".

Commercial Extrusion

A total of 28 billets was extruded on March 13th, 19th, 31st, and April 7th, making $1-5/32$ " rod which has been used to a large extent for coating experiments. These runs show that it is possible to obtain rod with a metal loss through oxidation in the neighborhood of 1%. The data on these runs are summarized in Table 20. These test runs used a raw natural gas atmosphere throughout and, beginning with Billet No. 7, were run in the production furnace.

This furnace was constructed under supervision of the Battelle ceramic division and consists of brick construction with supporting iron frame. Figure 55 shows the design used. This consists

TABLE 20. DATA ON COMMERCIAL EXTRUSIONS

Billet Mark	Billet No.	Billett	Butt	Rod	Per Cent Yield	Per Cent Loss Wt.	Die Used		Size of Rod			Condition of Billet	Macro-Etched Bar Ends		Condition of Rod Surface	Condition of Die
							Material	Size	Butt End	Front Middle	End		Butt	Leading		
None	1	80*	15	65	81.3	-	Cr. plated	1.187					-	-	Slight scoring	Leading edge failing
"	2	71.7*	12.5	59.2	82.6	-	" "	"					-	-	" "	" "
"	3	80.4*	11	69.4	86.3	-	" "	"					-	-	" "	" "
"	4	65.5*	12	53.5	81.7	-	" "	"					-	-	" "	1/2 bearing still good
"	5	111.9*	26.5	85.4	76.3	-	(Chill	"					-	-	" "	No change
"	6	101*	20	81	80.2	-	(Cast alloy	"					-	-	" "	" "
"	7	93	10.75	81.25	87.5	1.0	" "	1.20			1.185	Surface folds - Top piped	Large Pipe**	Slight porosity	Badly Scored	Scored
"	8	91.5	16.1	75	82	0.5	" "	"			1.165	Top cropped - Bottom piped	"	"	Oval and scored	Bearing completely removed
"	9(1)	92.5	15.3	76.2	82.3	1.0	" "	"			1.188	Cropped - Bottom piped	Small "	"	Fair Surface - Longitudinal cracks	Leading edge failing
"	10	76	11.6	63.75	83.8	0.8	" "	"			1.196	Top cropped - piped	Large "	Sound	Fair Surface	Failure progressing
"	11	82	13.25	68.75	83.5	0	" "	"			1.186	Hot top - small pipe	"	"	" "	" "
"	12(1)	66.75	12.25	54.2	81.2	0.5	" "	"			1.184	Surface folds - Bottom cropped	Medium "	Slight porosity	Longitudinal Cracks	" "
M870	13	93.5	13.5	79.2	84.7	0.9	" "	"			1.193	Top cropped - large pipe	Large "	Sound	Fair Surface	" "
M899	14	90.5	13.1	77.2	85	0.3	" "	"			1.196	Cropped - Bottom wide pipe	"	Small pipe	" "	" "
None	15	97.5	13	83.9	85.2	0.6	" "	"			1.180	Top cropped - deep pipe	Piped	Medium pipe	Slight Scoring	Bearing scored
M704-RI	16	88	14.25	73.5	83.6	0.2	" "	1.187			1.150	Top cropped - Bottom drossy	Spongy, Piped	Spongy	Good	Good
M701-RI	17	87.25	14.25	70.2	80.5	3.2	" "	"			1.147	" " - piped	Piped	Small pipe	"	Fair
M702-RI	18(1)	85	16.1	71.5	84	?	" "	"			1.146	" " - "	Medium pipe	Medium Pipe	"	Some failure on leading edge
M712-RI	19(1)	75	8.25	66	88	1.0	Stoodite	1.221	1.175	1.165	1.159	" " - piped - Bottom drossy	Porous	Very small pipes	Smooth	Good
M708-RI	20	77	9.0	67.5	87.7	0.7	Hard face	"			1.170	Top cropped - Bottom as cast	Large pipe	Small pipe	"	"
M713-RI	21	84.9	10	74.25	87.6	0.8	"	"	1.174	1.164	1.161	" " - Piped - Bottom as cast	"	Sound	"	"
M716-RI	22	83.5	-	-	-	-	"	"	-	-	-	" " No pipe Bottom as cast	-	-	-	-
M723-RI	23(1)	82	10.5	72	87.8	0	"	"	1.172	1.162	1.155	" " Piped	Medium pipe	Sound	"	Slight failure on leading edge
M720-RI	24	93	13	68.5	73.7	12.4?	L.C.Ferro Chrome, Hard Face	1.215	1.215	1.185	1.174	" " - No pipe	Sound	Sound	Scored	Scored badly

* Total of butt and rod weights.

(1) Extruded top forward - others bottom of billet forward.

** Pipe at butt end is usually an extrusion pipe but may be both - at the leading end pipe indicates unsoundness of that end of the billet.

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TABLE 20. (CONTINUED)

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Billet Mark	Billet No.	Billet	Butt	Rod	Per Cent	Per Cent	Die Used		Size of Rod			Condition of Billet	Macro-Etched Bar Ends		Condition of Rod Surface	Condition of Die
					Yield	Loss Wt.	Material	Size	Butt End	Middle	Front End		Butt	Leading		
M727	25	74		68.5	92.6		Tantung G. Hard face	1.198	1.167			Top cropped - Bottom drossy	-	-	Smooth	Unchanged
M725	26	82.5		76.5	92.7		"	"	1.170	"	"	"	-	-	"	"
M729-RI	27	90		84.5	93.8		"	"	1.162	"	"	"	-	-	"	"
M731-RI	28	83		77.25	93.2		"	"	1.168	"	"	"	-	-	"	"

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FIG. 55-BILLET HEATING FURNACE.

21110

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essentially of two parallel muffles 10-3/4" wide x 9 1/2" high x 6" long, capable of holding 15 billets each. These muffles are constructed of a Carbofrax hearth with firebrick sides and top. The muffles are surrounded with suitable combustion spaces for six No. 3 Premix gas burners, fired alternately from each side of the furnace. The bottom of the muffles is on a slope sufficient to permit the billets to roll from the charging end to the discharge end.

The furnace is equipped for automatic temperature control through thermocouples in each muffle which operate a motor actuated valve at the Premix blower. In operation, it is necessary to control from the hotter muffle to avoid over heating. The natural gas atmosphere is introduced at the top near the charging end of each muffle and solenoid valves are provided to allow greater gas flow when the muffle doors at either end are opened.

In regular production, there has been some tendency to have cold billets. This appears partly caused by the extrusion press operation being somewhat faster than the heating rate of the furnace but, to a larger extent, by the necessity for heating billets which are too long to roll through the muffle and which must be pushed through, causing long periods when the doors are open. On the whole, furnace operation has been fairly satisfactory when the furnace is fully loaded. Partial loading, such as using only one muffle, causes an unbalanced heat load which makes temperature control more difficult.

During each run, various die materials were used, as indicated in the table and discussed previously. The data on die sizes and rod sizes show a contraction of .035 to .040" and provide necessary information in establishing proper die sizes.

It will be noted that the butt end of the rods is about .015" larger in diameter than the leading end. This is believed caused by upsetting of the soft metal in pushing the rod along the angle iron. Provision of rollers instead of angle to receive the rod from the die should be of assistance in preventing this effect, should it become desirable.

The condition of the billets and macro-etched bar ends shows some correlation. Billets which appeared drossy lead to sponginess and porosity in the bar ends. Billet #19 is an example of this. This billet also shows that pipe in the leading end of the billet leads to pipe in the leading end of the bar while Billet #21, while piped at the back end, gave a sound bar at the leading end. Extrusion of billets piped at the top lead to more unsoundness in the leading end of the rod when the top of the billet is the leading end. Billet #13, which had a large pipe at the top but was extruded bottom first, gave a sound bar at the leading end while the pipe appeared in the butt end. Billet #24 appeared sound and produced rod sound at both ends but with a somewhat larger butt, thus reducing the tendency for an extrusion pipe. Billets #9 and 12, which show piping at the bottom, both gave longitudinal cracks in the bars in spite of extruding the top end first. This cracking appears to be connected with the condition of the billet as quenching tests made on an uncracked bar with a longitudinal V-groove did not show cracking at any temperature in the range where the bar was quenched.

Figure 56 shows the nature of the porosity of Bar #19, which is believed caused by sponginess of the billet. It will be noted that

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21061

Leading
End

Butt End of Bar Showing Decrease in
Porosity During Cropping

Extrusion Butt

Figure 56. Macro-Etched Bar Ends and Butt from Billet #19 Showing Porosity
Caused by Poor Billet

the porosity persists for about 12" forward of the butt. The leading end at the extreme left appears quite sound in the photograph, but actually had some very small pipes. Figure 57 shows the macro-structure of Bar #20 which is representative of average conditions in these bars. A fairly large pipe is evident near the butt end but this section is much better than Bar #19 at the same location. The forward end appears fairly sound but some small openings may be discerned.

blocks
Two types of dummy have been used: hot die steel hard faced with "Stoodite" and cast Cr-Mo steel as tried for dies. Seven blocks were used for 248 billets, only two of which were hard faced. All of these were in bad condition, being dented and caved and not suitable for further use. At present, the dummy blocks appear to be more of a problem than dies.

Drop Hammer Tests on Deformation at Elevated Temperatures

It has been stated that there appeared to be some ductility in the temperature range between 662°C (1220°F) and 772°C (1420°F). A series of drop hammer tests were made to determine the amount of deformation obtained under a fixed load of 225 ft. lbs. Fixed anvils were used on both ends of the specimen which was 3/4" diameter x 1-7/8" long. This caused some cooling of the metal in contact with the anvils which produced interesting effects on the manner of deformation. The specimens were heated at first in a lead bath but this showed some wetting at 662°C (1220°F) and the specimen heated to 760°C (1400°F) could not be used on account of alloying. A new series was started using a natural gas atmosphere. In all cases, a furnace temperature

19

21060

-123-

19

Figure 57. Macro-Etched Bar Ends and Butt from Billet #20 Showing Small Extrusion pipe in Butt End of Bar

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was selected 25°F above the temperature desired and as shown in Table 21. The time interval from removal of the specimen from the furnace until the impact of the hammer was 11 to 13 seconds.

It will be noted that a marked decrease in ductility occurs as the temperature is increased from 540°C (1000°F) to 677°C (1250°F). Furthermore, all the deformation at 660°C (1220°F) and 677°C (1250°F) took place at the ends where the metal was cooled by the anvil. Increasing the temperature to 705°C (1300°F) gave a sharp increase in the amount of deformation but the temperature of the metal was sufficiently low that the surface air cooled into the brittle region causing the surface to have a fish scale appearance, as shown in Figure 58. The cooling effect of the anvils was still apparent but, at this temperature, it caused lack of deformation. Above this temperature, there was little change in the amount of deformation although the effect of the end cooling was less noticeable.

These data confirm the ductility of the metal in the beta region and indicate that the brittle range is apparently a very small one closely associated with the transition at 662°C. It would also appear that extrusion should be possible at temperatures lower than 1450°F (788°C), as shown previously. However, it is believed that the difference in the rate of applying pressure may be the determining factor. Making hot impact extrusions should be possible.

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TABLE 21. EFFECT OF TEMPERATURE ON DEFORMATION ON DROP HAMMER

Temperature		Per Cent Decrease in Length (1-7/8" Initial)
°F	°C	
1000	540	7.43
		7.20
1200	650	5.06
1220	660	2.32
1230	664	3.85
1250	677	1.65
		1.46
1300	705	44.5
1325	718	49.8
1350	732	50.5
1400	760	46.5
1420	772	54.6
1450	788	54.4
1500	816	51.5

22

20651

Figure 58. Specimens Tested Under 225 ft. lb. Drop, Hammer at Temperatures of 1000, 1220, 1250, 1325, 1350, 1420, and 1500°F in Order Given

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Coating Methods

Zinc-Aluminum Hot Dip Coating

Failures obtained on tests of hot dipped coatings consisting of 85 zinc-15 aluminum alloy were believed possibly caused by differences in thermal expansion between the coating and the base metal. A series of 8 alloys containing 1, 3, and 5% base metal, both with and without 1% Mg added to the 85 Zn-15 Al base, were made and cast into 1" bars. Thermal expansion specimens $3/8$ " by $3\frac{1}{2}$ " long were machined from these bars and expansion measurements made up to 300°C with some data on contraction during cooling. No significant differences in the expansion characteristics were found.

The micro-structure of the coating was examined when applied in the prescribed procedure, as well as for longer immersion periods. Figure 59 shows the structure of the coating down to the brittle base metal layer. Particles of base metal (white) may be seen to have floated away from the interface while there seems to be a substantial concentration of these particles at the interface. Figure 60 shows the junction of the base metal with the hard base metal layer. It will be noted that this layer has many cracks which are circumferential to the base metal. With such cracks existing in the coating, it is difficult to believe that such a coating would adhere permanently and provide the necessary protection.

Jacketing with Aluminum

Samples of metal 1.1" diameter x 2" long have been jacketed with aluminum using aluminum tubing .031" wall x 1.187" OD. Shallow

21121
500X

Figure 59. Structure of 85 Zn-15 Al Coating Adjacent to Base Metal Layer (bottom) for 4 Minute Dip.

21122
500X

Figure 60. Structure of Hard Base Metal Layer Adjacent to Base Metal (at bottom) Showing Circumferential Cracks for 4 Minute Dip.

aluminum cups were made of the proper size to fit the aluminum tube after drawing through the die and one end of the tube closed by welding the cup in place. After testing the weld for leaks, the metal was placed in the tube and a second cup placed on the upper end of the metal. This assembly was then pressed through a die sized to reduce the wall thickness of the aluminum from .031" to .020" by pressure against the metal core. This operation forced the aluminum tightly against the core and held the upper cup in place against the end of the core and inside the aluminum tubing. The end of the tubing was then trimmed even with the edge of the cupped end and the enclosure completed by welding. Regular aluminum welding flux and an oxy-hydrogen torch was used for welding.

A number of these assemblies were placed on test at 200 and 550°C as shown in Table 22. The earlier tests at 550°C showed failures by oxidation of the metal core which are believed caused by leaks in the final weld. The gas swell obtained on Test #6 probably represents the first satisfactory final weld. Following this failure, five assemblies which were ready for final welding were heated at 600°C for 45 hours in argon as an out-gassing treatment. Two of these were welded and placed on test April 2nd and show no signs of failure to April 10th. This would appear to indicate that the out-gassing treatment was effective as the gas swell occurred in 16 hours. Test #15 at 300°C on an extruded rod has shown no indications of swelling after two days on test, showing that gas evolution at this temperature is not sufficient to cause distortion of the jacket. The tests at 200°C have not shown failures of any kind at this time.

These tests indicate that, for operation at temperatures near

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TABLE 22. TESTS ON ALUMINUM JACKETED METAL

Test No.	Pretreatment	Metal	Temperature • C.	Date Started	Time On Test	Type of Failure
1	None	Bar #9	550		46 hrs.	Oxide swell-leak in weld
3	"	"	"		23	" " " " "
4	"	"	"		23	" " " " "
6	"	"	"		16	Gas swell - no leak
11	45 hours at 600°C. in Argon	"	"	4-2-43		OK on 4-10-43 - 2 on test
12*	" " " " " "	"	"	4-6-43		" " " " " "
15	None	"	300	4-8-43		OK on 4-10-43
2	None	"	200	3-27-43		OK on 4-10-43
5	"	"	"	3-29-43		" " " " "
7	" - hole in weld	"	"	3-29-43		" " " " "
13*	45 hours at 600°C. in Argon	"	"	4-6-43		" " " " "
8	None	3/8" rod	200	3-30-43		OK on 4-10-43
14	Spun Ends-not welded	" "	"	3-25-43		" " " " "
9	None	" "	550	4-1-43		" " " " "

* Brazed with Aluminum Co's Flux #33 and rod X716.

550°C, some out-gassing treatment of the metal would be necessary while, for 300°C and under, it does not appear necessary.

Gas-Metal Studies

In view of the gas swell obtained on the aluminum jacketed metal, investigation of the metal for gas which would be evolved in this temperature range seemed desirable. Preliminary tests are reported here. Two specimens of the same metal, Bar #9, as extruded, and the same size as that used for jacketing, were heated in the vacuum fusion gas analysis apparatus and the gas was pumped out at various temperatures and showed the following:

<u>Temperature</u> °C	<u>Hydrogen Evolved</u>	
	<u>Specimen 1</u>	<u>Specimen 2</u>
200	0.45 cc in 45 min.	1.10 cc in 90 min.
310-380	7.30 cc in 100 min.	-----
550	8.50 cc in 68 min.	-----
750	10.95 cc in 61 min.	20.45 cc in 120 min.
820	-----	4.00 cc in 40 min.
Total	27.20 cc	25.55 cc

Analysis of the gas showed it to be hydrogen. It will be noted that out-gassing at 750°C on the second specimen showed that most of the gas was removed at this temperature. For further tests, it was concluded that pumping at 750°C for 2 to 3 hours would be used as a standard procedure. A specimen of another bar, which was not cracked as was Bar #9, was out-gassed 3 hours at 750°C and yielded 52.90 cc of hydrogen.

Several as cast billets, 1" x 4" long, used on the laboratory extrusion press were available, and a series of these was prepared representing the heating cycle of the large billets in natural gas, substitution of argon, and with cooling conditions simulating the treatment of the extruded bars, that is, air cool to about 600 to 650°C and quench as well as air cooling. Table 23 summarizes the data obtained from these specimens. These data are not corrected to standard conditions but are comparative. The total hydrogen content was obtained by plotting the total hydrogen evolved in solid extraction at the end of various times against the reciprocal of the square root of the time. Such a curve can be extrapolated to infinite time and a value for total gas evolvable at the extraction temperature obtained, as shown in the table.

Observations on gas extraction in a vacuum indicate the following:

1. At any constant temperature, the rate of gas extraction decreases.
2. After about one hour at constant temperature, the total hydrogen evolved is approximately proportional to the reciprocal of the square root of the time. At first, the rate of evolution is somewhat higher.
3. After extraction at any constant temperature for a period of time, the rate of evolution is always increased at a higher temperature.
4. The vacuum fusion total hydrogen contents are approximately half the extrapolated totals, indicating possibly greater solubility in molten than solid metal in the temperature ranges investigated.

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TABLE 23. HYDROGEN EXTRACTION DATA

Pretreatment of Specimen	Time at 750°C.	Evolved H ₂ , cc.	Total H ₂ Evolved	Extrapolated Total H ₂		Wt. %	Vacuum Fusion Total cc/550 gm.
				cc/550 gm.	Vol. H ₂ /Vol. metal		
As Cast	30 min.	7.40					
	60	5.10					
	90	3.90					
	120	2.90	19.30	34	1.1	.0005	---
Heated in Argon Air Cooled	30	5.75					
	60	2.40					
	90	1.75					
	120	1.30					
	150	1.15	12.35	17	0.55	.00025	8.3
Heat in Argon - Air Cooled to 600°C. Quench	30	6.25					
	60	1.60					
	90	1.30					
	120	1.10					
	150	1.00	11.25	17	0.55	.00025	9.1
Heated in Natural Gas Air Cooled to 600°C., Quenched	40	18.40					
	70	9.10					
	100	5.45					
	130	3.70					
	160	2.85					
	190	2.25	41.75	64	2.1	.00095	27.0
Sound bar, As Extruded	30	27.95					
	60	10.00					
	90	5.88					
	120	3.75					
	150	3.00					
	180	2.35	52.90	74	2.4	.0011	---

These tests indicate that heating in argon removes some hydrogen, heating in natural gas adds hydrogen, and that quenching in water from about 600°C does not affect the hydrogen content appreciably. The amount of hydrogen in as cast metal appears to be sufficient to produce gas swells of aluminum jackets at 550°C.

Metallography

The microstructure received some further study on metal of known purity. A series of heat treatments was made on samples of the hot rolled bar prepared from Billet M310, L128. Examination of the micro-structure developed by these heat treatments was made.

Figure 61 shows the structure of this metal after hot rolling at 1680°F (920°C) followed by quenching. After receiving this treatment, the metal was then subjected to 2 hours heating at 900°C followed by quenching, furnace cooling, and a gas quench equivalent to air cooling. Figures 62, 63, and 64 show the results of these heat treatments. The difference between the three heat treatments is quite marked. These specimens were polished and etched in the same mounting and only one, the furnace cooled specimen developed the lamellar structure. The gas cooled specimen shows a structure intermediate between the quenched and furnace cooled treatments.

Figures 65 and 66 show similar specimens heated 2 hours at 700°C, somewhat above the lower transition, followed by quenching and furnace cooling. The cooling rate from this temperature appears to have been roughly equivalent to that of the specimen gas cooled from 900°C as a very similar structure was obtained. The specimen furnace

21111
500X

Figure 61. Billet M310, L128 after Hot Rolling at 920°C and Quenching.

21112
500X

Figure 62. M310, L128 Heated to 900°C 2 Hours and Quenched after Hot Rolling.

21113
500X

Figure 63. M310, L128 Hot Rolled, Reheated to 900°C 2 Hours and Furnace Cooled.

21114
500X

Figure 64. M310, L128 Hot Rolled, Reheated to 900°C 2 Hours and Gas Cooled (air quench).

21115
500X

Figure 65. M310, L128 Hot Rolled, Quenched and Reheated to 700°C 2 Hours and Water Quenched.

21116
500X

Figure 66. M310, L128 Hot Rolled, Quenched and Reheated 700°C 2 Hours and Furnace Cooled.

cooled from 700°C shows the lamellar structure but to a less extent than the specimen furnace cooled from 900°C, showing somewhat more tendency for spheroidization of the lamellae.

Specimens of the same metal were similarly treated at 600°C just below the lower transition, and show, in Figures 67, 68, and 69, similar structures, the furnace cooled specimen showing a partially spheroidized but more lamellar structure than when treated at 700°C.

In considering the structures obtained at 600 and 700°C, the previous thermal history should be kept in mind. Since this metal was quenched from about 920°C after hot rolling, the reheating at these lower temperatures would correspond to a tempering treatment on quenched steel, and intermediate structures between the quenched and furnace cooled specimens would be anticipated. While the cooling rate from these lower temperature treatments appears to modify the structures obtained, these structures are all similar in that a phase appears to be separating from solid solution, at grain boundaries, slip planes or, with furnace cooling, as lamellae. The structures of the metal when heat treated in the same manner after a furnace cooling or full annealing treatment would be of considerable assistance in studying these structural changes further.

Figure 70 shows the structure of a sample submitted as pure metal in the as received condition. This shows considerable areas of the lamellar structure but also large plain areas similar to a hypoeutectoid steel.

21117
500X

Figure 67. M310, L128 Hot Rolled and Quenched,
Reheated at 600°C 2 Hours and
Quenched.

21118
500X

Figure 68. M310, L128 Hot Rolled and Quenched,
Reheated at 600°C 2 Hours and
Furnace Cooled.

21119
500X

Figure 69. M310, L128 Hot Rolled and Quenched, Reheated to 600°C and Gas Cooled (air quench).

21120
500X

Figure 70. Structure of "pure metal" in As Received Condition.

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FUTURE WORK

Additional tests on aluminum jackets are in progress and these tests are being continued.

Specimens suitable for jacketing are being out-gassed by heating to 600°C in lead, argon, and vacuum. Some of these will be jacketed and tested by heating in air at 550°C and others will be tested to determine the amount of gas evolved at 750°C.

Further studies of the gas-metal relationship are to be made to establish the most suitable temperature for out-gassing treatment and the maximum temperature which may safely be used without out-gassing.

Carbon analyses are to be made on the metal studied metallographically to assist in explanation of the structures obtained.

Some further studies on machinability are in progress and will soon be available.

April 10, 1943

HWR: HAP: LHG:va

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