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PROGRESS REPORT

ON

# HIGH TEMPERATURE METALLURGICAL CHARACTERISTICS OF 2 1/4 Cr 1 Mo STEEL TUBES MADE BY THE MICHIGAN SEAMLESS TUBE COMPANY

by

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# HIGH TEMPERATURE METALLURGICAL CHARACTERISTICS OF 2 1/2 Cr 1 Mo STEEL TUBES MADE BY THE MICHIGAN SEAMLESS TUBE COMPANY

The Michigan Seamless Tube Company produces seamless tubing which is used at high temperatures in applications under the jurisdiction of the Codes of the ASME Power and Pressure Vessel Committee. An investigation is in progress to determine if the tubing as produced by the Michigan Seamless Tubing Company has the creep-rupture properties expected for the stresses allowed by the Codes for such tubing.

The creep-rupture properties of 2 1/4 Cr 1 Mo steel are known to vary over a considerable range. The reasons for the range are not understood. Presently specified chemical composition alone will not determine the level of the properties. ASTM Specification A-213 attempts to insure at least a minimum level of strength by restricting heat treatment. The matter is sufficiently uncertain, however, so that users of the tubing, particularly in superheaters, can be concerned that the production procedures of each supplier will not result in unexpectedly low properties. It is just as important to the Michigan Seamless Tube Company to know that their production procedures do not result in low properties as it is to their advantage to know if the properties developed by their procedures are at high levels.

The research currently in progress extends the information resulting from a survey of tubing made and heat treated several ways as presented in The University of Michigan Report No. 05007-30-T, entitled: "A Survey of Creep Rupture Properties at 1100°F of 2 1/4 Cr 1 Mo Steel Tubes Made by Michigan Seamless Tube Company," and dated July 31, 1964. The following are the specific objectives:

1. Extend the creep-rupture data from the July 31, 1964 Report on selected tubes to time periods approaching 10,000 hours in order to satisfy the presently widely held view that tests of this duration are necessary to establish reliable creep-rupture strengths.

2. Determine the creep-rupture properties of tubes heat treated by a new modified "isothermal" process.

3. Make laboratory heats with low and high nitrogen to determine if there is an affect of nitrogen on creep-rupture properties.

## Materials Investigated

The compositions of the tubes being considered in this investigation are given in Table 1. This Table was made up by simply adding the information to Table 1 of the July 31, 1964 Report for Tube No. 15. Tube No. 15 is the new tube with the modified isothermal treatment.

## Extension of Creep-Rupture Testing at 1100°F on Selected Tubes Surveyed for Report No. 05007-30-T

### Tube No. 12 (Special Anneal at 1450°F)

Tube No. 12 had the highest extrapolated 100,000-hour rupture strength at 1100°F (9,600 psi) of all those investigated for Report 05007-30-T. A specimen was machined and a test was started at 11,800 psi to check this high strength with the expectation that it would have a rupture time of 6000-7000 hours if the original stress-rupture time curve was correct. The following test results were obtained:

| | |
|---|---|
| Temperature | 1100°F |
| Stress | 11,800 psi |
| Rupture Time | 2443.6 hours |
| Elongation | 32.5% |
| Reduction of Area | 62.8% |

This test result is included in Table 2 along with the results previously obtained. The specimen used was of full wall thickness and 0.5-inch wide.

The rupture time was much shorter than the stress-rupture time curve of Figure 2 of Report No. 05007-30-T predicted.* Experience with stress rupture testing indicates that the stress-rupture time curve increases in slope

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* Figures 2 and 3 from Report No. 05007-30-T were reproduced and the data from the current research added in red to show the results of the research in progress.

at about 1000 hours and probably extrapolates to about 7000 psi at 100,000 hours. It will be noted that, as drawn, the slope of the curve after the increase in slope is similar to that for the other tubes, lending support to the extrapolation of the curve as now shown.

The specimen was examined microscopically and no unusual features were observed. The ductility dropped from the previous test. Both observations tend to support the increase in slope of the stress-rupture time curve as characteristic of the material.

There has been discussion of conducting a test on a 0.250-inch diameter specimen to check the slope of the curve. This should receive further consideration before conducting the test.

## Long Time Rupture Test on a Full Sized Specimen

After the test on Tube No. 12 at 11,800 psi ruptured, it was decided to start another full wall specimen for a long duration (5000-10,000 hours) test. A specimen was machined from Tube No. 14 for this purpose. The status of this test is:

| | |
|---|---|
| 12,000 psi | In Progress 646 hours (as of 4-10-67) |

## Modified Isothermal Treatment (Tube No. 15)

Four full wall rupture specimens were machined and two rupture tests have been started at 1100°F:

| | |
|---|---|
| 19,000 psi | In Progress 142 hours (as of 4-10-67) |
| 16,000 psi | In Progress 646 hours (as of 4-10-67) |

These points have been added to a copy of Figures 2 and 3 taken from Report No. 05007-30-T. The tests have been in progress longer than the tests previously conducted on material annealed at 1750°F or normalized plus tempered. The rupture times are approaching the longest of any obtained to date after the special 1450°F anneal (Tube No. 14).

Creep Test on Tube No. 13

Both rupture and creep strengths are used in evaluating steels for Code use. Because annealing at 1450°F is a somewhat unusual heat treatment, it was deemed necessary to be sure that it does not result in abnormally low creep strength. The tube (No. 13) with the apparently lowest rupture strength with the special 1450°F anneal was selected on the basis that if its creep strength was adequate, there would be no question about the creep strength of the tubes with higher rupture strengths.

The test was run at a stress of 4,200 psi, the current Code stress, at 1100°F. The creep curve (Fig. 2) indicates a creep rate of 0.02 per cent per 1000 hours. This would extrapolate to a stress of 3600 to 3800 psi for a creep rate of 0.01 per cent per 1000 hours. This creep strength is at the low side of the range for heat treatment "above or near top of critical range"--one interpretation of the heat treatment clause of the present A-213 Specification and the treatments used for setting the Code stress. Because Tube No. 1 also had a creep strength of about 3800 psi, there seems to be an indication that treatment at 1450°F might not develop as high creep strengths as treatment at higher temperatures. While the creep rate now appears to be fairly constant, it is suggested that the test be continued for another 1000 to 1500 hours in order to check whether or not it might not still decrease to a lower rate. Tests on 2 1/4 Cr 1 Mo steel often require rather long time periods before attaining minimum creep rate.

Effect of Nitrogen

In accordance with previous agreement, laboratory heats have been made with low and normal or high nitrogen content. The heats have been made, rolled to bar stock and heat treated. The aim compositions were as follows:

| | |
|---|---|
| Carbon | 0.12 per cent |
| Manganese | 0.45 per cent |
| Silicon | 0.30 per cent |
| Chromium | 2.25 per cent |
| Molybdenum | 1.00 per cent |
| Nitrogen | low (none added), 0.01 and 0.02 per cent |

The heats were melting in a vacuum induction furnace and carbon deoxidized. Experience has indicated that this procedure provides material with properties similar to production furnace heats by avoiding the oxygen-nitrogen reactions of air melting in small induction furnaces with the accompanying complications from the need for excessive use of solid deoxidizers.

Nitrogen was added as chrome nitride. Split heats were used, the first half with no added nitrogen and the second half with added nitrogen, so that the nitrogen effect would not be influenced by other heat-to-heat effects, if any. The heats were hot rolled, cold reduced 40 per cent and heat treated. Samples have been sent to a commercial laboratory for chemical analysis.

Stock sufficient for 3 or 4 specimens were given the following heat treatments:

a) Heated 1/2 hour at 1450°F, air cooled.

b) Heated 1/2 hour at 1750°F, cooled at 50°F per hour to 1100°F, and furnace cooled to room temperature.

Currently it is planned to machine 0.250-inch diameter specimens and to conduct tests on the material with no nitrogen added and then with an addition of 0.02 per cent. The material heat treated at 1450°F will be used.

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Note: A student melted, rolled, and heat treated the heats with MST support only for materials and as technicians assistance.

Table 1

## DESCRIPTION OF SAMPLES OF TUBES SUBMITTED

| Tube No. | Heat No. | Tube Size | | Heat Treatment | Chemical Composition Reported | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Dia. (in.) | Wall (in.) | | C (%) | Mn (%) | Si (%) | Cr (%) | Mo (%) | P (%) | S (%) |
| 1 | 3341384 | 2 | 0.404 | Annealed 1450°F[a] | .13 | .44 | .28 | 2.22 | 1.02 | .010 | .017 |
| 2 | 3341384 | 2 | 0.404 | Norm. 1650°+Temp. 1375°F[b] | .13 | .44 | .28 | 2.22 | 1.02 | .010 | .017 |
| 3 | ? | 2 3/8 | 0.404 | Annealed 1450°F[a] | | | | ? | | | |
| 4 | 3320643 | 2 1/8 | 0.471 | Annealed 1450°F[a] | .11 | .44 | .31 | 2.25 | 1.02 | .010 | .008 |
| 5 | 27218[e] | 2 1/8 | 0.471 | Annealed 1450°F[a] | .11 | .46 | .30 | 2.31 | 0.94 | .013 | .025 |
| 6 | 27218[e] | 2 1/8 | 0.471 | Annealed 1750°F[c] | Same as Tube No. 5 | | | | | | |
| 7 | 3341339 | 2 1/8 | 0.450 | Cold drawn+1450°F[a] | .11 | .42 | .29 | 2.23 | 0.95 | .008 | .013 |
| 8 | 3341339 | 2 1/8 | 0.450 | Tube reduced+1450°F[a] | .11 | .42 | .29 | 2.23 | 0.95 | .008 | .013 |
| 9 | 3341339 | 2 1/8 | 0.440 | Cold drawn+Annealed+ 1750°F[c] | .11 | .42 | .29 | 2.23 | 0.95 | .008 | .013 |
| 10 | 3341339 | 2 1/8 | 0.450 | Cold drawn+Norm. and Temper.[d] | .11 | .42 | .29 | 2.23 | 0.95 | .008 | .013 |
| 11 | 3341339 | 2 1/8 | 0.450 | Cold drawn+Water quench. 1700°F+ temper. 1450°F | .11 | .42 | .29 | 2.23 | 0.95 | .008 | .013 |
| 12 | 3342219 | 2 1/8 | 0.452 | Duplicates No. 7 | .105 | .47 | .30 | 2.27 | 0.96 | .008 | .012 |
| 13 | 332108 | 2 1/8 | 0.452 | Duplicates No. 12 | .105 | .45 | .33 | 2.29 | 0.98 | .008 | .016 |
| 14 | 30558[e] | 2 1/8 | 0.452 | Duplicates No. 12 | .11 | .45 | .32 | 2.25 | 0.90 | .013 | .025 |
| 15 | 3322242 | 2 1/8 | 0.452 | Modified isothermal[f] | .115 | .45 | .29 | 2.10 | 0.97 | .010 | .007 |

(a) Cold worked tube was heated to 1450°F, 1 hour at heat and air cooled.
(b) Tubing heat treated at 1450°F from the same lot as Tube No. 1 was reheated to 1650°F for 1 hour and air cooled plus tempered at 1375°F for 1 hour, air cooled.
(c) Cooled at 50°F per hour from 1750°F.
(d) Fast cooled from 1750°F+tempered at 1375°F for 1 hour.
(e) Tubes 5, 6 and 14 were made from steel from a different steel producer than the other tubes.
(f) Cold draw before modified isothermal anneal.

Table 1 (continued)

| Tube No. | Ultimate Strength (psi) | Yield Strength (psi) | Elongation (% in 2") | Hardness Rockwell "B" | Piercing and Rolling | Heat Treatment Prior to Cold Working to Size (°F) | (hours) | (cooling) |
|---|---|---|---|---|---|---|---|---|
| 1 | 67,600 | 34,700 | 57 | 72 | >1800°F | 1650 | 3 | 50°F/hr to 1100°F |
| 2 | 69,300 | 46,600 | 65.6 | 76/77 | Same as 1 except reheat treated | | | |
| 3 | 68,700 | 40,100 | 64.8 | 75/77 | >1800°F | 1450 | 1 | Air cooled |
| 4(a) | 69,000 | 50,800 | 57 | 74/75 | to 2 7/8" x 9/16" | 1450 | 1 | Air cooled |
| 5(a) | 70,000 | 48,700 | 54.6 | 77/78 | to 2 7/8" x 9/16" | 1450 | 1 | Air cooled |
| 6 | | | | | Same as Tube No. 5 except final heat treatment | | | |
| 7-10 | 84,400(b) | 58,700 | 53.1 | 72/76 | | | | |
| 11 | 78,200 | 63,200 | 53.9 | 85 | | | | |
| 12 | 72,100 | 41,000 | 56.2 | 72/74 | | | | |
| 13 | 84,200 | 61,600 | 48.4 | 85 | Same as Tube No. 12 | | | |
| 14 | 84,000 | 56,600 | 53.9 | 85 | Same as Tube No. 12 | | | |
| 15 | 80,600 | 63,400 | 51.5 | 81/82 | | | | |

(a) To compare tubes of steel from two suppliers, made the same way.
(b) May be heat certification properties.

Table 2

STRESS RUPTURE TIME DATA AT 1100°F FOR
MICHIGAN SEAMLESS TUBE COMPANY 2 1/4 Cr - 1 Mo STEEL TUBING

| Tube No. | Stress (psi) | Rupture Time (hrs) | Elongation (% in 2 in.) | Reduction of Area (%) |
|---|---|---|---|---|
| | | Heated for one hour at 1450°F, air cooled | | |
| 1 | 20,000 | 25.9 | 63.5 | 87.0 |
| | 17,000 | 63 | 72.0 | 84.5 |
| | 15,000 | 155 | 71.0 | 84.5 |
| | 13,000 | 560 | 74.5 | 81.0 |
| | 11,000 | 1664 | 62.5 | 77.0 |
| 3 | 17,000 | 86.2 | 47.0 | 83.0 |
| | 15,500 | 174 | 71.0 | 84.0 |
| | 14,000 | 562 | 52.5 | 81.0 |
| | 12,500 | 1187 | 69.5 | 83.5 |
| 4 | 16,000 | 106 | 54.0 | 59.0 |
| | 14,800 | 151 | 45.5 | 60.0 |
| | 13,000 | 401 | 39.0 | 55.5 |
| 5 | 16,000 | 187 | 38.5 | 53.5 |
| | 14,500 | 280 | 33.5 | 46.5 |
| | 13,500 | 410 | 35.5 | 49.5 |
| | 12,000 | 974 | 38.5 | 50.0 |
| 7 | 19,000 | 238 | 49.0 | 80.0 |
| | 17,000 | 384 | 68.5 | 81.0 |
| | 15,500 | 814 | 55.5 | 77.5 |
| 8 | 17,000 | 79.4 | 67.0 | 87.5 |
| | 14,000 | 265 | 59.0 | 86.5 |
| | 12,000 | 1005 | 68.5 | 86.5 |
| 12 | 16,000 | 123 | 51.0 | 55.5 |
| | 14,500 | 449 | 50.0 | 70.0 |
| | 13,500 | 1063 | 56.0 | 62.5 |
| | 11,800 | 2443.6 | 32.5 | 62.8 |

Table 2 (continued)

| Tube No. | Stress (psi) | Rupture Time (hrs) | Elongation (% in 2 in.) | Reduction of Area (%) |
|---|---|---|---|---|
| 13 | 16,500 | 369 | 59.5 | 65.5 |
| | 14,500 | 905 | 41.0 | 66.0 |
| | 13,500 | 1516 | 45.0 | 61.5 |
| 14 | 16,500 | 864 | 43.5 | 63.5 |
| | 14,500 | 1575 | 43.5 | 67.5 |
| | 13,500 | 3225 | 36.5 | 56.0 |
| | 12,000 | In Progress 646 hours (4-10-67) | | |
| Heated for one hour at 1650°F, air cooled and tempered for one hour at 1375°F, air cooled | | | | |
| 2 | 17,000 | 180 | 55.0 | 81.0 |
| | 15,000 | 393 | 58.0 | 82.5 |
| | 12,000 | 1924 | 55.0 | 74.0 |
| Heated for one hour at 1750°F, air cooled and tempered for one hour at 1375°F | | | | |
| 10 | 17,000 | 299 | 60.5 | 85.0 |
| | 15,000 | 726 | 49.0 | 82.5 |
| | 14,000 | 1181 | 50.5 | 83.0 |
| Furnace cooled 50°/hour from 1750°F | | | | |
| 6 | 17,000 | 70.5 | 85.0 | 76.5 |
| | 14,000 | 335 | 59.5 | 70.5 |
| | 12,500 | 799 | 60.0 | 61.0 |
| 9 | 17,000 | 62.2 | 76.5 | 91.5 |
| | 14,000 | 305 | 72.0 | 89.0 |
| | 12,500 | 697 | 62.5 | 90.0 |
| Modified Isothermal treatment | | | | |
| 15 | 19,000 | In Progress 142 hours (4-10-67) | | |
| | 16,000 | In Progress 646 hours (4-10-67) | | |