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# ELEVATED TEMPERATURE PROPERTIES FOR 17 - 14 Cu + Mo ALLOY

by

R. Jackowski J. W. Freeman

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THE TIMKEN ROLLER BEARING COMPANY STEEL AND TUBE DIVISION CANTON, OHIO

# ELEVATED TEMPERATURE PROPERTIES FOR 17 - 14 Cu + Mo ALLOY

The 17 - 14 Cu + Mo alloy had been developed as a high strength material at and above 1200°F, particularly at about 1350°F. Available information indicated that it was suitable for use as superheater tubing in boilers generating steam at 1100°F to 1200°F range. The high strength for metal temperatures in excess of 1200°F was particularly attractive.

The data in this report present the results of creep-rupture tests mainly at 1350°F with some testing at 1200° and 1500°F. The materials investigated covered:

- (a) Barstock from experimental induction heats
- (b) Tubing made from centrifugal castings
- (c) Tubing from small arc furnace heats
- (d) Tubing from production heats

Conditions of heat treatment were surveyed for the treatment best suited for tubing. The initial tubing investigated was centrifugally cast and rotorolled from a heat with Ti omitted from the composition. Subsequent specimens were supplied from conventionally pierced and rolled tubes. The centrifugally cast blanks were used early in the program before it was known that the alloy could be pierced.

Chemical composition other than the omission of Ti in the centrifugally cast tube blanks was only varied to the extent that one experimental heat was made with a boron addition.

#### CONCLUSIONS

The high long time strengths proposed as characteristic for the alloy were reproduced only in Heats 02706 and 02708 when heat treated at 2150°F and water quenched. The stresses for rupture in 100,000 hours were generally otherwise no higher than the upper side of the range for Types 316H and 347H austenitic steel tubing.

The data indicated that a water quench from 2150°F would produce the best creep-rupture strength. However, this apparently did not produce the high strength in tubes from production heats indicated by the initial "Zero" heats. This conclusion may have been incorrect due to inadequate testing to be sure of the extrapolation to 100,000 hours. This in combination with the other data suggest that the properties were considerably dependent on prior processing and/or melting conditions. For instance, an 1850°F W.Q. resulted in better properties for the cast and cold reduced material than a 1950°F W.Q. Material made from ingots hot rolled to bars or tubes had to be heat treated to about 2150°F to produce best properties. It is uncertain to what degree the omission of Ti from the cast and rotorolled tubes influenced the properties in comparison with pierced tubes.

In one case "Aging" at 1350°F appeared to reduce strength after a 2150°F water quench with possibly a slight increase in ductility in the rupture tests. It apparently caused a marked reduction in time for the start of third-stage creep. This is difficult to correlate with the data for unaged specimens tested at 1350°F. The unaged specimens were necessarily considerably exposed to 1350°F prior to the application of loads for the tests.

The experimental heat with boron added showed no beneficial effect. The analyzed boron was, however, 0.0005%, an amount normally too low to be expected to appreciably improve properties, although low Ti and Cb in the heat may have masked the effect of the boron addition.

#### MATERIAL INVESTIGATED

Specimens from six heats of 17-14Cu + Mo alloy were supplied during the course of the investigation. Four of the heats (Heats A218, 02706, 18443 and 18439) had compositions within the nominal range for the alloy. The titanium addition was not made to the fifth heat (Heat 2649) and the sixth heat (Heat 02708) was modified by the addition of boron. The chemical analysis reported by The Timken Roller Bearing Company for these six heats are presented in Table 1.

All specimens from tubes were taken longitudinally. The heat treatments, grain size, hardness and other information Timken reported for materials tested were as follows:

#### Barstock - Heat A218

Standard 0.505-inch diameter tensile specimens were supplied from barstock from this experimental heat. The barstock was reported to have been oil quenched from 2000°F. The hardness after heat treating was 152 Brinell and the ASTM grain size was 7 and 8.

# Centrifugally Cast and Rotorolled Tube - Heat 2649

Tensile specimens, 0.250-inch in diameter, were supplied from a 2.125-inch O.D. by 0.375-inch wall rotorolled tube. A centrifugally cast shell was given two rotoroll passes with and in-between pass anneal of 2100°F. In addition to being cast and cold reduced, titanium had been omitted from the composition. Sections taken from the tube were given the following heat treatments:

Heat Treatment	BHN	ASTM Grain Size
(1) Air cooled from 1750°F	163	7-8
(2) Water quenched from 1850°F	159	7-8
(3) Water quenched from 1950°F	159	7-8
(4) Specimens water quenched from 2250°F (a)	136 (b)	5-7

<sup>(</sup>a) - The 2250°F treatment was performed at the University of Michigan on specimens originally water quenched from 1750°F.

<sup>(</sup>b) - Converted from Vickers (50 Kg load) Diamond Pyramid Hardness Number.

## Pierced Tube - Heat 02706

Specimens 0.357-inch in diameter from a 3.625-inch O.D. by 0.625-inch wall tube produced by piercing a 3 7/8-inch billet were supplied in the following conditions:

Heat Treatment	BHN	ASTM Grain Size
(1) Water quenched from 2150°F	143	6-7(5)
(2) Water quenched from 2250°F	131 Mi:	xed from 6 to larger than 1

### Pierced Tubes - Heats 18443 and 18439

Specimens 0.357-inch in diameter were supplied from a pierced tube from each of two heats (Heats 18443 and 18439). The machined specimens were heat treated at the University of Michigan as follows:

		BHN*		ASTM Grain Size		
		Heat	Heat	Heat	Heat	
	Heat Treatment	18443	18439	18443	18439	
(1)	As received	148	160	6-8	5-6	
(2)	Water quenched from 2050°F	121	130	7 -8	6-8	
(3)	Water quenched from 2150°F	119	122	7-8(6)	6-7(5)	
(4)	Water quenched from 2150°F and aged 5 hours at 1350°F	134	136	6-8(5)	6-7(5)	

<sup>\*</sup> Converted from Vickers (50 Kg load) Diamond Pyramid Hardness Number.

After heat treatment, the gage section diameter of each specimen was remachined to 0.300 inch to eliminate surface imperfections resulting from heat treatment.

# Boron Modified 17-14 Cu + Mo - Heat 02708

Specimens 0.357-inch in diameter were supplied from a heat modified by the addition of boron. The analyzed boron was only 0.0005 percent, an amount too low to be expected to have much effect if the alloy responds to boron. The titanium and columbium were lower than normal for 17-14 CuMo alloy. The heat treatment was a water quench from 2150°F. The hardness was 134 Brinell and the ASTM grain size was 4-6.

#### RESULTS

The properties of the 17-14Cu + Mo alloy were evaluated by means of creep-rupture tests and/or tensile tests at temperatures from 1200°F to 1500°F. In addition, metallographic studies were made of the structures before and after testing.

#### Barstock - Heat A218

Rupture tests were run on specimens from barstock oil quenched from 2000°F to establish the strength at 1200° and 1350°F. The rupture strengths and ductilities are presented in Table 2 as derived from the data (Table 3) and stress-rupture time curves of Figure 1.

The maximum time for rupture was approximately 1000 hours. The stress-rupture time curves underwent increases in slope at about 400 hours at 1200°F and about 100 hours at 1350°F. While the strengths for short time periods were about as high as expected for the alloy (Table 2), the long time strengths were considerably lower. The estimated stresses for rupture in 100,000 hours were only slightly above average for Types 316H and 347H austenitic steels.

The slopes of the stress-rupture time curves as extrapolated (Fig. 1) are not well established. The long time strengths could not therefore be accurately established. The extrapolation at 1200°F was based on only one point on the curve after the increase in slope. The slope of the curve at 1200°F was not as steep as at 1350°F, suggesting that the long time strengths indicated could be high.

Representative structures of the material prior to and after testing are shown by Plates 1, 2 and 3. The as-received material, Plate 1, had a structure of twinned austenite grains and considerable undissolved precipitates that are presumably complex carbides. During testing at 1200°F, Plate 2, a fine precipitate formed in the grain boundaries which etched readily and outlined the grain boundaries clearly. When tested at 1350°F, Plate 3, the precipitates were larger and did not outline the grain boundaries

as clearly. The grain size was mostly 7-8 but included some as large as 5-6. There was some banding. The intergranular cracks at the fracture and at the surface were most predominant in the specimen tested at 1350°F.

The microstructure of the specimen tested at 1350°F suggested that some sigma phase might be forming as small particles in the grain boundaries.

# Centrifugally Cast and Rotorolled Tube - Heat 2649

Tensile tests, run at 1200°, 1350° and 1500°F on specimens of the tube as normalized from 1750°F to aid in choosing the initial stress for the rupture tests, gave the following properties:

Elevated Temperature Tensile Properties of the Centrifugally Cast and Rotorolled Tube Normalized from 1750°F

Temp.	Tensile Strength	Offset Yield	d Strength (psi)	Elongation	Reduction of
(°F)	(psi)	0.1%	0.2%	(% in 1 inch)	Area (%)
1200	58,000	29,500	32,250	43.0	51.0
1350	38,700	25,000	26,500	49.0	45.0
1500	25,300	18,500	20,000	47.0	40.5

These tensile properties are within the range for 18-8 type austenitic steels and indicate the high ductility to be expected for heat treatment at as low a temperature as 1750°F. Rupture tests (Table 4) gave stress-rupture time curves (Fig. 2) which indicated that both short and long time strengths were low in comparison with those to be expected for the alloy as a result of heat treatment at the low temperature of 1750°F. When this was recognized, testing was discontinued. Therefore, the maximum times for rupture were short. The ductility was high but perhaps not as high as would be expected for heat treatment at 1750°F. Extrapolated values for long time strengths, although very uncertain, have been included in Table 2 to indicate the level of strength. The values obtained by extrapolation at 1200°F are probably high as suggested by the much steeper

curves at 1350° and 1500°F.

When the temperature of heat treatment was 1850°F, the stress-rupture time data (Table 4 and Fig. 2) indicated considerably higher strengths with good ductility. The strengths were on the low side of the range (Table 2) to be expected for the alloy and considerably above those to be expected for 18-8 type steels. Due to considerably less slope of the curve at 1200°F than at 1350°F, the long time strengths in Table 2 for 1200°F probably are somewhat high.

When heat treated at 1950°F the rupture tests gave data (Table 4) which when plotted as stress-rupture time curves indicated marked increases in the slopes (Fig. 2) at 1000 and 1500 hours at 1200° and 1350°F and probably at 1500°F. The result was somewhat lower strengths (Table 2) at long time periods than when heat treated at 1850°F. Short time strengths were at most only slightly higher than those of the material treated at 1850°F. Tests were as long as 12,594 hours at 1350°F. The long time strength was therefore established quite well. The tests at 1200° and 1500°F were shorter in duration. The curves were drawn consistent with the curve based on the long time tests at 1350°F and should therefore be quite reliable. Elongation in the rupture tests was only slightly lower than that of the tubing treated at 1850°F over the range of time periods used in the tests for both materials. Extending the testing time at 1350°F resulted in elongation of the order of 4 to 5 percent.

The heat treatments used developed strengths only on the low side of the range considered typical for the alloy. Because there would be the possibility that heat treatment at higher temperatures would dissolve Ti and Cb carbides and thereby raise strength, specimens were re-heat treated at 2250°F. Single survey tests were run at 1350° and 1500°F (Table 4). When plotted with the data for the other heat treatments (Fig. 2) the points were somewhat higher than those for treatment at 1750° or 1950°F and slightly below those for 1850°F. Single tests do not, of course, define a curve for extrapolation to long time periods. The very low elong-

ation at 1350°F of 1.5 percent indicated that the heat treatment at 2250°F would not be useful, at least for cast, cold worked and heat treated materials.

A single creep test was run 2461 hours at 1350°F under a stress of 8000 psi (Fig. 3) for material water quenched from 1950°F. The creep rate at this time was 0.041 percent per 1000 hours. The creep curves from the more prolonged rupture tests at 1200°, 1350° and 1500°F are included as Figures 3 and 4. The very limited creep data indicate that the creep resistance was lower than that reported for wrought barstock water quenched from 2050°F in the ASTM-ASME Special Technical Publication No. 124. It would, however, be at the top of the range or higher than those reported for 18-8+Mo (Type 316) and for 18-8+Cb (Type 347) austenitic steels. The test at 10,000 psi (Fig. 4) gave creep data which plotted as a very unusual curve. It is possible that complex precipitation caused this although this would be unusual. The rupture time was consistent with the shorter time tests. The presence of patches of a eutectic structure was very noticeable in the microstructure. Increasing the temperature of solution treatment from 1750° to 2250°F (Plates 4, 8, 12 and 16) reduced the amount of the eutectic type structure present as "grains" strung out lengthwise to the tube by the cold reduction. The eutectic formed during solidification of the casting. Even heat treating at 2250°F did not completely eliminate it. This type of structure is common in such complex alloys, particularly those containing Cb.

The increasing temperatures of heat treatment did not seem to dissolve the general matrix precipitates as much as might be expected (compare Plates 4, 8, 12 and 16). The grain size was about the same (7-8) for heat treatments at 1750°, 1850° and 1950°F. Only the treatment at 2250°F gave some coarsening (2 to 6).

The specimens after rupture testing (Plates 4 through 18) all exhibited considerable precipitation both within the grains and in the grain boundaries. The structures apparently were delineated by etching much more easily after rupture testing, as a result of the precipitation. Consequently,

the easily etched eutectic structure patches of the original material were not very prominent after testing at 1200° and 1350°F. When tested at 1500°F, however, the precipitates were agglomerated more and the structure became more difficult to etch. As a consequence, the patches of eutectic were nearly as prominent as in the original material.

The microstructures at 100X showed dark bands associated with the eutectic patches. These presumably originated with the solution of elements in the eutectic structures during heat treatment. Evidently some of the elements in the eutectic structure had not diffused into the matrix very far even when the temperature of treatments was 2250°F. During testing these formed compounds and precipitated.

The 12,594-hour test at 1350°F on the material heat treated at 1950°F (Plate 14) had a precipitate in the grain boundaries which etched readily. The precipitate could be an agglomerated complex carbide or carbonitride although it could have also been largely sigma phase. In general, there was surprisingly little difference in microstructures with either heat treatment or testing. It is suspected that the change in slope of the stress-rupture time curve for the material heat treated at 1950°F was due to reduced ductility resulting from the increased solution during heat treatment and precipitation in the grain boundaries. Fractures and cracking during tests were largely intergranular.

The most evident result of the metallographic examination was the inability to associate microstructure with creep-rupture properties.

The heat was made without an addition of titanium. For this reason it is difficult to be sure to what degree the properties were characteristic of the alloy cast, rotorolled and heat treated, or of the omission of titanium. In particular, this could have a considerable effect on the amount of eutectic in the structure. Such eutectics are characteristic of Cb but it could be that suppression of the eutectic was involved in the structure of the normal alloy.

### Pierced Tube - Heat 02706

The pierced tubing from the experimental arc furnace Heat 02706 was rupture tested at 1350°F as water quenched from 2150°F and as water quenched from 2250°F. These temperatures of heat treatment were higher than had been used in previous heats in an effort to develop the high strength considered typical of the alloy.

The rupture data (Table 5) and the stress-rupture time curves (Fig. 5) for the heat treatment at 2150°F indicated rupture strengths at 1350°F (Table 2) which were as high as those expected for the alloy. When heat treated at 2250°F the short time strengths were high. The stress-rupture time curve was, however, steep, with the result that the extrapolated long time strengths were low.

Three tests ranging from 1000 to 5800 hours were used to establish the curves. These prolonged tests should have established the stress-rupture time curves quite well for extrapolation.

The material heat treated at 2150°F had good ductility while that heat treated at 2250°F fractured with low ductility (see Table 5 and Fig. 5).

Creep curves (Fig. 6) for the two longer time rupture tests in each condition show that the specimens solution treated at 2150°F had a very short period of primary and secondary creep followed by a very long period of tertiary creep. The creep rates for those heat treated at 2150°F were too high to establish a "creep strength". The specimens solution treated at 2250°F had less primary creep with lower creep rates during prolonged secondary creep compared with the material heat treated at 2150°F. As the low elongation indicated, there was practically no third-stage creep.

The minimum creep rate for the test at 12,500 psi on the specimen water quenched from 2250°F was 0.016 percent per 1000 hours (Fig. 7). The 0.016 percent per 1000 hours was close enough to 0.01 percent per 1000 hours to indicate a creep strength for this rate of 10,000 psi. The early onset of third stage creep, however, shows that this strength is unsuit-

able for extrapolation. The extrapolated 100,000-hour rupture strength was only 3500 psi. It is evident that rupture in a brittle manner would occur in less than 10,000 hours under the stress for a creep rate of 0.01 percent per 1000 hours. Unless structural changes occurred fairly rapidly to induce more ductility and to flatten out the stress-rupture time curve, the heat treatment at 2250°F in this case at least results in a poor material.

The grain size after solution treatment at 2150°F (Plate 19) was 6 to 7 with some as large as 5. The most noticeable feature, however, was the apparent complete solution of precipitate compared with the previously discussed heats. The grain size when solution treated at 2250°F was mixed, ranging from larger than 1 to 6 (Plate 21). During testing at 1350°F, fine precipitates formed throughout the matrix (Plates 20 and 22). A heavier concentration of the fine precipitates tended to outline the grain boundaries. Some of the precipitation in the specimens water quenched from 2250°F formed on orientated crystallographic planes. This was not evident in the specimen water quenched from 2150°F. The cracking at the fracture and at the surface adjacent the fracture was predominantly intergranular in nature.

The material heat treated at 2250°F had a peculiar structure at the grain boundaries. The boundaries seemed to be free of precipitates while there were line of precipitates on both sides of the boundary.

# Pierced Production Tubes - Heat 18443 and 18439

A limited number of creep and rupture tests were run at 1350°F on specimens from two production heats. Three conditions of heat treatment were evaluated, water quenched from 2050°F, from 2150°F and from 2150°F, followed by a five-hour age at 1350°F.

The tests on specimens from Heat 18443 were all conducted at 20,000 psi (Table 6). There were no real differences in the rupture times (Fig. 8) which ranged from 250 to 350 hours. Elongations were of the order of 5 to 8 percent. It is quite evident that the tube tested did not have either

the high strength or high ductility of the tube from Heat 02706 heat treated at 2150°F as discussed in the preceding section.

Tests on Heat 18439 specimens were run at two stresses. The data (Table 6) and the stress-rupture time relationships (Fig. 8) indicate rather steep stress-rupture time curves. Extrapolation of the "Curves" based on the two tests suggests rather low long time strengths for both heat treatments. The specimens from Heat 18439 had considerably higher ductility that those from Heat 18443 but not nearly as high as Heat 02706. The "rupture strengths" given in Table 2 are mainly to indicate the general level of strength. A dashed line is shown in Figure 8 to indicate that it might be possible that tests would have indicated higher strengths at the longer times than the conservative curves used. The testing was certainly not sufficient to define the 100,000 hour strengths. The 2150°F treatment resulted in slightly higher strengths than did 2050°F. The tests on specimens aged for 5 hours at 1350°F prior to testing showed no improvement in strength and any improvemen in ductility was small.

The test data show considerably lower rupture properties than would have been expected on the basis of the results for Heat 02706, unless data scatter resulted in an unduly low extrapolated strength.

The creep curves for the rupture tests (Fig. 9) show that the creep resistance of the Heat 18439 specimens was considerably higher when heat treated at 2150°F than at 2050°F or 2150°F plus 5 hours at 1350°F. All tests had very little primary creep, extensive secondary creep with rather rapid increased in creep rate during the tertiary creep periods.

The influence of the three heat treatments used for Heat 18443 specimens on the creep resistance at 1350°F under 7000 psi was determined using tests of 12,541 to 14,666 hours in duration. The creep curves (Fig. 10) indicate that all three heat treatments initially had similar creep strength. The water quench from 2150°F, however, resulted in the creep resistance being retained to time periods considerably longer than the other two treatments. The creep rate apparently started to increase

at about 11,500 hours whereas the material aged at 1350°F for 5 hours started to increase in creep rate at 4500 hours. The specimen treated at 2050°F had only a very brief period, if any, of secondary creep before the rate started to increase with time. There was very little difference in the amount of creep for the three heat treatments to about 4500 hours. The increasing creep rates with time of testing for the materials treated at 2050°F and 2150°F plus 1350°F then caused a distinct separation from the curve for the specimens heat treated at 2150°F. It will be noted that the curves for the rupture tests (Fig. 9) also showed little difference in creep resistance until the increasing creep rates with time caused a separation between heat treatments.

The rupture tests suggested stresses of the order of 10,000 to 11,000 psi for rupture in 10,000 hours and 5400 to 5000 psi for 100,000 hours. The rupture times suggested by Figure 8 for 7000 psi are in the range of 40,000 to 60,000 hours. The creep curves of Figure 10 seem to be indicating increases in creep rate with time which could lead to rupture before 40,000 to 60,000 hours. This is, however, very uncertain. The rupture tests suggest that the periods of increasing creep rate could be very long and the rupture times under 7000 psi would be as long as suggested by Figure 8. If elongations did continue to decrease with time as suggested by some of the rupture tests, there would be even less chance of the rupture times being 40,000 to 60,000 hours.

The creep curves of Figure 10 show that for 7000 psi at 1350°F the times for total creep of 1 and 1.5 percent were as follows:

1	-percent creep (hours)	1.5-percent creep (hours)
W.Q. 2050°F	9,600	12,700
W.Q. 2150°F + 5 hours at 1350°	F 10,500	13,800 (estimated)
W.Q. 2150°F	13,600	*

<sup>\*</sup> Probably would be as long as 18,000 hours.

It is difficult to judge the rupture time and the creep strength of materials with such prolonged periods of increasing creep rate. The creep curves in any event indicate that the time for 1.0 or 1.5 percent creep should be small compared with the time for rupture. If it should be necessary to limit creep to the order of 1 percent, as is common design practice, the stress should be a rather small fraction of the rupture strength.

Tensile Properties after 12,541 to 14,666 Hours at 7000 psi and 1350°F

Room temperature tensile tests were run on the specimens after the creep tests at 1350°F and 7000 psi were discontinued, with the following results:

Duration of Test	Tensile Strength	Offset Yield (ps:	O	Elongation (% in 1.5	Reduction of
(hours)	(psi)	0.1%	0.2%	inches)	<u>Area (%)</u>
Water Quer	nched 2050°I	<u>-</u>			
As treated 14,499	81,000 72,800	31,000 39,500	34,000 44,600	52.5 4.5	70.0 8.0
Water Quer	nched 2150°I				
14,666	78,200	37,800	41,800	7.0	9.0
Water Quer	nched 2150°I	F plus Aged 5	hours at 13	350°F	
As treated 12,541	85,100 82,300	37,000 39,500	40,000 44,400	51.5 6.5	67.0 9.5

The specimens from the creep tests all had low ductility at room temperature as a result of a large reduction from creep exposure.

Yield strengths were increased while tensile strengths were reduced. While comparative data are not available for the unexposed W.Q. 2150°F material it unquestionably changed properties about as much as the other two treatments.

Hardness measurements were made (Table 7) on the heat treated material and on the specimens from the longest time creep and rupture tests. The hardness measurements on the rupture test specimens were

made in the gage section and on the creep test specimens in the threaded end away from the section cold worked in the tensile test. The precipitation occurring during testing for 1582 hours or more resulted in the hardness values of 150-166 Brinell. As heat treated the hardness was 119-130 BHN except for the material aged at 1350°F for 5 hours after a 2150°F W.Q. which was 135 BHN.

#### Metallographic Examination

Heat treatment at 2050°F caused recrystallization (compare Plates 23 and 24 with Plates 25 and 26). Most of the "dots" in the matrix of these two structures were probably etching pits. The grain boundaries seemed to be somewhat more distinct in Heat 18439 as well as somewhat larger. The main effect of heat treating at 2150°F (Plates 27 and 28) was some increase in grain size and less distinct grain boundaries compared with treatment at 2050°F. The dots in the background seemed to be a function of pitting rather than heat treatment.

Aging for 5 hours at 1350°F caused some precipitation in the grain boundaries (Plates 29 and 30). This was more extensive in Heat 18439 than 18443 - so much so that it is very surprising that the "heat-to-heat" differences in structure would be so marked. Reference to the rupture data did show a substantial difference in ductility in that Heat 18439 at least had higher ductility than Heat 18443.

All three heat treatments underwent extensive general precipitation (Plates 31, 32 and 33) during rupture testing at 1350°F. There were a few somewhat larger particles in the grain boundaries. Fractures were largely intercrystalline. The grain boundaries of the material aged for 5 hours at 1350°F were considerably more distinct than the solution treated specimens.

The specimens subjected to creep at 7000 psi for 12,541 to 14,666 hours (Plates 34, 35 and 36) had numerous rather large precipitate particles. The difference in the particles from those of the other tests was very striking. Insofaras these microstructures are concerned it is difficult

to see why treatment at 2150°F resulted in delaying third-stage creep. While the micros were taken near the shoulders of the specimens, out of the zone deformed by tensile testing at room temperature, they were typical of the gage section microstructure.

The rather large particles in the three creep specimens show a remarkable resemblance to sigma phase as it forms in fine grained Type 316 or 347 austenitic steel. They apparently formed from the same phases as the extensive fine precipitate shown by the rupture specimen (Plates 31, 32 and 33). This is not positive since the presence or absence of extensive creep could have influenced the precipitation reactions and the large particles could have formed directly in the creep specimens and might even be a different phase. If these particles were sigma rather than agglomerated carbides, they could suppress the etching of the fine precipitated carbides. A more extensive study would be needed to define the microstructures.

It was noted that the "carbide" phase in the eutectic structure shown previously for Heat 2649 was well broken-up and strung out in the longitudinal direction of the tubes. It is difficult to judge whether or not there had been more solution of this phase than in Heat 2649, although it did seem to be less, as might be expected as a result of the heating for piercing and working at the high temperatures used for this operation.

#### Boron Modified 17-14 Cu + Mo - Heat 02708

Stress-rupture tests were run out to 1662 hours at 1350°F (Table 8) on the boron modified 17-14 Cu+Mo alloy as water quenched from 2150°F. The stress-rupture time curves (Fig. 11 and Table 2) indicate that there was little if any difference in rupture properties at 1350°F between the boron modified heat and a similar heat without boron (Heat 02706) when water quenched from 2150°F. The ductility of the heat with boron was somewhat lower than for Heat 02706. Both had high rupture strength and ductility compared with the other examples of the alloy tested.

The original structure and the structure after testing at 1350°F are shown in Plates 37 and 38. The original structure is quite typical for the

alloy in the condition tested. The precipitation occurring during exposure in the test was similar to but not quite as extensive as was observed in Heat 02706.

The chemical analyses in Table 1 indicate that the actual boron content was only 0.0005 percent. This amount is less than is generally considered necessary to produce a noticeable effect. It is therefore not surprising that there was so little difference between Heats 02708 and 02706. The most significant result is the duplication of apparently high long time strengths and ductility for the two "Zero" heats compared with the other heats investigated. These zero heats show considerably less general precipitation than the other heats after rupture testing.

#### DISCUSSION

The data have been discussed and correlated as they were presented for each heat.

There seemed to be unduly wide variations from heat to heat (Table 2 and Fig. 12). This could be evidence of sensitivity to prior history or to unidentified heat-to-heat variations.

Presumably the strength of experimental Heat A218 would have come up to expected levels if the temperature of heat treatment had been raised from 2000° to 2150°F.

The centrifugally cast and rotorolled tubing from Heat 2649 had quite good properties when heat treated at 1850°F. There is a strong probability that this was due to the cast structure being cold worked with a rather high intermediate anneal, i.e., not much chance for the high creep strengths usually observed in castings to be destroyed by precipitation and agglomeration of key compounds. The loss in ductility with increasing temperature of heat treatment and testing time suggested that either the unusual prior history or the omission of Ti from the composition resulted in low ductility.

The tubing made from the small arc furnace heats, Heats 02706 and 02708, had high strengths when heat treated at 2150°F. Reduced ductility from treatment at 2250°F was associated with lowering of the strength to very low values at long time periods at 1350°F. The low Ti and Cb in Heat 02708 did not seem to have reduced the strength appreciably. The 0.0005 percent analyzed boron would not be expected to improve properties as the data seem to indicate.

These high strengths were not reproduced in the production tubing from Heats 18443 and 18439. The reasons for the difference were not found except that for some reason the production tubing may have been specially prone to form sigma phase. This seems, in view of the microstructure, to be a possible explanation for the creep rates increasing with time after short times of testing. There was no obvious reason why heat treatment at 2150°F delayed the onset of third-stage creep at 1350°F.

Only the centrifugally cast and rotorolled tubing from Heat 2649 were tested at 1500°F. This is also true for 1200°F except for Heat A218. The indicated long time strengths did not show much variation with heat treatment. The values were on the high side of those for Type 316 and 347 austenitic steels but below those presumed to be typical of the alloy. Either the conditions of making the tubes or the omission of Ti from the analyses may have been responsible. Low ductility probably limited long time strength.

The data show that tubing had higher long time strengths than the standard austenitic steels. The strengths were however below those considered characteristic for the alloy except for the tubes made from the small "zero" arc furnace heats. This was not reproduced in the production tubes although their strengths were probably higher than Types 316H and 347H austenitic steels. Strengths seemed to be limited by the early onset of third-stage creep at 1350°F and by low ductility. Creep tests of 12 000-14 000 hours on production tubing showed no indication

of a decrease in creep rate - the rates steadily increased with time, except that heat treatment at 2150°F seemed to delay the third-stage creep.

Table 1

Chemical Composition (Weight Percent) for Six Heats of 17-14 Cu+Mo Alloy

Cu Ti Cb	3.0627 .50	3.08 (.51Cb+Ta)	2.90 .19 .45	3.20 .31 .62	3.26 .20 .52	. 14	
Mo	2,55	2.44	2.16	2.25	2.25	2.10	
Ni I	14.46	14.34	15.12	14.32	14.25	14.96	
Cr	15.99	15.19	15.63	16.33	16.05	15.35	
Si	. 67	. 50	. 22	. 64	99.	. 25	
ω	. 020	. 018	.012	.014	.012	. 011	
다	. 013	. 014	. 019	. 021	. 024	. 013	
$\overline{\mathrm{Mn}}$	. 78	. 74	. 72	. 84	. 88	92.	
이	.12	. 15	. 11	. 11	. 11	.117	
Heat No.	A218	2649	02706	18443	18439	02708*	

\* Plus 0.0005% Boron

Table 2

Rupture Properties at 1200°, 1350° and 1500°F for 17-14 Cu+Mo Alloy

racture 100,000 hr.	(14, 500) (17, 500) (22, 000) (14, 000) 11, 600 12, 250	(5,200) (6,000) (7,700) (5,800) (9,200) (3,500)	(5,400) (6,000) (8,400) (9.0)	4,600 4,500
Rupture Strength (psi) and Elongation (%) at Fracture         1r.       1000 hr.       10,000 hr.       100,000	(21, 500) (21, 000) (26, 500) (22, 000) (29-32)	(8,700) (8,600) (11,000) 10,500(5) - 13,500(17) (9,600)(3.5)	(10,000) (11,000) (13,000) (11,2-16.5)	
e Strength (psi) and 1000 hr.	32, 000 (15) 25, 000 (12) 32, 000 (18) 34, 000 (15) (34-37)	14, 500 (7) (12, 000) 16, 000 (12) 19, 000 (15) (~20, 000)(1.5) 20, 000 (21) 24, 500 (3.5)	7, 500)(14 8, 000)(11 7, 500)(16 0, 000 (19	
Ruptur. 100 hr.	41,000 (12) 30,000 (18) 39,000 (25) 42,000 (25) (40-43)	24, 000 (16) 18, 000 (14) 22, 500 (30) 22, 500 (20) - 29, 000 (35)	(22, 500) (22, 000) (22, 500) 31, 000 (22-26)	
BHN	152 164 159 159	152 164 159 159 136 143	121/130 119/122 134/136 134	
Heat Treatment	118 Barstock OQ 2000°F 49 (a) Tube AC 1750°F 49 (a) Tube WQ 1850°F 49 (a) Tube WQ 1950°F STP 124 Range in ksi 316H Average 347H Average	Barstock OQ 2000°F Tube AC 1750°F Tube WQ 1950°F Tube AC 1750°+ WQ 2250°F Tube WQ 2150°F Tube WQ 2250°F	Tube WQ Tube WQ Tube WQ 5 hr. Age WQ	rerage rerage
Heat No.	A218 Barst 2649 (a) T 2649 (a) T 2649 (a) T STP 124 Rang 316H Average 347H Average	A218 2649 (a) 2649 (a) 2649 (a) 2649 (a) 02706 02706	(b) (b) (b) 02708 (c) STP 124	316H Average 347H Average

continued ....

Table 2 concluded

Rupture Strength (psi) and Elongation (%) at Fracture	Heat Treatment BHN 100 hr. 1000 hr. 10,000 hr. 100,000 hr.	1500° F		159 11,500 (18) 7,200 (10) (	159 12,500 (20) 8,400 (7) (	4C 1750°+	NQ 2250°F 136 - (~10,000)	250°F 136 - (~10,000) - (11.5-16.5) (7.2-12.8) (4.4	250°F 136 - (~10,000) - (4.4-7.6)
	•				Tube WQ 1950°F 159	Tube AC 1750°+		WQ 2250°F in ksi	WQ 2250°F in ksi
	Heat No.		2649 (a)	2649 (a)	2649 (a)	2649 (a)		STP 124 R	STP 124 Range 316H Average

(a) Centrifugally cast and rotorolled tubing - no Ti in heat

(b) Very approximate values based on combined data from Heats 18443 and 18439

(c) Boron added - low Ti and Cb

Table 3

Stress-Rupture Data at 1200° and 1350°F for 17-14 Cu
+Mo Alloy Barstock from Heat A218

Heat Treatment	Temp.	Stress (psi)	Rupture Time (hours)	Elongation (% in 2 ins)	Reduction of Area (%)
2000°F, O.Q.	1200	42,000 37,000 34,000	85 398 672	15.0(a) 12.5(a) 15.0	21.0 19.0 19.5
	1350	27,000 24,000 19,000 15,000	29.2 106 419 921	21.5 15.5 11.0 7.0(a)	36.0 25.0 18.5 13.5

<sup>(</sup>a) Broke in gage mark

Table 4

Stress-Rupture Data at 1200°, 1350° and 1500°F for a 17-14 Cu +Mo Alloy Centrifugally Cast and Rotorolled Tube from Heat 2649

	m.	Q.	<b>D</b>	771	
II.a. Turaturat	Temp.	Stress	Rupture	Elongation	Reduction
Heat Treatment	(°F)	(psi)	Time (hrs)	(% in 1 in.)	of Area (%)
17509F A C	1200	58,000	STTT	43.0	51.0
1750°F, A.C.	1200	35,000	18.1	23.0	25.5
		27,000	459	12.0	17.0
				40.0	45.0
	1350	38,700	STTT	49.0 22.0	22.5
		22,000	26.3	13.0	17.0
		17,000	135	15.0	11.0
	1500	25,300	STTT	47.0	40.5
	1300	13,000	18.1	25.0	28.0
		9,000	132	21.0	22.0
				2/ 0	25.5
1850°F, W.Q.	1200	40,000	75	26.0	22.0
		33,000	836	18.0(a)	22.0
	1350	24,000	66	31.0	30.0
	1550	14,000	2175	12.0	16.0
					30 5
	1500	13,000	54	18.0	29.5
		8,000	662	12.0	18.0
1950°F, W.Q.	1200	40,000	187	24.0(a)	22.0
2,50 2,		33,000	1456	14.0(a)	18.5
		28,000	3195	10.5	24.0
	1250	24 000	54	21.0	27.5
	1350	24,000 20,000	499	16.0	21.0
		16,000	2138	11.0	14.5
		14,000	3747	4.0	9.5
		10,000	12594	5.0	7.5
		10,000	,_		
	1500	13,000	90	21.0(a)	27.5
		10,000	359	10.5	19.5
		8,000	1366	6.0	(b)
		6,000	Discon	tinued after 2	682 hours
1750°F A.C. +	1350	21,000	691	1.5	1.5
2250°F W.Q.			4.6.5	35.5	20.5
	1500	11,500	683	15.5	<b>40.</b> J

<sup>(</sup>a) Broke in gage mark

<sup>(</sup>b) Broke in fillet

Table 5

Stress-Rupture Data at 1350°F for 17-14 Cu+Mo Alloy Pierced
Tube from Heat 02706

Heat Treatment	Temp.	Stress (psi)	Rupture <u>Time (hrs</u> )	Elongation $(\frac{\% \text{ in } 1^{\frac{1}{2}} \text{ins}}{})$	Reduction of Area (%)
2150°F, W.Q.	1350	25,000 20,000 17,000 15,000	200 1008 3007 5763	33.0 21.0 15.5 17.0	56.0 49.0 27.5 22.5
2250°F, W.Q.	1350	25,000 20,000 17,000 12,500	841 1983 2913 5751	3.5 4.5 3.5 3.5	6.0, 6.0 5.5 5.0

Table 6

Stress-Rupture Data at 1350°F for 17-14 Cu+Mo Alloy Pierced
Tubes from Heats 18443 and 18439

	Temp.	Stress	Rupture	Elongation	Reduction
Heat Treatment	(°F)	(psi) T	Time (hrs)	$(\frac{\% \text{ in } 1^{\frac{1}{2}} \text{ins}})$	of Area (%)
2050°F, W.Q.	1350	20,000(a)	324	5 <b>.</b> 5	15.5
2030 F, W. Z.	1000	17,000	1363	14.0	26.5
		15,000	2359	8.5	20.0
2150°F, W.Q.	1350	20,000(a)	253	6.5	14.5
2130 1, 11.2.		17,000	2236	10.5	22.5
		15,000	3356	10.0	19.5
2150°F, W.Q.+	1350	20,000(a)	345	8.5	13.5
Age 5 hrs. at	1330	17,000	1582	16.0	39.0
1350°F					

<sup>(</sup>a) Tests on specimens from Heat 18443 - all others from Heat 18439

Table 7

Hardness Data before and after testing of 17-14 Cu+Mo Alloy
Pierced Tubes from Heats 18443
and 18439

		Test Condition			
Heat No.	Treatment	Stress (psi)	Test Duration (hrs)	BHN	
18443	W.Q.2050°F	As heat treated	. <del>-</del>	121	
18439	W.Q.2050°F	As heat treated	. <del>-</del>	130	
18443	W.Q.2050°F	7000 *	14, 499	152(a)	
18439	W.Q.2050°F	15000 *	2,359(R)	154	
18443	W.Q.2150°F	As heat treated	<del>-</del>	119	
18439	W.Q.2150°F	As heat treated	_	122	
18443	W.Q.2150°F	7000 *	14,666	166(a)	
18439	W.Q.2150°F	15000 *	3,356(R)	151	
18443	W.Q.2.150°F + Age	As heat treated	-	134	
18439	W.Q.2150°F + Age	As heat treated	-	136	
18443	W.Q.2150°F + Age	7000 *	12,541	155(a)	
18439	W.Q.2150°F + Age	17000 *	1,582(R)	150	

<sup>(</sup>a) End of specimen away from section cold worked in tensile testing

Table 8

Stress-Rupture Data at 1350°F for 17-14 Cu+Mo Alloy (0.0005%B, 0.14%Ti and 0.20%Cb - Heat 02708)

Heat Treatment	Temp.	Stress (psi)	Rupture Time (hours)	Elongation $(\frac{\% \text{ in } l^{\frac{1}{2}} \text{ins})}$	Reduction of Area (%)
2150°F, W.Q.	1350	25,000	287	24.5	48.5
		21,000	722	19.5	45.5
		18,000	1662	12.0	34.0

<sup>(</sup>R) Rupture specimen

<sup>\* -</sup> Tested at 1350°F



























