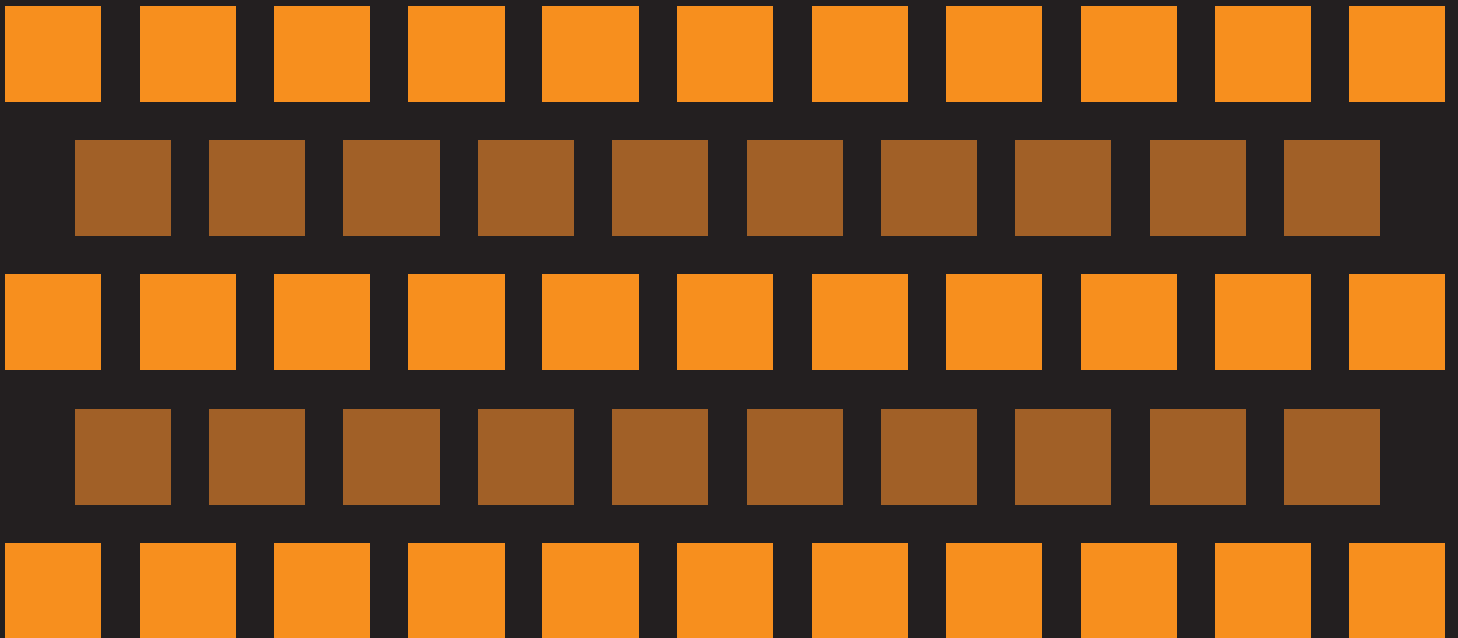


CORRECTIONS TO STAINLESS STEEL ALLOWABLE STRESSES



STP-NU-059

CORRECTIONS TO STAINLESS STEEL ALLOWABLE STRESSES

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FOREWORD

This document is the result of work resulting from Cooperative Agreement DE-NE0000288 between the U.S. Department of Energy (DOE) and ASME Standards Technology, LLC (ASME ST-LLC) for the Generation IV (Gen IV) Reactor Materials Project. The objective of the project is to provide technical information necessary to update and expand appropriate ASME materials, construction and design codes for application in future Gen IV nuclear reactor systems that operate at elevated temperatures. The scope of work is divided into specific areas that are tied to the Generation IV Reactors Integrated Materials Technology Program Plan. This report is the result of work performed under Task 14 titled “Corrections to Stainless Steel Allowable Stresses.”

ASME ST-LLC has introduced the results of the project into the American Society of Mechanical Engineers (ASME) volunteer standards committees developing new code rules for Generation IV nuclear reactors. The project deliverables are expected to become vital references for the committees and serve as important technical bases for new rules. These new rules will be developed under ASME’s voluntary consensus process, which requires balance of interest, openness, consensus and due process. Through the course of the project, ASME ST-LLC has involved key stakeholders from industry and government to help ensure that the technical direction of the research supports the anticipated codes and standards needs. This directed approach and early stakeholder involvement is expected to result in consensus building that will ultimately expedite the standards development process as well as commercialization of the technology.

ASME has been involved in nuclear codes and standards since 1956. The Society created Section III of the Boiler and Pressure Vessel Code, which addresses nuclear reactor technology, in 1963. ASME Standards promote safety, reliability and component interchangeability in mechanical systems.

Established in 1880, ASME is a professional not-for-profit organization with more than 127,000 members promoting the art, science and practice of mechanical and multidisciplinary engineering and allied sciences. ASME develops codes and standards that enhance public safety, and provides lifelong learning and technical exchange opportunities benefiting the engineering and technology community. Visit www.asme.org for more information.

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EXECUTIVE SUMMARY

The primary controlling variables for predictable 105 hour creep rupture properties in Type 304H and 316H stainless steel at elevated temperatures have been identified as nitrogen in interstitial solid solution (available nitrogen) and copper above 0.25 wt% [1]. An expression was developed to account for the varying residual strong nitride forming elements in a heat, where targeted nitrogen additions can be made to ensure sufficient available nitrogen levels without complicating the material certification process. Appendix A shows the current 2010 Subsection NH, Appendix X, Table X-1 restrictions for service up to 1100°F (595°C), with Appendix B presenting the new proposed restrictions in Table X-2 for long-term service at temperatures between 1100°F (595°C) and 1337°F (725°C). It is recommended that these proposed Table X-2 restrictions be mandatory for long term service at these temperatures to take advantage of the demonstrated creep performance improvements associated with these restrictions.

A review of 340 recent Type 316 SS heat compositions identified residual titanium, aluminum, boron, niobium and vanadium at sufficient levels to impact the amount of available nitrogen necessary for optimum creep properties. These elements were targeted for restrictions in the proposed Table X-2 nitrogen calculation due to their presence as residuals in steel, their low free energy of nitride formation [2] and the stability of these nitrides in steels at the anticipated service temperatures. Zirconium and tantalum were excluded from the calculation because they are typically present only at trace levels; however, these elements will be reported for future use and control if necessary. It is anticipated that the reporting of zirconium and tantalum could be eliminated if these elements continue to be at trace or less than minimum detection levels (MDL). It is proposed that the copper control be accomplished using a minimum–maximum composition range, with a nominal composition typically found in production heats. This approach of determining the residual nitride forming elements, and adjusting the nitrogen on a heat basis, has been reviewed by a major stainless steel producer and it has concurred that the proposed Table X-2 restrictions are acceptable for production quantities, and would not compromise the material certification process.

This study examined 105 hour creep rupture data from the Japanese National Institute for Materials Science (NIMS), and consisted of nine Type 304HTB and nine Type 316HTB heats that exhibited considerable scatter in creep results. The compositions of the NIMS heats indicate that they were likely produced to requirements similar to the conventional Type 304H (UNS# S30409) and Type 316H (UNS# S31609) requirements. Given that several of the NIMS heats did not meet the current Subsection NH, Appendix X, Table X-1 composition restrictions, they nonetheless exhibited significantly reduced scatter in the creep results, and, in fact, were the best performers. These data indicate that a review of the applicability of the current Table X-1 restrictions is warranted given the strong correlations established between available nitrogen and creep properties. The proposed Table X-2 is an attempt to further reduce scatter in creep data by using targeted restrictions mostly for copper and nitride forming species, as presented herein, to modify the conventional UNS alloy composition requirements for Type 304H and 316H SS. A potential economic benefit may be realized by re-establishing the conventional H grade composition ranges for non-restricted species, by providing suppliers some leeway to achieve the desired creep rupture properties.

After eliminating the Type 316H heats in the NIMS database that did not satisfy the proposed Table X-2 restrictions, and then extrapolating the remaining compliant heat creep results to 1337°F (725°C), the data suggests that these compliant heats represent a minimum creep rupture strength of approximately 2553 psi (17.6 MPa) vs. the Section III, Division 1, Subsection NH allowable of 2321 psi (16 MPa) at 1337°F (725°C). Given that only three relevant data points from the NIMS study satisfy the proposed Table X-2 restrictions, additional confidence in the 1337°F (725°C) upper temperature limit could be realized by including additional 105 hour creep rupture data from other sources. An additional benefit of evaluating additional Table X-2 compliant heat creep data may

allow a more definitive upper service temperature limit that the NIMS data suggests may, in fact, be slightly above 1337°F (725°C).

Regardless of whether the proposed Table X-2 is adopted or not, it is recommended that additional available 105 hour plus creep rupture data be screened for compliance to the proposed Table X-2 restrictions, and the minimum stress to rupture be recalculated for comparison to the current Section III, Division I, Subsection NH allowables. This approach is expected to allow a more accurate determination of the acceptable upper service temperature limits for Type 304H and 316H materials, and improve the confidence for designers of high temperature components.

Additional recommendations include considering if ongoing creep testing organizations should begin recording ferrite number data for Type 304H and 316H creep samples and begin to consider the effects of weld ferrite content on creep performance.

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1 OBJECTIVE

ASME Standards Technology, LLC has sponsored a thorough review of current allowable stress values in ASME Boiler and Pressure Vessel Code, Section III, Division I, Subsection NH, and to identify inconsistencies and potential limitations on the use of some current values in Subsection NH for austenitic stainless steel. More specifically, long term creep tests on AISI Type 304H and 316H stainless steel (SS), completed after the current allowable stress values were established, identified some heats whose rupture life fell below currently published allowable values, particularly above 1200°F (650°C). Since some of these errors and limitations could impact near term design activities for Gen IV applications, there is an urgent need to address this issue.

1.1 Technical Approach

The technical approach to address this objective was to break the effort into three distinct parts where at the conclusion of Part 1, it would be evident as to whether the Part 2 stress modifications would be necessary. The scope of work for each part is identified as follows:

Part 1 – Assess Available Data

Assess available data on Type 304H and 316H SS (Note: 316H SS is the primary material of interest in this assessment) to determine if there are restrictions that could be placed on specifications and procurement packages or additional acceptance test and examination requirements, e.g., chemical composition, physical or mechanical properties or processing variables, that would exclude from use those heats of material that are not representative of the database from which the currently published allowable stress values were derived.

Part 2 – Determine Time and Temperature Limits

In the event that the results of the Part 1 assessment do not identify applicable restrictions and/or additional acceptance requirements, the time and temperature limits beyond which the validity of the current allowable stress values cannot be guaranteed shall be determined. Allowable stresses beyond their range of validity should be recommended for deletion.

Part 3 – Draft Code Rules

Based on the results obtained after completion of Parts 1 and 2, above, prepare a submittal of draft code rules with supporting information. Recommendations should include specific Code words or modifications to tables implementing the restrictions that are ready for consideration as a Code Case or Code revision. This submittal shall be a formal proposed standard action and shall have an assigned tracking number.

2 CURRENT RESTRICTIONS

Appendix A of this report shows the current 2010 non-mandatory Subsection NH Appendix X, and includes the Table X-1 restrictions recommended for Type 304H and 316H SS in elevated temperature service up to 1100°F (595°C). The basis for many of these existing restrictions were documented in Reference [6], and were incorporated in an effort to reduce the scatter in creep rupture data while promoting some ferrite formation for fabricability. The current Table X-1 restrictions appear to have been formulated from a variety of foreign and domestic creep data, with the intention of narrowing select composition ranges shown to reduce the heat-to-heat scatter in creep performance. There was some attention given to nitrogen, niobium, boron and aluminum restrictions indicating empirical evidence at the time that these elements were involved in creep performance; however, they could not have quantified their significance without the data provided herein. With the strong correlation identified between available nitrogen – creep performance in hand, the applicability for the current Table X-1 restrictions should be reviewed for effectiveness.

Several of the current Table X-1 restrictions bias the composition ranges away from austenite stability, promoting ferrite formation. The microstructural stability of Type 316 SS has been the subject of much investigation related to balancing the composition to promote austenite stability while maintaining some ferrite for fabricability. Given the extremely long service times at elevated temperatures, compositional effects related to ferrite formation cannot be ignored. The current Table X-1 restriction for nickel, a strong austenite former, was biased low in the conventional range (11.0 to 12.5 wt% as compared to 11.0 to 14.0 wt% for standard Type 316) and would be expected to slightly increase the likelihood of ferrite formation. The current restrictions for chromium and molybdenum, both ferrite formers, are biasing these elements toward the high end of the conventional ranges, and again would be expected to promote the formation of additional ferrite. To provide long term phase stability, it is believed the optimum approach would be to specify a ferrite requirement and provide suppliers the leeway to utilize the full UNS specifications alloy composition ranges to achieve the desired ferrite contents. These concerns over phase stability also apply to Type 304H.

3 RESULTS

3.1 Part 1 - Assess Available Data

To gather sufficient information to choose between a restriction on specifications, or limitation of ASME Boiler and Pressure Vessel Code, Section III, Division I, Subsection NH coverage, long-term creep results for Type 304HTB and 316HTB SS heats were obtained from ASME ST-LLC STP-NU-037 (Task 6) [3] and NIMS Creep Data Sheets. Unfortunately, the Task 6 data did not include sufficient heat compositional data, and could not be included in this study. The effect of chemical composition on creep results were evaluated for each heat in the NIMS database. A total of nine Type 304H heats and nine Type 316H heats were studied, with Table 1 showing the chemical compositions for each NIMS heat.

Evaluations performed on the long-time test data from NIMS consisted of a detailed compositional analysis relative to the reported creep results for each available heat. Initial review of this data indicated few of the NIMS heats met the current Table X-1 requirements. Many of the Type 304H heats exhibited higher carbon, molybdenum and nickel, with about half having lower chromium and a few exhibiting high niobium. Interestingly, none of the Type 304H heats met the current Table X-1 values for nitrogen, with all heats lower than the identified range. Many of the Type 316H heats exhibited higher carbon and low chromium, while all were low in molybdenum, nickel and nitrogen. It was concluded that the NIMS Type 304H and 316H heats were produced using compositions similar to standard H grade alloying.

Figure 1 shows the 105 hour creep rupture strengths determined for the Type 304H and 316H NIMS heats. Figure 2 shows the time to rupture data for Type 304H and 316H NIMS heats at 1292°F (700°C) where the time to rupture shows significant variability. The heat-to-heat variation in time to rupture becomes more significant at low stresses and long times, as shown by more than one order of magnitude difference between the best and worst performing heats. Compositional comparisons between the best and worst performing heats identified that the worst performing heats exhibited high levels of the known nitride formers aluminum and/or titanium. Mass balance calculations identified the worst performing heats exhibited sufficient aluminum and titanium to account for all the nitrogen present, and that the best performing heats exhibited low aluminum and/or titanium levels where there was excess nitrogen believed to be present as an interstitial solid solution species. This compositional analysis identified a strong correlation between the creep performance and the amount of nitrogen in solution (available nitrogen, NAV) for these NIMS heats. Plots comparing the Type 316H aluminum and nitrogen concentrations showed a large variation in the aluminum concentration of the study heats (see Figure 3). Further scrutiny of the data also identified a correlation with the copper level, as seen Figure 4. Figure 5 shows that the available nitrogen concentration and impurity copper were controlling variables responsible for the heat-to-heat variation in creep performance for Type 316H.

Transmission electron microscopy (TEM) studies on Type 304H and 316H heats to characterize precipitates present after extended exposures at 1292°F (700°C) and 1472°F (800°C) identified aluminum nitride (AlN) precipitates associated with sigma (σ) and Chi (χ) phases at the grain boundaries (see Figure 6). Sigma is a potent embrittling phase that forms in austenitic SS primarily due to long-term elevated-temperature exposure of delta ferrite. Chi also forms in austenitic SS, however, it typically forms at grain boundaries from austenite and has not been implicated as a significant embrittling phase. The presence of these AlN precipitates after exposure at these temperatures confirms the AlN precipitates are stable up to 1472°F (800°C), and coarsened during long-term exposure at these temperatures, ultimately reducing the available nitrogen to the levels in equilibrium with AlN.

Figure 7 and Figure 8 show creep rupture data and TEM micrographs where the available nitrogen and impurity niobium explain the heat-to-heat variability in creep life of Type 304H SS. At short exposure times, the presence of fine niobium carbides initially improves the creep strength, but the benefit disappears after extended exposure times. Niobium carbides (NbC) are seen to form initially as very fine precipitates at exposure times up to approximately 6000 hours, while after approximately 75,000 hours exposure, the precipitates are seen to coarsen significantly as they lose their ability to provide a precipitation strengthening effect. Figure 9 shows an illustration explaining that the observed heat-to-heat variability in creep performance of Type 304H at short exposure times is dominated by the NbC precipitates that coarsen and become ineffective for precipitation strengthening after long-term exposure. While these data suggest an explanation for the temporary effect of niobium on creep properties, the longer term effect of niobium is believed to be on the available nitrogen levels because these precipitates are known to also contain varying amounts of nitrogen. The difficulties in accurately determining the nitrogen content of these MC type precipitates using TEM techniques are well understood, and these precipitates are believed to contain at least some amount of nitrogen.

Table 1 - Chemical Composition of NIMS Heats Studies

Chemical compositions of 9 heats of 304HTB and 9 heats of 316HTB steels examined in NIMS Creep Data Sheets

JIS SUS 304HTB (18Cr-8Ni), NIMS Creep Data Sheet No.4B (1986)

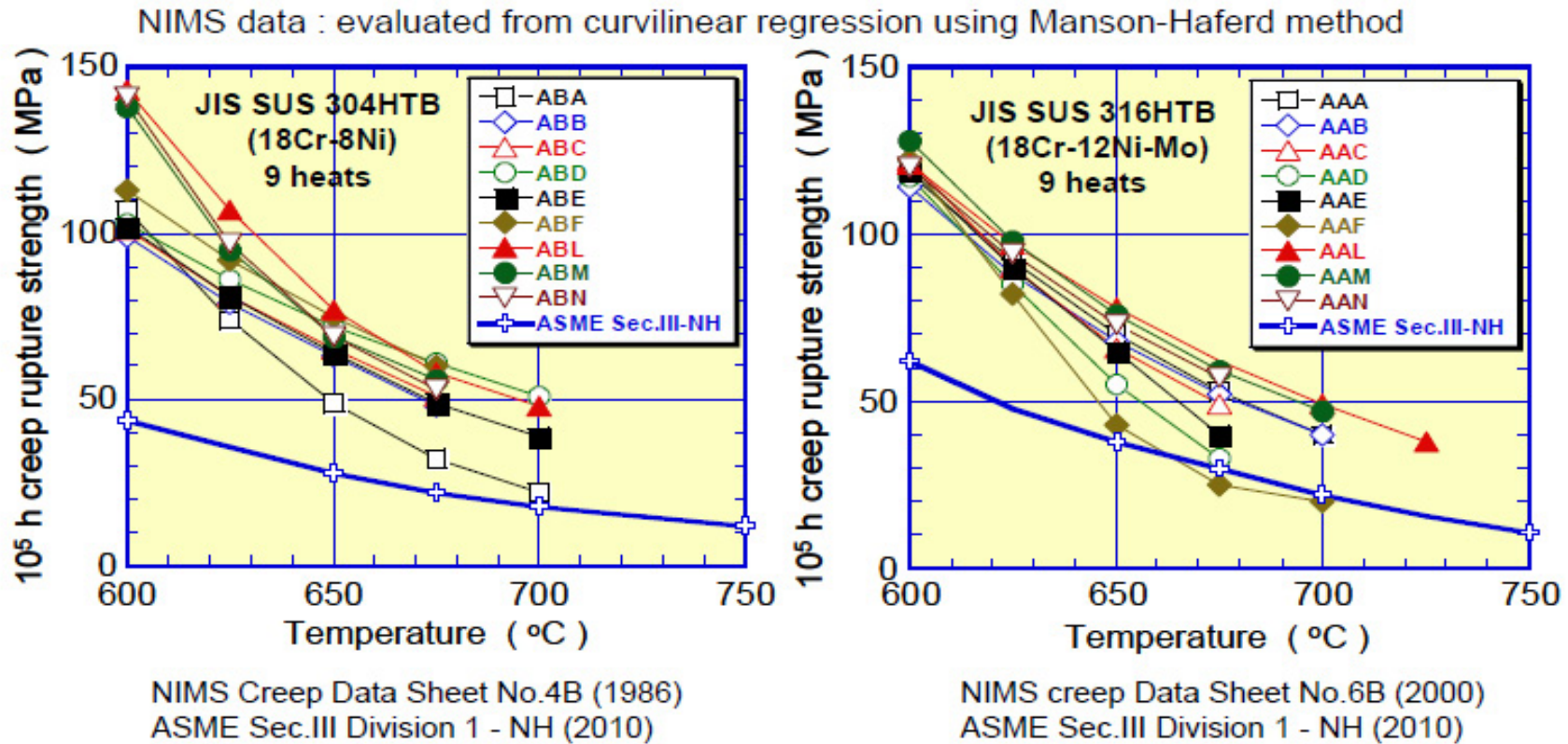
	C	Si	Mn	P	S	Ni	Cr	Mo	Ti	Al (mass%)		B	N	Nb + Ta	N _{AV} (at %)	N _{AV} (wt %)
										col.	incol.					
Requirement	0.04 ~ 0.1	≤ 0.75	≤ 2.00	≤ 0.040	≤ 0.030	8.00 ~ 11.00	18.00 ~ 20.00									
ABA	0.082	0.82	1.58	0.025	0.013	10.89	18.70	0.47	0.040	0.040	0.007	0.0007	0.0310	0.01	-0.008	-0.001
ABB	0.054	0.41	1.52	0.031	0.020	10.15	18.34	0.38	0.084	0.005	0.005	0.0010	0.0280	0.01	0.031	0.008
ABC	0.058	0.40	1.52	0.030	0.018	10.10	18.50	0.38	0.054	0.008	0.008	0.0011	0.0300	0.01	0.044	0.011
ABD	0.070	0.62	1.48	0.023	0.008	9.88	18.70	0.08	0.008	0.004	0.008	0.0013	0.0270	0.01	0.082	0.023
ABE	0.070	0.55	1.48	0.023	0.008	9.57	18.85	0.04	0.082	0.008	0.008	0.0018	0.0278	0.01	0.022	0.008
ABF	0.080	0.82	1.34	0.021	0.008	9.80	18.25	0.08	0.020	0.008	0.005	0.0017	0.0348	0.01	0.088	0.026
ABL	0.070	0.68	1.47	0.022	0.013	9.80	18.18	0.05	0.031	0.008	0.007	0.0003	0.0310	0.04	0.070	0.018
ABM	0.080	0.81	1.58	0.022	0.014	10.27	18.24	0.32	0.038	0.008	0.008	0.0001	0.0380	0.03	0.082	0.023
ABN	0.070	0.80	1.58	0.022	0.012	10.28	18.18	0.31	0.040	0.008	0.008	0.0008	0.0280	0.04	0.043	0.012

JIS SUS 316HTB (18Cr-12Ni-Mo), NIMS Creep Data Sheet No.6B (2000)

SIS 305 STAINLESS (100Cr-12Ni-Mo), NIMS Creep Data Sheet No.02 (2006)																
	C	Si	Mn	P	S	Ni	Cr	Mo	Cu	Ti	col. Al	B	N	Nb + Ta	N _{AV} (at %)	N _{AV} (wt %)
requirement*	0.04 - 0.10	< 0.75	< 2.00	< 0.040	< 0.030	11.00 - 14.00	18.00 - 18.00	2.00 - 3.00								
AAA	0.080	0.69	1.89	0.024	0.017	13.32	18.73	2.38	0.07	0.011	0.012	0.0010	0.0300	0.02	0.0815	0.02058
AAB	0.050	0.62	1.51	0.021	0.010	13.21	18.42	2.34	0.14	0.011	0.013	0.0005	0.0340	0.01	0.0854	0.02404
AAC	0.050	0.71	1.52	0.022	0.013	13.50	17.50	2.28	0.17	0.055	0.023	0.0013	0.0350	0.02	0.0278	0.007
AAD	0.058	0.68	1.58	0.027	0.008	13.85	18.88	2.14	0.17	0.028	0.037	0.0009	0.0250	0.01	-0.0085	-0.0024
AAE	0.078	0.82	1.80	0.021	0.008	13.51	18.90	2.08	0.18	0.037	0.027	0.0010	0.0280	0.01	0.0125	0.00318
AAF	0.088	0.83	1.73	0.029	0.024	13.18	17.07	2.34	0.13	0.055	0.082	0.0020	0.0280	0.01	-0.1417	-0.0368
AAL	0.070	0.81	1.85	0.025	0.007	13.80	18.80	2.33	0.28	0.043	0.008	0.0011	0.0250	0.01	0.0307	0.00777
AAM	0.080	0.62	1.80	0.025	0.007	13.30	18.70	2.25	0.24	0.060	0.012	0.0008	0.0318	0.01	0.0318	0.00806
AAN	0.080	0.62	1.58	0.025	0.007	13.80	18.80	2.31	0.28	0.028	0.011	0.0007	0.0224	0.01	0.0328	0.00822

$$N_{AV} = N - Al - Ti - Zr \text{ (at \%)}$$

$$N_{AV} = N - 0.519 Al - 0.292 Ti - 0.154 Zr \text{ (wt \%)}$$

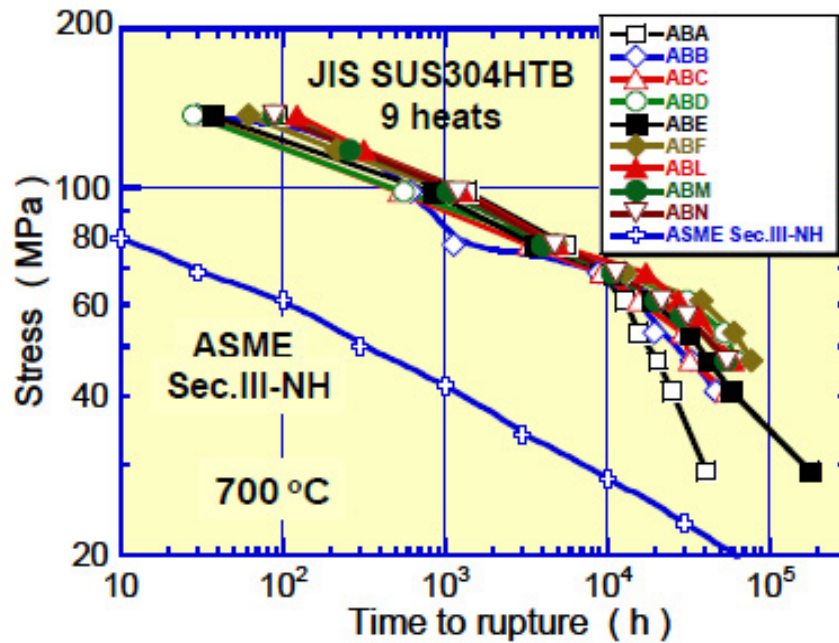


10^5 h creep rupture strength of individual heats of 304HTB and 316HTB steels

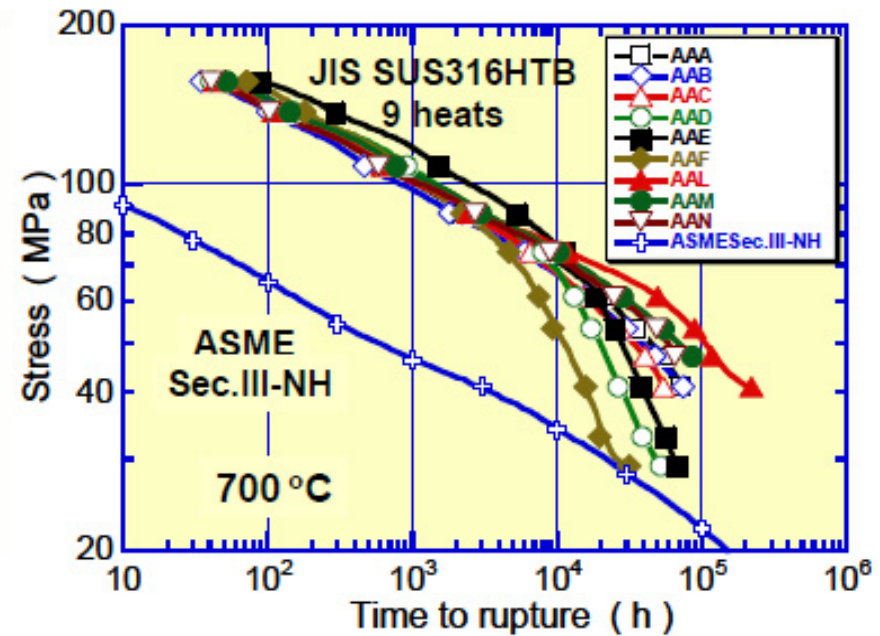
The 10^5 h creep rupture strength of the heat AAF of 316HTB steel is lower than that of ASME Sec.III Division 1 - NH at 675 and 700 °C.

At long times above 1×10^5 h, such as 3×10^5 h and more, it is suggested that several heats including the heat AAF exhibit lower strength than the Sec.III Division 1 - NH.

Figure 1 - 10^5 Hour Creep Rupture Results for the Type 304H and 316H NIMS heats



NIMS Creep Data Sheet No.4B (1986)
ASME Sec.III Division 1 - NH (2010)



NIMS creep Data Sheet No.6B (2000)
ASME Sec.III Division 1 - NH (2010)

**Heat-to-heat variation in creep rupture data
for JIS SUS 304HTB and 316HTB steels at 700 °C**

The heat-to-heat variation in time to rupture becomes more significant at low stresses and long times, as shown by more than one order of magnitude difference between the strongest and weakest heats.

Figure 2 - Variations in Creep Rupture Strength for the Type 304H and 316H NIMS heats at 1292°F (700°C)

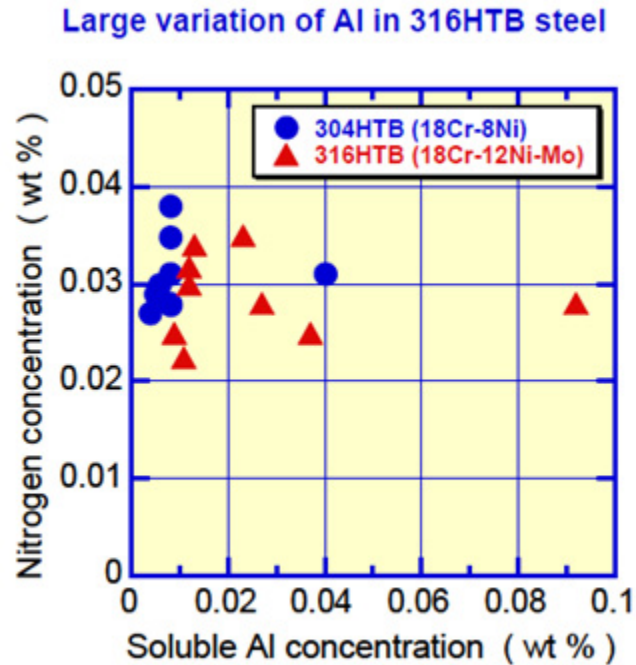


Figure 3 - Plot of Aluminum vs. Nitrogen Shows a Large Variation in the Aluminum for the Type 316H NIMS Heats

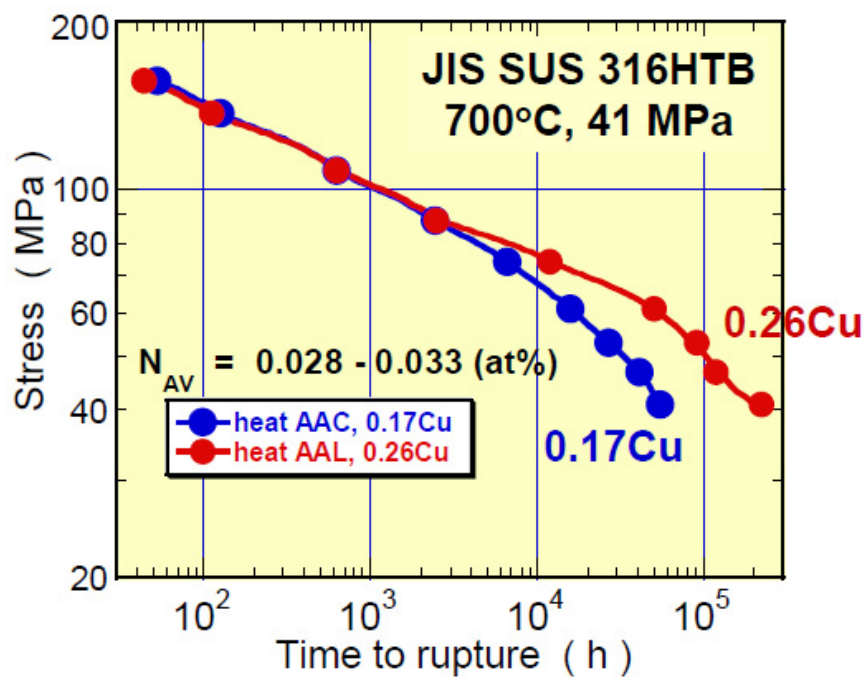


Figure 4 - The Effect of Elevated Copper on the Rupture Life of Similar Type 316H Heats at 1292°F (700°C) that Exhibit an N_{AV} of Approximately 0.007wt%

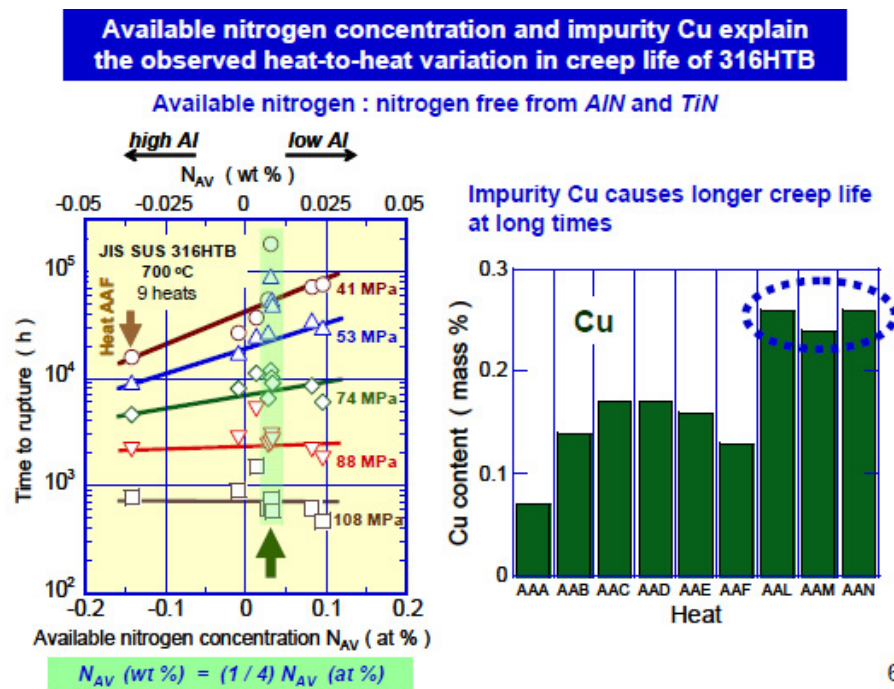
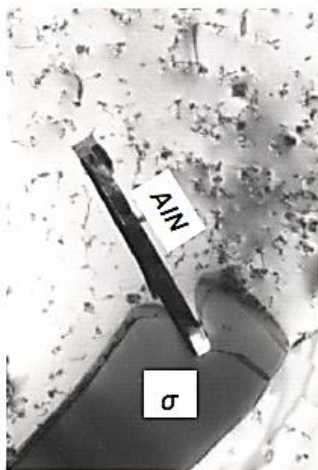


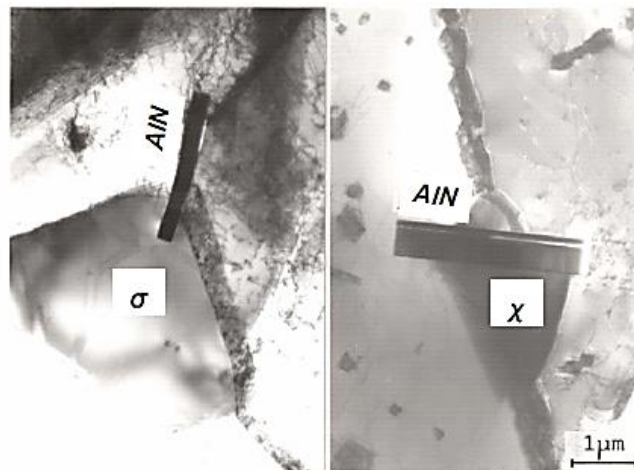
Figure 5 - The Available Nitrogen Concentration and Impurity Copper were Identified as Controlling Variables Responsible for the Observed Heat-to-heat Variation in Creep Performance for Type 316H

JIS SUS 304HTB
heat A



700 °C, 20500 h

JIS SUS 316HTB
heat F



700 °C, 31800 h

800 °C, 3000 h

(N.Shinya, H.Tanaka, M.Murata, M.Kaise and S.Yokoi: 1985)

Figure 6 - TEM Micrographs Showing AlN Precipitates Associated with Sigma (σ) and Chi (χ) Phases at the Grain Boundaries in Type 304H and 316H after Extended High Temperature Exposure

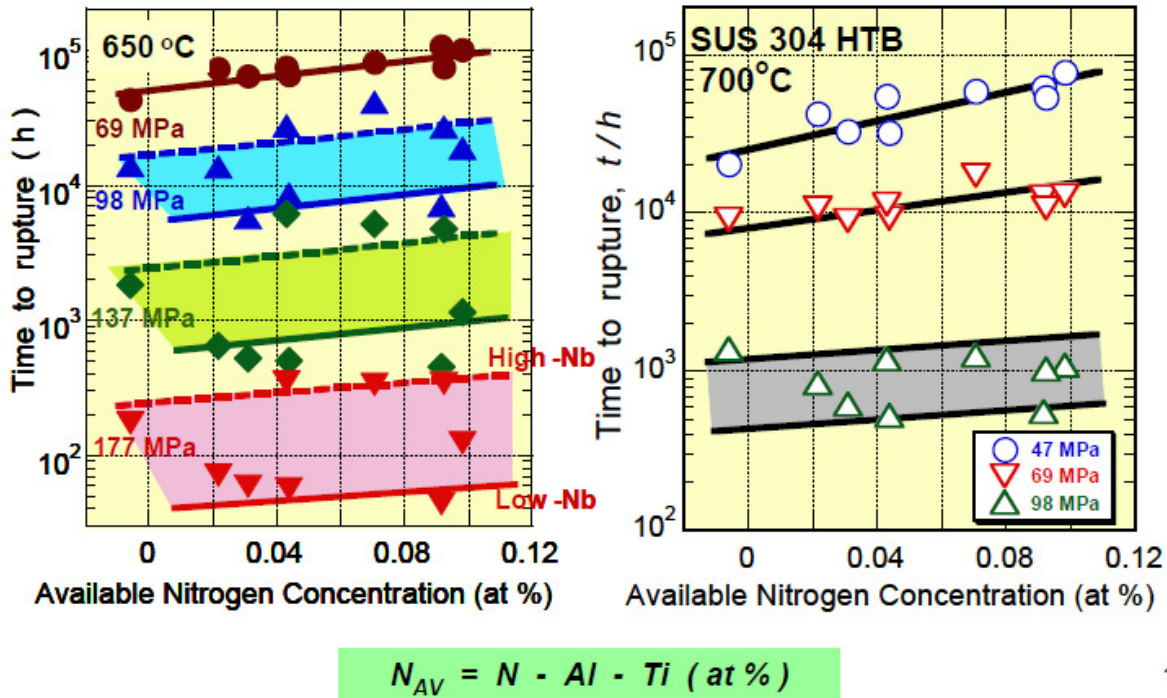


Figure 7 - Creep Rupture Data Showing the Available Nitrogen and Impurity Niobium Explain Heat-to-heat Variability in Creep Life of Type 304H SS

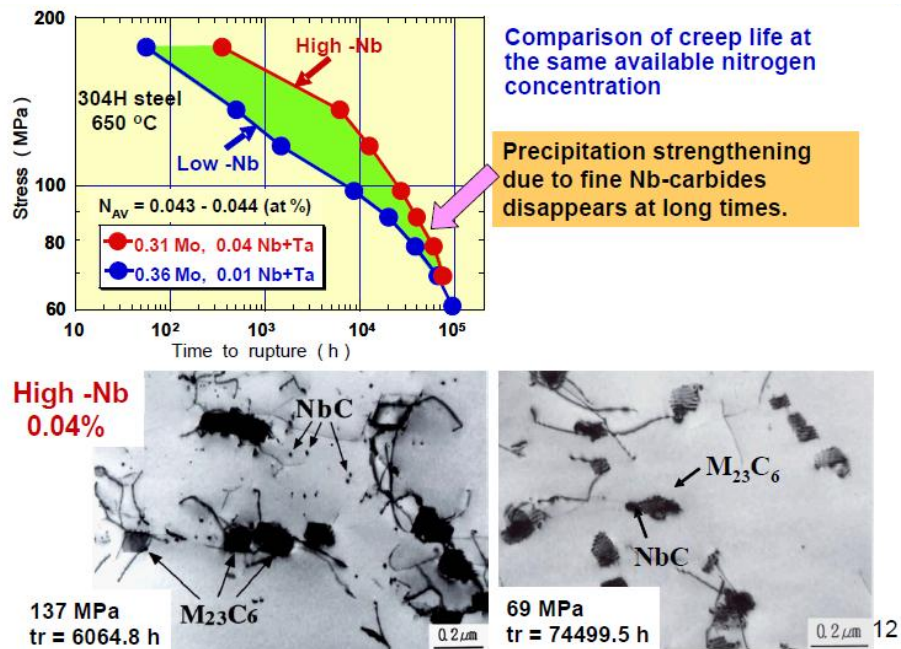


Figure 8 - Creep Rupture Data Showing that at Short Exposure Times, the Presence of Fine Niobium Carbides Improves Strength but the Benefit Disappears after Extended Exposure Times

Explanation of observed heat-to-heat variation in creep life of 304HTB steel

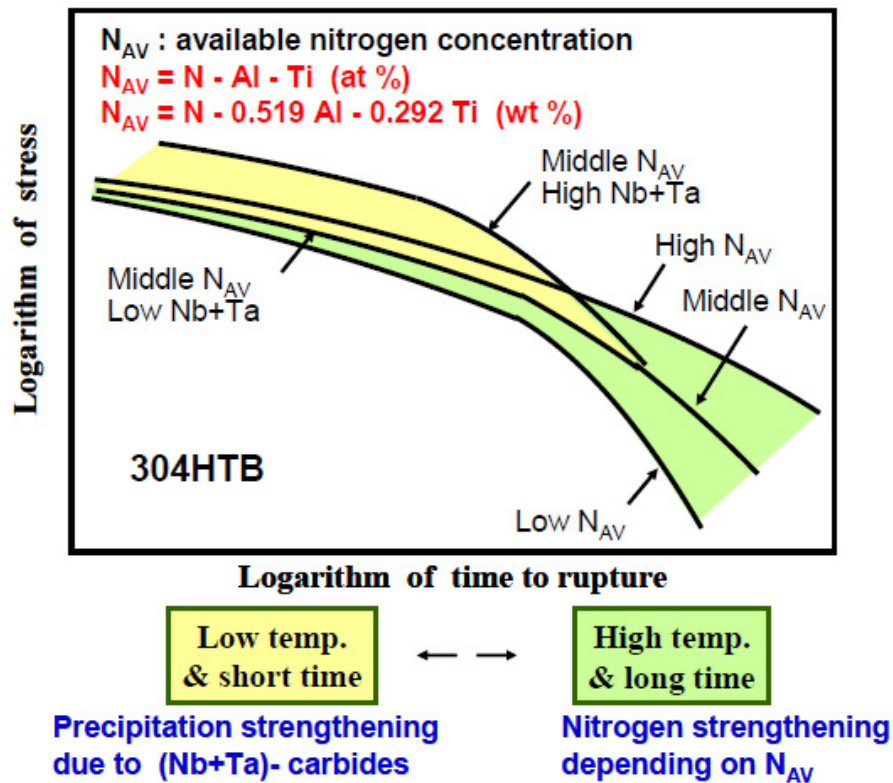


Figure 9 - Illustration Explaining the Observed Heat-to-heat Variability in Creep Performance of Type 304H at Short and Long Exposure Times

3.2 Part 2 - Determine Time and Temperature Limits

As can be seen from the Part 1 results, the primary controlling variables for long-term creep performance in both Type 304H and 316H SS were identified as the amount of nitrogen in interstitial solid solution (available nitrogen), and a minimum of 0.25 wt% of copper. Modifications to the allowable time and temperature limits to accommodate some heats exhibiting poor creep properties can be avoided if the restrictions identified in Part 1 are implemented. These data confirm that it is not necessary to modify the allowable stress values to mitigate the issues associated with scatter in creep data.

3.3 Part 3 - Draft Code Rules

To develop a draft of restriction modifications suitable for consideration as an ASME Code revision to Subsection NH, Appendix X, it was necessary to account for all the potential strong nitride formers present as residual elements in these. Figure 10 shows an Ellingham diagram with the free energy of formation for nitrides typically found in steel. Several of the residual elements present (titanium, niobium and vanadium) are known to form MC type carbo-nitrides at these operating temperatures, making any mass balance calculations conservative due to carbon substitution for nitrogen. MC type carbides are typically stable cubic precipitates that can freely substitute nitrogen for carbon in the structure. There was some disagreement over inclusion of niobium and vanadium because their effect on nitrogen levels would be minor [5]. The commercial heats evaluated contained up to 0.14 wt% niobium and 0.15 wt% vanadium, and at those levels they would be likely to combine with at least 0.03 wt% nitrogen and must be taken into account.

Collaboration with commercial producers was done to ensure recommended restrictions are economical and producible. This arrangement allowed the incorporation of additional Type 316 SS chemical composition data that were useful in establishing that the proposed restrictions were reasonable. Chemical compositions were obtained for 340 domestically produced commercial heats of Type 316 SS, where the amount of copper, nitrogen and residual elements expected to form nitrides (Ti, Al, Nb, V and B) were evaluated. The chemical composition results were organized as listed in

Table 2 shows the average, maximum, minimum and standard deviation for each element of concern. Data for Zr and Ta were not available; however, because of their extremely low free energy of nitride formation, they cannot be discounted. The proposed Table X-2 restrictions include reporting for zirconium and tantalum during initial production runs to develop a database for these elements. If these elements continue to be present at trace levels only, and the restrictions continue to be effective, their reporting can be eliminated if desired at a later date. Figure 11 contains plots showing the distributions of residual elements of concern found in all 340 production heats evaluated. It was apparent that some of the NIMS heats exhibited higher aluminum and titanium while having lower nitrogens than those seen in the 340 commercial heats. Figure 12, Figure 13 and Figure 14 show bar graphs comparing the commercial and NIMS heats for aluminum, titanium and nitrogen.

Figure 15 presents simple extrapolated creep rupture strength results for the 3 proposed Table X-2 compliant NIMS heats, where the average strength value was calculated at 1337°F (725°C). Following typical ASME practice when these Subsection NH allowables were initially established, the minimum stress was calculated at 80% of the average, with the allowable stress for these heats determined as 67% of the minimum stress. This calculation indicated these compliant heats represent an allowable stress that was 1.6 MPa higher than the current ASME Section III, Division I, Subsection NH allowable of 16 MPa at 1337°F (725°C). These results suggest that the proposed Table X-2 compliant heats exhibit significantly better predictability in the creep results, and the allowable stresses and maximum service temperatures should be reestablished using only creep data from Table X-2 compliant heats. Given that only three heats were included in this assessment, additional proposed Table X-2 compliant heats should be included to improve confidence and may allow a more definitive upper service temperature limit that the NIMS data suggests may be above 1337°F (725°C).

This study confirmed that the primary controlling variable for scatter in long-term high temperature creep rupture life is the amount of available nitrogen (in interstitial solid solution). Copper has also shown improvements in creep life at long times at concentrations above 0.25 wt%. The amount of residual strong nitride formers such as aluminum, titanium, vanadium, niobium, tantalum and boron ultimately determine the amount of available nitrogen.

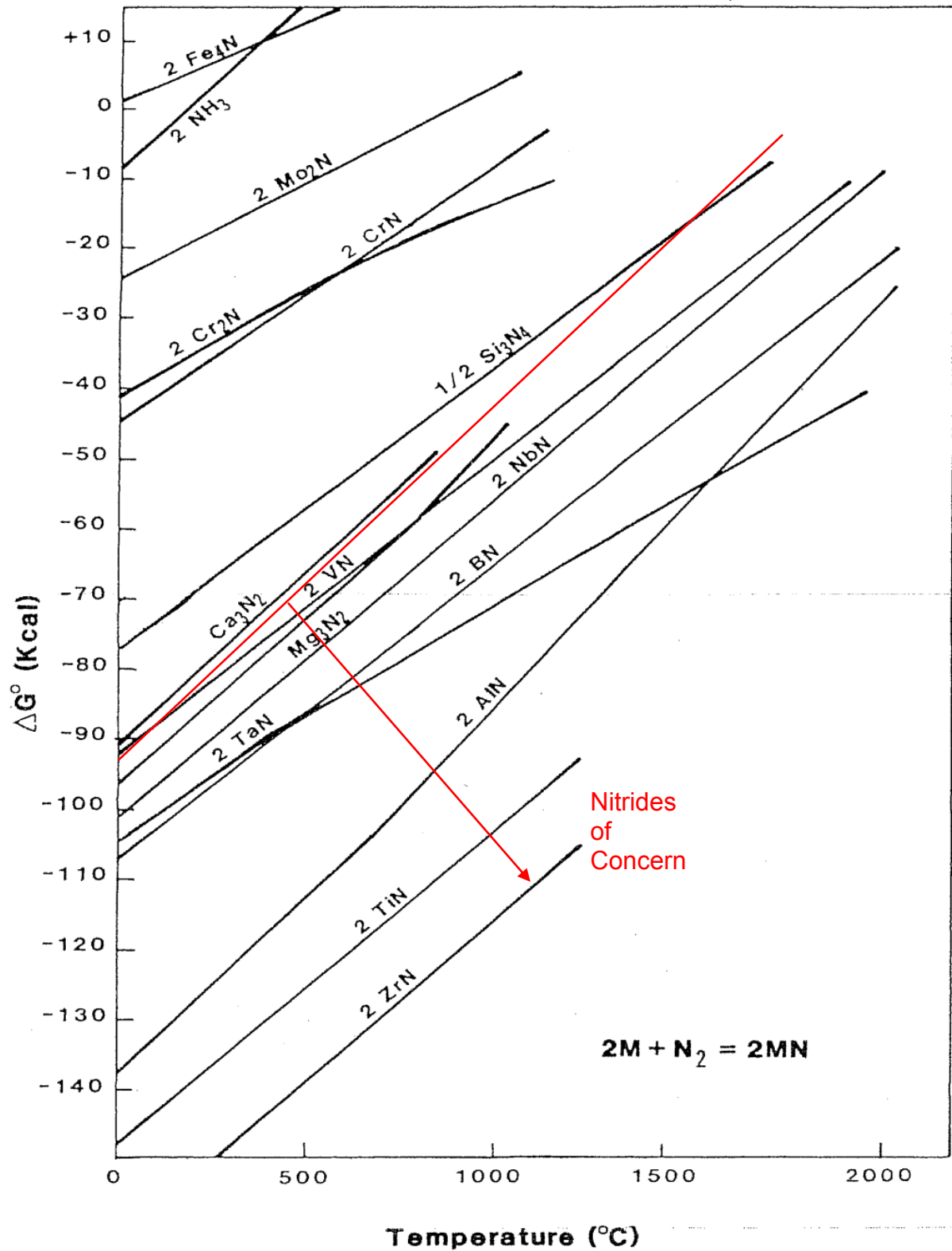


Figure 10 - Ellingham Diagram Showing the Free Energy of Formation for Nitrides, with the Stable Nitrides of Concern in Steel Identified

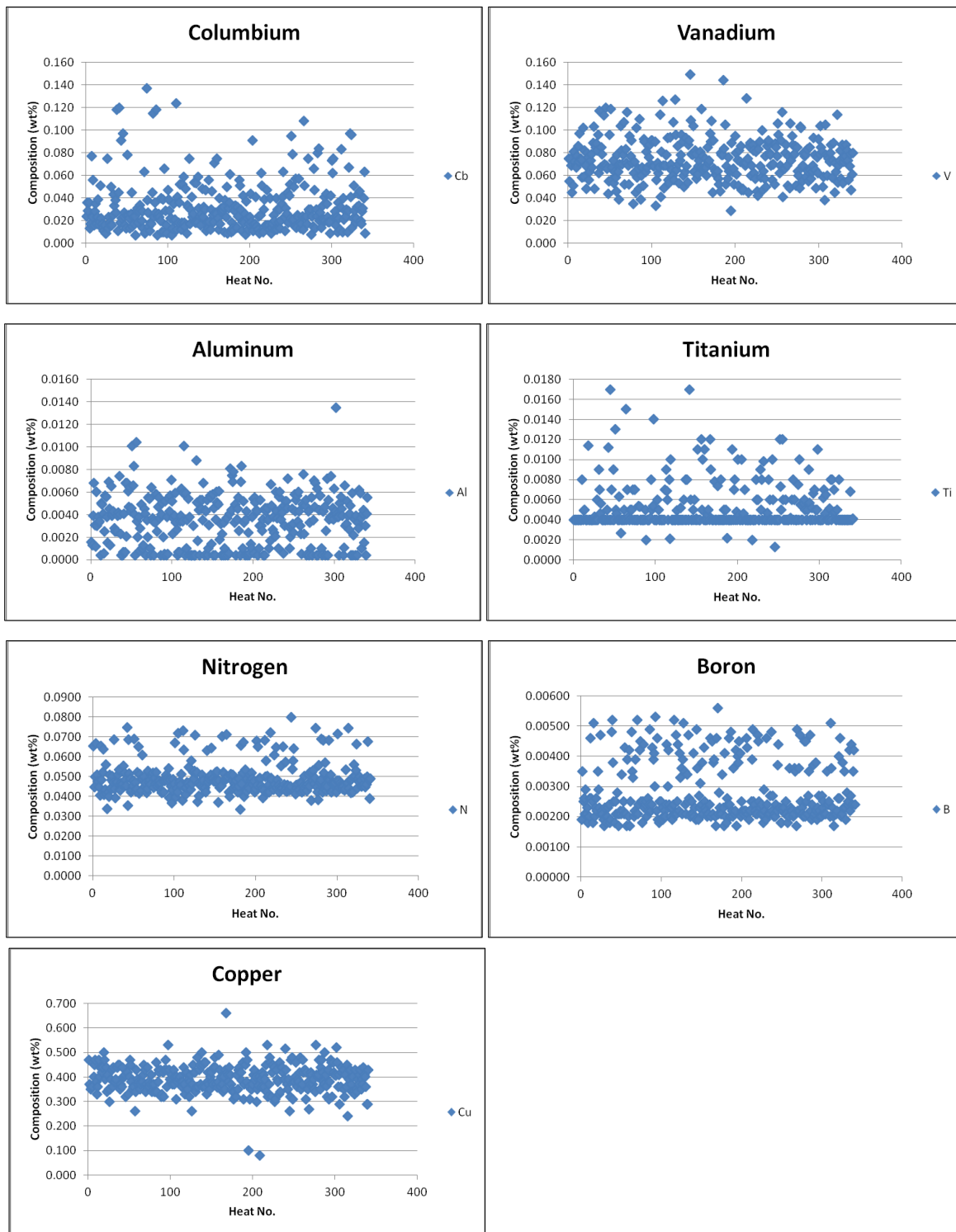


Figure 11 - Plots of the Distribution of Residual Elements Found in the 340 Production Heats

Table 2 - Statistical Composition Results Showing the Average, Maximum, Minimum and Standard Deviations in Weight % for Select Residual Elements Present in the 340 Production Type 316 Heats

	Aluminum	Copper	Columbium	Vanadium	Titanium	Nitrogen	Boron
Average	0.0035	0.392	0.032	0.074	0.0051	0.0490	0.00276
Max	0.0135	0.660	0.137	0.149	0.0170	0.0800	0.00560
Min	0.0004	0.080	0.007	0.029	0.0013	0.0333	0.00170
STDev	0.002253	0.06	0.023	0.02	0.0022	0.0079	0.00097

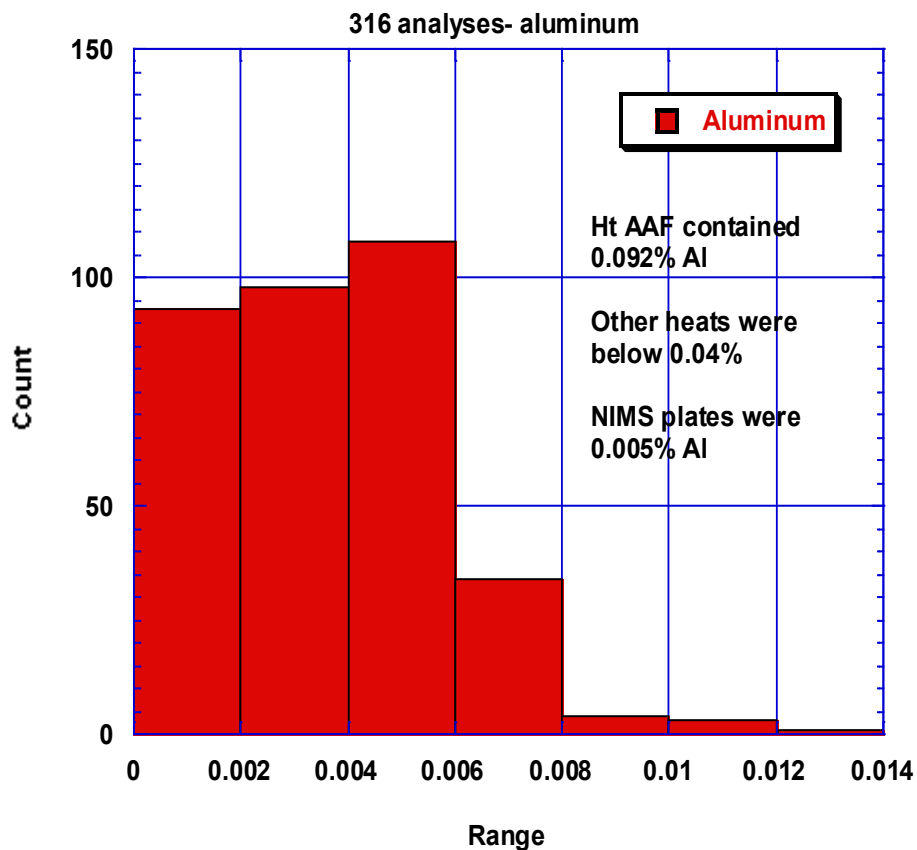


Figure 12 - Bar Graph Showing the 340 Commercial Heats Exhibited Lower Aluminum than the NIMS Heats

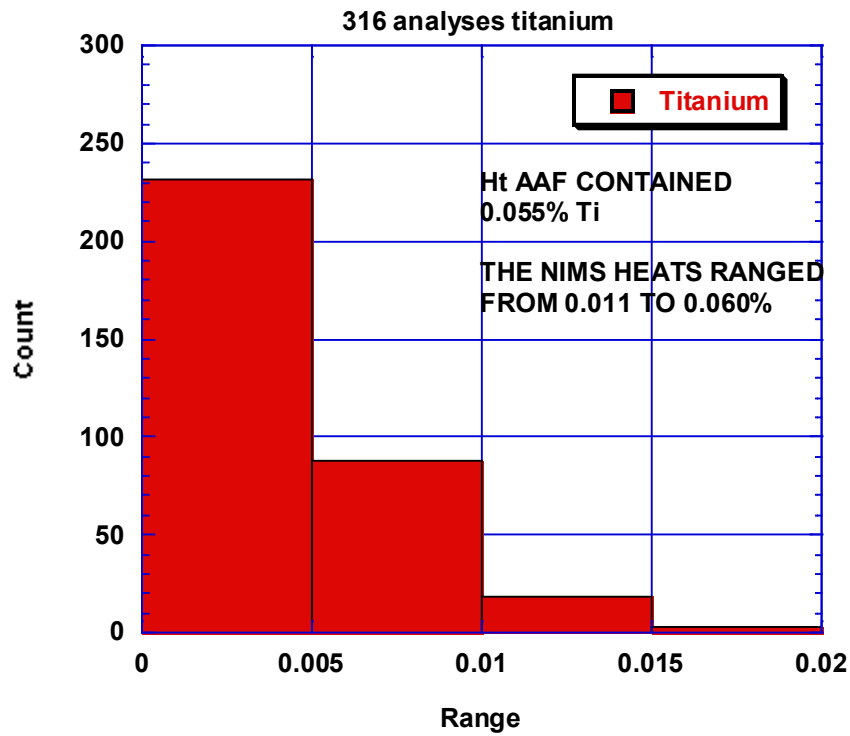


Figure 13 - Bar Graph Showing the 340 Commercial Heats Were Low in Residual Titanium while the NIMS Heats Varied Considerably

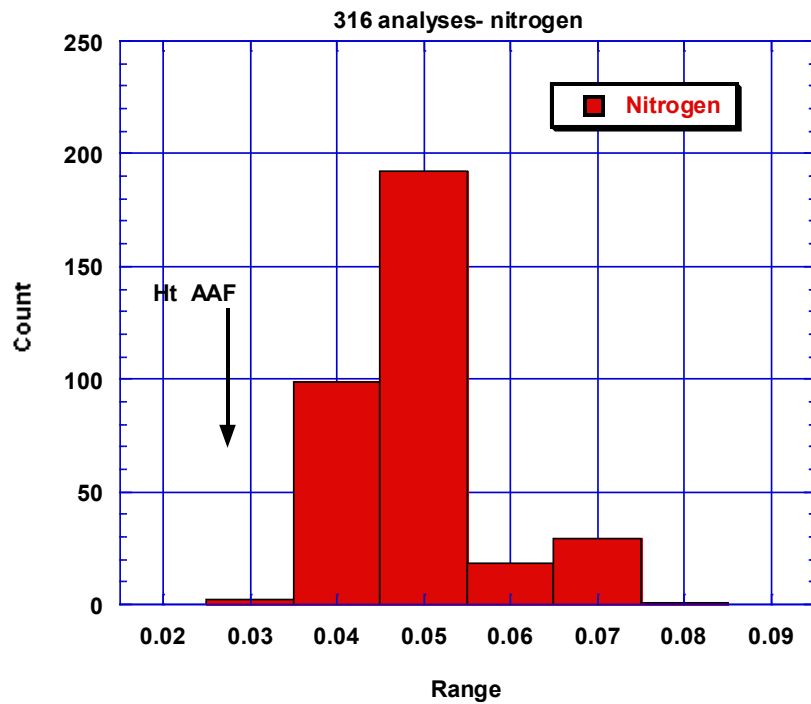


Figure 14 - Bar Graph Showing the 340 Commercial Heats Exhibited Higher Nitrogen than the NIMS Heatsz

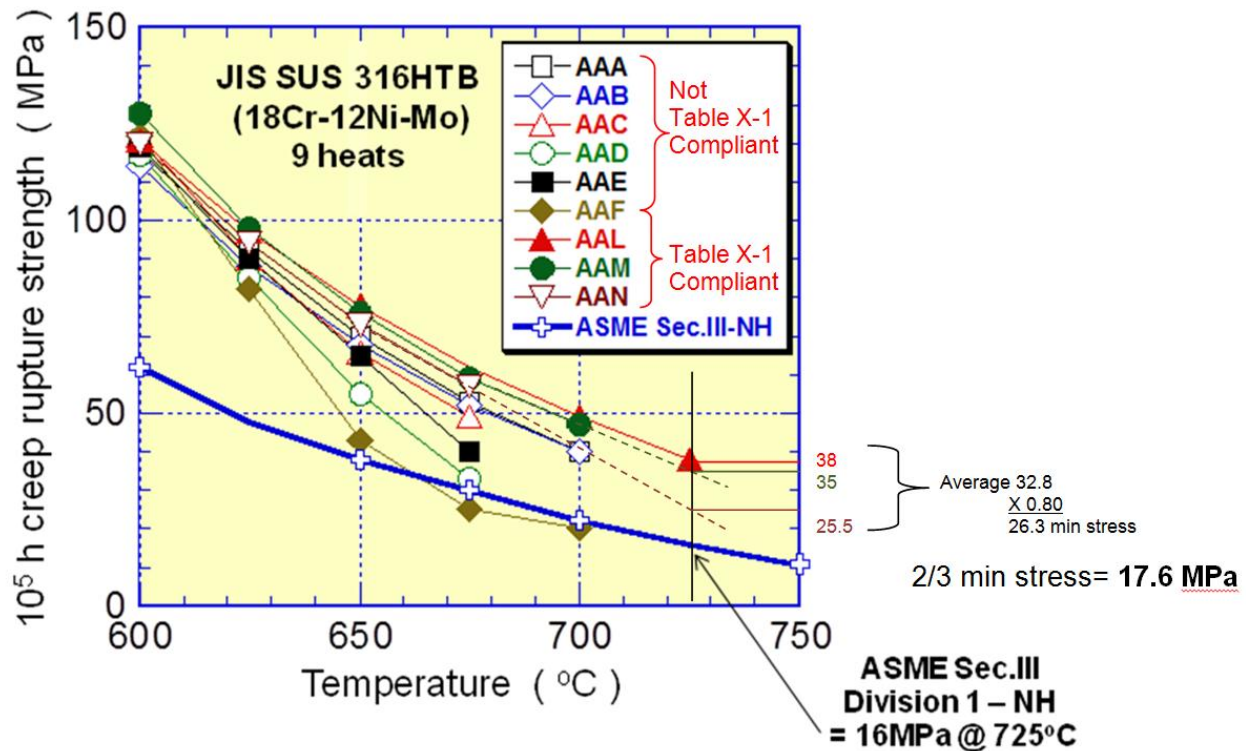


Figure 15 - Minimum Stress Determination for Proposed Table X-1 Compliant NIMS Heats at 1337°F (725°C)

3.4 Recommendations

The current Subsection NH, Appendix X, Table X-1, should be retained as-is for Type 304H and 316H SS service to 1100°F (595°C) until the basis for many of the current restrictions can be determined. A review of the current Table X-1 applicability appears warranted given the strong correlations established between available nitrogen and creep performance as identified herein.

New proposed restrictions are presented in Appendix B, Table X-2, that have been structured to reflect the standard Type 304H and 316H compositions for most primary alloying constituents, with targeted restrictions for copper, nitrogen, nitride formers and undesirable impurities. Long term phase stability has been addressed by including a restriction on the primary ingot ferrite content. These proposed restrictions are considered applicable for long-term service at temperatures between 1100°F (595°C) and 1337°F (725°C). It is recommended that these proposed Table X-2 restrictions be mandatory for this temperature range to take advantage of the improved creep performance associated with these restrictions.

While this investigation focused primarily on the base material properties, fabrication issues such as welding must also be considered to ensure adequate creep performance in assembled components. The properties and phase stability of Type 304H and 316H welds were investigated by Hauser and Van Echo [7], providing significant fabrication guidance to ensure adequate properties during long term service at these temperatures.

4 JUSTIFICATIONS

A justification is provided below for each proposed change as listed in the attached Appendix B, Table X-2. These restrictions are recommended to be mandatory for service temperatures between 1100°F (595°C) and 1337°F (725°C) due to the probability of long operating times at elevated temperatures where there is little data and experience. These proposed restrictions are primarily focused on phase stability and the improvement in creep properties obtained by ensuring sufficient available nitrogen and copper is present to provide a more predictable creep performance. These restrictions were developed with a desire to use the existing UNS numbers for certification of the Type 304H and 316H materials, eliminating additional cost and effort required to certify a new UNS numbered alloy for this application. Use of the basic H grade compositions is also expected to increase the number of potential suppliers for these materials, with the usual benefits associated with competition for contracts. Undesirable impurities such as sulfur and the low melting residuals (tin, antimony, lead, selenium and zinc) along with the grain size range and melt practice are recommended to be left intact from the current Table X-1.

4.1 Carbon

Carbon did not appear to be a significant variable affecting the creep performance, and any effect from the higher carbon levels in the NIMS heats did not appear to be detrimental. The best performers for each alloy exhibited higher carbon than the current Table X-1 range of 0.04 to 0.06 wt%. If there are no identified corrosion, sensitization or other performance concerns with the standard H grade carbon levels, the committee should consider raising the upper carbon limit to 0.10 wt% max. Potential benefits with higher carbon include higher strength with the likelihood that increased competition with nitrogen to form the MC type carbo-nitrides of titanium, vanadium and niobium could result in additional available nitrogen.

4.2 Nitrogen

The levels of available nitrogen (interstitial solid solution) have been shown to have a strong effect on the long-term creep performance of Type 304H and 316H SS. The proposed maximum nitrogen content of 0.10 wt% was established from the proposed Table X-2, Note 2 expression using the maximum values determined for each element of concern from the entire 340 heat population listed in

Table 2. Assuming the compositional variability in the 340 heat population is representative of the capabilities of typical industrial melters, this maximum nitrogen limit should be an easy target for suppliers. Most stainless steel melters have the capability to make conscious nitrogen additions. The button melt analysis used by melt shops to identify final alloying additions is also expected to be used for calculating the final heat nitrogen targets using the Note 2 expression to calculate the amount of nitrogen. Maintaining the nitrogen below 0.10 wt% is considered important for material certifications because it stays below the threshold composition requiring the use of another UNS alloy designation. The proposed Note 2 expression used to calculate the target nitrogen is as follows:

- Nitrogen shall be greater than the sum of $(1.3 \text{ B}\% + 0.52 \text{ Al}\% + 0.29 \text{ Ti}\% + 0.28 \text{ V}\% + 0.15 \text{ Nb}\%)$ but less than 0.100 wt%.

As these elements typically form MC type carbo-nitride compounds in a 1:1 stoichiometric ratio, the constants associated with each element in this expression only represent a conversion from atomic percent to weight percent.

4.3 Silicon

All of the Type 316H NIMS heats exhibited silicon above the current Table X-1 value, indicating that silicon in this range did not appear to significantly impact creep properties, and would be a candidate to be relaxed to standard Type 316H values. However, high silicon levels are known to promote formation of

sigma phase in these alloys during extended high temperature service, and, as such, are recommended to be maintained at the current Table X-1 values.

4.4 Nickel

Many of the good creep performing heats exhibited nickel values above the current Table X-1 levels, indicating the current restriction does not by itself significantly affect creep properties. Nickel was initially restricted to increase the probability of ferrite formation. Higher nickel is usually necessary in Type 316 grades to “balance” the composition, by offsetting the tendency to form ferrite from chromium and higher molybdenum additions [4]. Relaxing the nickel restriction to reestablish the high end of the range to conventional Type 304H and 316H compositions would promote austenite phase stability, which is considered important for long-term high temperature service, and provide the melter with the flexibility necessary to achieve a balanced composition.

4.5 Chromium

Chromium is a ferrite stabilizer, and as such, may promote ferrite formation when at the high end of the allowable range. Chromium was initially restricted to increase the probability of ferrite formation [6]. There did not appear to be a direct correlation between creep performance and chromium levels; however, the best performing heat exhibited the lowest chromium, while the worst performer exhibited the 2nd highest. The chromium levels determined for the Type 316H NIMS heats were all at the low end of the range or slightly below the current restriction of 17.0% minimum, indicating the current restriction may be ineffective. Allowing melters to produce heats with chromium at the low end of the Type 304H and 316H ranges would encourage austenite phase stability and possibly yield a small economic benefit.

4.6 Copper

Copper was not a controlled element in the current Table X-1, yet it is a common impurity in austenitic stainless steels. The positive effect on creep performance became apparent after 103 hours at 1292°F (700°C) for the Type 316H SS, but unfortunately, the NIMS data did not list copper contents for the Type 304H heats. The average copper content identified in the 340 commercial heat database was 0.392 wt%, while the average for the NIMS data was less than 0.20 wt%. Data was only available for Type 316H; however, the copper restriction is also recommended for the Type 304H due to the effect of copper on Type 316H creep performance and the expected effect on Type 304H due to its similar composition.

4.7 Molybdenum

The current Table X-1 restriction for molybdenum was to bias the composition to the high end of the range, likely to take advantage of its effect on high temperature strength [4]. All of the Type 316H NIMS heats exhibited lower molybdenum than the current Table X-1, including the three best performing heats, and it does not appear that a restriction for molybdenum is warranted.

4.8 Sulfur and Phosphorous

While sulfur did not appear to have any noticeable effect on creep properties, the current sulfur restrictions for Type 304H and 316H appears appropriate given the recommendation to restrict the ferrite content to 1-2%. High levels of sulfur have been associated with weld solidification cracking at these low ferrite levels, and it is recommended to continue the 0.02 wt% maximum restrictions.

While phosphorous did not appear to have any noticeable effect on creep properties, the current restriction for Type 316H appears appropriate given the recommendation to restrict the ferrite content to 1-2%. Phosphorous has been associated with weld solidification cracking at these low ferrite levels, and it is recommended to apply the 0.030 wt% maximum restriction for 304H.

Figure 16 shows the susceptibility of austenitic chromium-nickel steels to solidification cracking as a function of the chromium equivalent/nickel equivalent and sulfur and phosphorous compositions. Modern

steelmaking practice makes it easy to reduce these tramp impurities to low levels, reducing the probability of solidification cracking due to low ferrite levels.

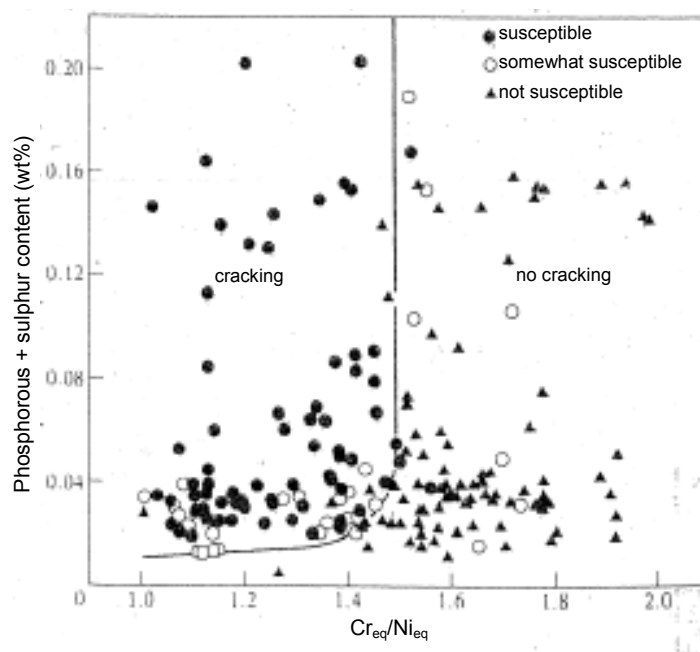


Figure 16 - The Susceptibility of Austenitic Chromium-nickel Steels to Solidification Cracking as a Function of Schaeffler Cr_{eq}/Ni_{eq} and Sulfur and Phosphorous Contents (from Kujampaa et al., 1980)

4.9 Nitride Formers

This group of elements is known to form stable nitrides in steels and as such must be accounted for to accurately control the amount of nitrogen in solid solution. The Note 2 expression in the proposed Table X-2 effectively accounts for all the potential nitride forming elements typically present in these alloys, and calculates the amount of nitrogen necessary to completely combine with all the nitride formers. When all the nitride formers are tied up as stable nitrides, additional nitrides cannot form, leaving the requisite amount of nitrogen in interstitial solid solution to affect the creep properties. This approach is considered conservative because some of the nitride formers such as titanium, vanadium and niobium also form stable MC type carbo-nitride compounds where the carbon displaces some of the nitrogen, leaving additional nitrogen in solution in excess of the stoichiometric amount necessary to form the pure nitrides. Two of the most stable nitride formers, zirconium and tantalum, are not typically reported on material certifications and as such their levels are not accurately known. These elements are assumed to be present at trace levels only, and as such are not expected to significantly affect the formation of stable nitrides. Their reporting for information during early production heats is expected to result in a database allowing a determination of whether these elements need to be accounted for in the Note 2 expression, or if they can be ignored because of their low levels.

4.10 Higher Service Temperature Range

Heats that are compliant with the proposed Table X-2 restrictions show a significant reduction in scatter at elevated temperatures and long times, and also exhibited the highest creep strengths in the materials studied. The performance of these compliant Type 316H heats suggests that there is significant margin in the current upper temperature service limit of 1100°F (595°C). A straight extrapolation of the NIMS creep strength data indicates the actual upper limit could be approximately 1337°F (725°C); however, only three

316H heats were used to estimate this upper limit. Additional confidence in this upper temperature value could be realized by including additional proposed Table X-2 compliant heats in the database.

4.11 Ferrite Number

Delta ferrite (δ) contents in austenitic SS are known to impact both long term high temperature phase stability and fabricability. Deleterious phases such as sigma (σ) form in austenitic stainless steels are due to decomposition of ferrite at elevated temperatures and long times. Sigma is known to significantly affect fracture and creep properties after long term exposure, and may have been responsible for at least some of the observed scatter in the creep database. Ferrite content is not currently measured or reported for creep test heats. It is recommended to include a 1% to 2% requirement for ferrite content to balance long term phase stability with fabricability.

A requirement for ferrite levels is not new, as Japanese [6] manufacturers have specified a 1% max ferrite requirement for elevated temperature service since the 1980s. This requirement reportedly restricts the amount of delta ferrite present in the primary ingot material, apparently to eliminate issues of ferrite determination such as from cold work during final product processing. The ferrite content can easily be measured using a commercial “ferrite indicator,” which is a non-destructive test with a typical range of 0.1 to 115 Ferrite Numbers (FN), equivalent to 0.1 to 83 wt% delta ferrite in austenitic and duplex steel.

The chemical composition balance specified for Type 316 stainless steel is driven by two major factors: minimizing fabrication problems and optimizing creep properties.

- a) The fabrication problems are primarily hot working and solidification cracking issues during welding, where experience has shown it can be minimized by the presence of a small amount of ferrite (typically 2 to 10%). This is readily achieved by a composition balance that favors high chromium equivalents that are defined in the text.
- b) Maximizing the creep properties is readily achieved by minimizing the amount of ferrite. This optimization is achieved by balancing a high chromium equivalent with a relatively high nickel equivalent. A recommendation to enhance creep properties while providing adequate fabricability would be a range of 1 to 2% percent ferrite in the primary ingot.

Reinstating the UNS composition ranges for these alloys and only targeting specific elements as identified herein is believed to provide suppliers sufficient leeway to balance heat compositions to achieve the target ferrite amounts in the most cost effective manner for their facilities. Schaeffler or DeLong Diagrams have proven reliable in establishing the composition makeup to balance fabrication and creep properties.

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- [2] T. Rosenquist, *Principals of Extractive Metallurgy*, McGraw Hill, New York, 1974
- [3] R. Swindeman, ASME ST-LLC Task 6, *Operating Condition Allowable Stress Values in ASME Section III Subsection NH*, STP-NU-037, ASME Standards Technology LLC, May 12, 2010
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- [5] Personal communication with Dr. Fujio Abe, January 31, 2012
- [6] C.R. Brinkman, V.K. Sikka and B.L.P. Booker, "Optimized Specification for Types 304 and 316 Stainless Steel in Long-Term High-Temperature Service Applications", ORNL-6005, June 1984
- [7] Daniel Hauser and John A. VanEcho, "Effects of Ferrite Content in Austenitic Stainless Steel Welds", Battelle Laboratories, July 29, 1977

APPENDIX A

CURRENT APPENDIX X GUIDELINES FOR RESTRICTED MATERIAL SPECIFICATIONS TO IMPROVE PERFORMANCE IN ELEVATED TEMPERATURE SERVICE (ASME Section III, Division I, Subsection NH, Appendix X)

2010 SECTION III, DIVISION 1 — NH

NONMANDATORY APPENDIX X GUIDELINES FOR RESTRICTED MATERIAL SPECIFICATIONS TO IMPROVE PERFORMANCE IN CERTAIN SERVICE APPLICATIONS

X-1100 SCOPE

This Appendix provides guidelines on specification restrictions for Types 304 and 316 stainless steels, which will, in the opinion of the Committee, improve the performance of the permitted materials in certain elevated temperature nuclear applications where creep effects are significant. The restrictions have the effect of narrowing chemical composition, grain size, and other aspects of material quality while staying within the broader specification limits defined in Table I-14.1(a) and its notes.

X-1110 OBJECTIVES

The purpose of these restrictions is to achieve materials which have significantly reduced scatter in creep and rupture properties, with mean strength values consistent with

or better than the current database, while maintaining satisfactory weldability and fabricability.

X-1200 SERVICE CONDITIONS

The restrictions of this Appendix will provide improved performance when materials are used within the temperature regimes of 800°F to 1,100°F (425°C to 595°C). For application outside of those regimes, this Appendix provides no guidance.

X-1300 RECOMMENDED RESTRICTIONS

The recommended restrictions are shown in Table X-1.

TABLE X-1
RECOMMENDED RESTRICTIONS

Element	Type 304	Type 316
(a) Chemical Composition [Note (1)]		
Carbon	0.04–0.06	0.04–0.06
Nitrogen	0.04–0.07	0.04–0.07
Silicon	0.6	0.6
Manganese	1.0–2.0	1.0–2.0
Nickel	8.00–10.00	11.00–12.5
Chromium	18.5–20.00	17.00–18.00
Molybdenum	0.2	2.5–3.0
Sulfur	0.02	<0.02
Phosphorus	0.045	<0.03
Niobium	0.02 [Note (2)]	...
Aluminum	0.05	0.05
Antimony	0.02	0.02
Boron	...	0.003 [Note (3)]
Lead	0.003	0.003
Selenium	0.015	0.015
Tin	0.015	0.015
Vanadium	0.05	0.05
Zinc	0.01	0.01
(b) Grain Size (ASTM)	3–6	3–6
(c) Melt Practice	AOD or AOD/ESR	AOD or AOD/ESR
(d) Suggested upper long-term use limit for improved performance:		
Temperature, °F (°C)	1,100 (595)	1,100 (595)

NOTES:

- (1) All values are maximum percentages unless indicated as ranges.
 (2) To further reduce data scatter, a minimum value of 0.005% should be specified.
 (3) To further reduce data scatter, a minimum value of 0.0015% should be specified.

APPENDIX B

PROPOSED APPENDIX X GUIDELINES FOR RESTRICTED MATERIAL SPECIFICATIONS TO IMPROVE PERFORMANCE IN ELEVATED TEMPERATURE SERVICE (ASME Section III, Division I, Subsection NH, Appendix X)

APPENDIX X GUIDELINES FOR RESTRICTED MATERIAL SPECIFICATIONS TO IMPROVE PERFORMANCE IN ELEVATED TEMPERATURE SERVICE

X-1100

SCOPE

This Appendix provides guidelines on specification restrictions for Types 304 and 316 stainless steels which will, in the opinion of the committee, improve the performance of permitted materials in certain elevated temperature nuclear applications where creep effects are significant. The restrictions have the effect of narrowing the chemical composition, grain size, and other aspects of material quality while staying within the broader specification limits defined in Table I-14.1(a) and its notes.

X-1110

OBJECTIVES

The purpose of these restrictions is to achieve materials which have significantly reduced scatter in creep and rupture properties, with mean strength values consistent with or better than the current database, while maintaining satisfactory weldability and fabricability.

X-1200

SERVICE CONDITIONS

The restrictions of this Appendix will provide improved performance when the materials are used within the temperature ranges of 800°F to 1,100°F (425°C to 595°C) and 1,100°F to 1,337°F (595°C to 725°C).

X-1300

RECOMMENDED RESTRICTIONS

The restrictions consist of non-mandatory requirements for service at 800°F to 1,100°F (425°C to 595°C) as identified in Table X-1, and mandatory requirements for service at 1,100°F to 1,337°F (595°C to 725°C) as identified in Table X-2.

TABLE X-2
REQUIRED RESTRICTIONS

Element	Type 304	Type 316
(a) Chemical Composition [Note (1)]		
Carbon	0.04-0.10	0.04-0.10
Nitrogen	[Note (2)]	[Note (2)]
Silicon	0.6	0.6
Manganese	1.0 - 2.0	1.0 - 2.0
Nickel	8.00-10.00	11.00-14.00
Chromium	18.0-19.50	16.00-17.50
Copper	0.25-0.55	0.25-0.55
Molybdenum	0.2	2.0-3.0
Sulfur	0.02	0.02
Phosphorus	0.030	0.030
Aluminum	For Information ³	For Information ³
Boron	For Information ³	For Information ³
Niobium	For Information ³	For Information ³
Tantalum	For Information ⁴	For Information ⁴
Titanium	For Information ⁴	For Information ⁴
Vanadium	For Information ³	For Information ³
Zirconium	For Information ⁴	For Information ⁴
Antimony	0.02	0.02
Lead	0.003	0.003
Selenium	0.015	0.015
Tin	0.015	0.015
Zinc	0.01	0.01
(b) Grain size (ASTM)		
	3-6	3-6
(c) Melt Practice		
	AOD or AOD/ESR	AOD or AOD/ESR
(d) Applicable long-term temperature range for identified restrictions:		
Temp, °F (°C)	1,100 (595) to 1,337 (725)	1,100 (595) to 1,337 (725)
(e) Ferrite Number (FN)		
	1-2 ⁵	1-2 ⁵

Notes:

- (1) All values are maximum percentages unless indicated as minimums or ranges.
- (2) Nitrogen shall be greater than the sum of $(1.3 \text{ B}\% + 0.52 \text{ Al}\% + 0.29 \text{ Ti}\% + 0.28 \text{ V}\% + 0.15 \text{ Nb}\%)$ but less than 0.100 wt%.
- (3) Changed from a composition limit to “For Information” to allow nitrogen calculation.
- (4) Added additional elements for nitrogen calculation and to develop database for additional nitride forming elements not currently reported.
- (5) Incorporated a ferrite number (FN) requirement applicable to the primary ingot to balance long-term high-temperature phase stability with allowance for fabricability.

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