by E. E. Gruber and T. H. Hughes

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EXPERIMENTAL DETERMINATION OF THE HIGH-TEMPERATURE
INTERMEDIATE-TIME MECHANICAL PROPERTIES OF ALLOY HT9

by

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ABSTRACT

The stainless-steel alloy HT9 is the leading candidate for the cladding and duct material of the Integral Fast Reactor (IFR) fuel system. This report describes an experimental program to obtain information concerning the mechanical behavior of HT9 at temperatures and strain rates relevant to the safety analyses of fast reactors with IFR fuel designs. Analysis of existing short-term tensile data and long-time creep data has revealed a significant discrepancy in the behavior of HT9 in the mid-time scale between tensile tests and creep tests. The material shows an unusually high creep strength during long-time creep tests in comparison to its high temperature flow strength from short-time tensile tests. This anomalous behavior is being studied in a series of low-strain-rate uniaxial tensile tests using an Instron 8500 series digital testing machine. The data is being used to develop constitutive equations describing the material flow and will be especially useful at temperatures and strain rates relevant to the study of hypothetical reactor accident events lasting hours or days. This report describes the equipment being used and the results of 11 tests at temperatures ranging from 20°C to 700°C, and constant strain rates ranging from 1.5×10^{-7} to 4×10^{-3} per second.

I. INTRODUCTION

This report describes an experimental program to obtain information concerning the mechanical behavior of alloy HT9 at temperatures and strain rates relevant to the study of hypothetical reactor accident situations. The stainless-steel alloy HT9 has been selected as the material for fuel cladding tubes, end plugs and subassembly duct walls in the Integral Fast Reactor (IFR) fuel system. The alloy HT9 has good corrosion resistance, good high-temperature strength and, in particular, excellent resistance to swelling in the fast neutron environment. The computer codes used to carry out design and safety studies of the reactor components fabricated from HT9 require constitutive equations describing the mechanical behavior of the material. Such constitutive models have been developed at ANL[1] using short-term tensile data and long-time creep data. This analysis has highlighted a significant discrepancy in the behavior of HT9 in the mid-time scale between tensile tests and creep tests. The material shows an unusually high creep strength during long-time creep tests in comparison to its high temperature flow strength from short time tensile tests. To account for this behavior, a fine distribution of carbide precipitates is postulated to evolve over time during creep.[2]

Unfortunately data is not available in this transition region between creep and tensile tests. The necessary data is being acquired in a series of low-strain-rate uniaxial tensile tests using an Instron 8500 series digital testing machine. These experiments will fill two voids in the database of mechanical properties. The first void is in data at temperatures between room temperature and 650°C at strain rates of 10^{-4} to 10^{-8} in./in./s. This data will help to resolve the apparent inconsistency in the values extrapolated from existing tensile and creep data. The second void in the database is the lack of any data above the ferrite-to-austenite phase transition beginning at about 840°C. Future tests will be run at temperatures in this regime up to about 1000°C. The specimens of HT9 have been fabricated from rod stock supplied by Carpenter Technology to Argonne National Laboratory for use as fuel cladding end plugs. In order to control the specimen temperature, the section of the load string holding the specimens is mounted inside a furnace. Although the alloy HT9 is considered stainless, there is substantial oxidation at elevated temperatures if the tests are carried out in air. The tests are being conducted using two different furnace configurations. In the series of "low-temperature" tests described in this report a split-tube type furnace is being used, in which the specimen oxidation is reduced to an acceptable level by introducing a flow of helium

through a quartz tube surrounding the specimen. This report describes the equipment and the results of 11 tests at temperatures ranging from 20°C to 700°C.

II. EXPERIMENTAL EQUIPMENT

A. Instron 8562 Testing System

An Instron 8562 universal testing machine is being used to perform the tensile tests. This machine is one of Instron's new generation of 8500 series mechanical testing systems which use computer-based technology to provide full digital control of the machine. A photograph of the Instron 8562 and peripheral equipment that have been assembled for the tests is shown in Fig. 1. The Instron 8500 series electronic control hardware contains several microprocessors that provide (1) load, position and strain control; (2) wave generation and (3) buffered data collection. These functions are accessed either by pressing keys on the front panel (just to the left of the computer in Fig. 1) or by sending commands from a computer over a communication link.

The Instron 8562 applies the required load to the test specimen by means of an electro-mechanical actuator, which is mounted in the base of a 250kN (55 kip) load frame. This actuator employs a single nonrotating screw driven vertically by a rotating, captive ball/nut assembly. The electro-mechanical actuator was specifically developed for applications, such as the experiments planned here, where high resolution, long-term reliability and economy of operation are required. The system is free from axial backlash and, since the crosshead is fixed, system inertia is minimized. The prime mover is a high performance, low inertia, electro servo-motor which provides high resolution drive combined with long-term stability. Axial resolution of the actuator is 0.05 μm with a velocity up to 350 mm/min. The highest velocity would correspond to a strain rate of 0.23 s^{-1} . There is virtually no limit to the minimum strain rate.

An Instron load cell attached to the top crosshead is used to measure the force applied to the tensile specimens. A mathematical model[1] has been developed for the mechanical behavior of HT9 which describes the results of short-term tensile tests. We have used this model to estimate the load-time behavior for the tensile tests that are required by this project. Even though we may

Fig. 1. Instron 8562 Universal Testing Machine and Peripheral Equipment
(ANL Neg. No. 11575K)

see changes in the microstructure that will strengthen the alloy during the tests, these calculations are satisfactory for the purpose of determining the required size of the load cell. For example, at 600°C and strain rates between 4.0×10^{-8} and $4.0 \times 10^{-2} \text{ s}^{-1}$ the maximum force range for 0.220 in.-dia specimens is between 300 and 2300 pounds. At 400°C the range is 2200-3000 pounds; at 800°C the range is 100 pounds to 800 pounds. The load cells that we have available for the tests are rated for dynamic testing up to 2200, 5600 and 22,400 pounds. Thus, for most of the tests the 5600 pound load cell is suitable. The accuracy of the Instron 2518 load cells is 0.005% of the rating or 0.5% of the indicated load, whichever is greater. Figure 2 shows the accuracy we can expect for the load cells at low loads.

Fig. 2. Accuracy of Instron No. 2518 Series Load Cells

B. Load Train

The load train consists of the hardware that is used to couple the tensile specimens to the Instron 8562 testing machine. A drawing of the load train that has been assembled for the initial tests of HT9 is given in Fig. 3. The threaded specimen couplings and the hot rods are made of a high-temperature nickel alloy to withstand the environment of the furnace that will be used to heat the samples. Two alignment couplings (universal joints) near the top and bottom of the train assure that no bending loads will be transmitted to the tensile specimens when they are pulled. The remaining components of the load train are dimensioned to span the distance between the actuator and the load cell and are designed to allow for assembly and disassembly of the apparatus. We have experienced difficulty in removing the threaded specimens from the holders after exposure to the higher temperatures. The solution is to use split holders fabricated of 316SS. A drawing of these holders is shown in Fig. 4. The two halves of these holders are held together by bolts that can be easily sheared off if they do not unthread easily.

C. Tube Furnace with Helium Flow System

The high-temperature tensile testing of alloy HT9 is complicated by the need to provide a protective atmosphere to prevent excessive oxidation of the HT9 samples during testing. We have performed some preliminary experiments[3] on oxidation rates and have found that the as-received HT9 stock oxidizes readily in air at high temperatures. However, the tensile specimens must be heat treated prior to mechanical testing to develop the same microstructure that HT9 cladding tubes have when fuel pins are fabricated. We perform this heat treatment in a tube furnace, with a flowing helium purge gas system to protect the tensile specimens. This procedure leaves a thin contiguous layer of chromium oxides that resist further oxidation. Our oxidation studies[3] showed, for instance, that the oxide layer on as-received material exposed to air at 600°C for 5 days was about 10 μm thick; the oxide layers on material previously heat-treated in flowing helium and then exposed to air under the same conditions was only 1 μm thick. Thus, it appears that our heat treating procedure forms a protective coating on the tensile specimens that resists further oxidation, at least at temperatures up to 600°C, but is sufficiently thin so as not to influence the mechanical properties measurements.

Fig. 3. Load Train

Fig. 4. Drawing of Split Specimen Holder

Our initial mechanical testing uses an Instron Model 3117 High Temperature Furnace System with a Model 3120 Self-Adaptive Temperature Controller. In the photograph in Fig. 1 the furnace is mounted in the load frame and the controller is in the center of the picture. The furnace is split vertically to provide free access to the test specimen area. It has three resistance-heated zones to minimize temperature gradients. The maximum rated temperature of the furnace is 1000°C. The Model 3120 controller is a micro-processor based instrument that automatically provides optimum temperature control within the desired temperature range. The temperature history can be programmed from the front console of the controller or from a personal computer via a GPIB interface.

Temperatures are controlled and monitored with two thermocouples (Type K or Type N), spot welded to the upper and lower shoulders of each specimen. The temperature controller automatically adjusts the power to the three zones of the furnace to reach and maintain the desired temperature at both of these thermocouple locations. The Type K thermocouples are made from

"Special Limits of Error" wire, with a claimed limit of error of 1.1°C or 0.4% of the measured temperature, whichever is greater. Calibration of the Type N thermocouples is reported in Appendix A.

We have also fabricated a helium purge gas system for the Instron furnace to further protect the test samples from oxidation during the experiments. This system consists of a large-diameter quartz tube which fits inside the furnace bore, along with end plates, supply tubing, and a helium tank. The quartz tube surrounds the test section and channels a steady stream of pure helium past the HT9 tensile specimens. Figure 5 is a photograph showing details of the furnace and purge gas system. The furnace is in the open position with the quartz tube suspended above the base plate by three chains. A tensile specimen can be seen in the center of the photograph with an extensometer attached. The heat shields that have been fabricated for use near the top and bottom of the quartz tube are not shown in the picture.

D. Centorr Furnace

The high temperature tests above 650°C will be performed within a sealed furnace that can be evacuated and backfilled with an inert gas. These tests will be needed to measure the mechanical response of HT9 at temperatures in excess of the ferrite-to-austenite transition temperature. For HT9 this transition begins at 840°C and is complete at 960°C. We will be using a Centorr M-60 high temperature vacuum furnace (shown in Fig. 6). This furnace is equipped with a physical testing kit for performing tensile tests using a loading machine such as the Instron 8562. The physical testing kit has water-cooled stainless-steel pull rods, adjustable to various specimen lengths, which penetrate through the top and bottom of the furnace. Welded stainless-steel bellows seal the pull rods to the furnace chamber. The furnace can be evacuated and backfilled with an inert gas. The maximum temperature of the Centorr furnace is 1650°C, which exceeds the melting point of HT9 (1400°C).

Fig. 5. Instron Model 3117 High Temperature Furnace and Purge Gas System
(ANL Neg. No. 11576K)

Fig. 6. Centorr M-60 High Temperature Vacuum Furnace
(ANL Neg. No. 11582K)

E. Extensometers

The strains in the HT9 tensile specimens are determined by means of an extensometer which measures the deformation within the gauge length. Our test setup requires an extensometer with rods transmitting the displacement in the gauge length to a transducer below the furnace and beyond the bottom end of the quartz tube. We have available two extensometers. The JVK model can be clamped directly to the reduced gauge section of the tensile specimens. The other model, manufactured by ATS (Applied Test Systems, Inc.), clamps to the grooves in the shoulders of the specimen. Our experience supports the assumption that essentially all of the deformation between the grooves can be attributed to strain in the 1.25 in. uniform section. Thus, the strain over this gauge length is approximately equal to the measured extension divided by 1.25 in.

The JVK extensometer was developed at Chalk River Nuclear Laboratories in Canada. Our JVK extensometer is shown clamped to a tensile specimen in the center of Fig. 7. The JVK

Fig. 7. Extensometers: ATS (top), JVK (bottom)
(ANL Neg. No. 11581K)

accurately transfers specimen strain to the core of an LVDT (Linear Variable Differential Transformer), located below the furnace, via small diameter lightweight tubes. Knife edges at the top ends of the tubes are clamped to the gauge length of the tensile specimens with leaf springs providing the clamping force. Although the tubes and knife edges are capable of withstanding higher temperatures, annealing of the springs may cause them to lose their gripping force at temperatures in excess of 550°-600°C.

The ATS Series 4112 Extensometer is shown at the top of Fig. 7. This extensometer fastens to the tensile specimens with bolts that clamp hardened inserts into the specimen grooves. The circular inserts are machined with a 90° knife edge that rides on the edges of the groove. This accommodates a certain amount of differential expansion between the specimen and the clamp without allowing slippage. The ATS extensometer is fabricated from MAR-M246 nickel alloy and has a maximum operating temperature of 1000°C, which is well above that for the JVK. The quartz

tube and purge gas system for the Instron furnace have been designed so that either the ATS or the JVK extensometer can be used. In addition, the ATS extensometer will fit into the instrument chamber of the Centorr furnace.

These initial tests were conducted under strain control using either the JVK or the ATS extensometer, with the split tube furnace. In both cases the LVDT transducer used to measure the displacements is below the quartz tube. Thus the transducer and lower part of the extensometer that extends below the quartz tube are subject to the air currents and temperature swings in the room. As a result we found irregularities in the extensometer signals that were eliminated when we enclosed the entire area between the load frame table and the furnace with plexiglass sheets.

F. Computer Control and Data Acquisition

The Instron 8500 series electronic control hardware contains several microprocessors that provide (1) load, position and strain control; (2) wave generation and (3) buffered data collection. These functions are accessed either by pressing keys on the front panel (just to the left of the computer in Fig. 1) or by sending commands from a computer over a communication link. The 8500 instrumentation uses a set of over 150 commands that have either a Q-format for passive requests for status information or a C-format to actively set control parameters. The Instron 8562 machine and the Instron self-adaptive temperature controller both use the GPIB (IEEE-488 General Purpose Interface Bus) communications technology. We use a 386DX microcomputer to control the experiments, collect data and analyze and plot the results. The computer, shown in Fig. 1, is an EVEREX 386 into which we have installed a National Instruments PCIIA board and software for the GPIB communication.

The computer programs used to carry out our test requirements can be either vendor supplied or written by the user. Instron provides two sets of programs: FLAPS-PLUS, which is used for dynamic (fatigue) testing; and SERIES IX, which is used for quasistatic testing of the type prescribed by ASTM test procedures. We used the FLAPS-PLUS program, which operates in the Microsoft Windows environment, for these tests. The FLAPS-PLUS materials testing software is designed to be an extension of the Instron 8500 operator's front panel providing: (i) automatic machine control and data acquisition, (ii) waveform generation, (iii) conditional logic for test

procedures and (iv) posttest graphics and processing facilities. Although FLAPS-PLUS is a powerful general purpose program, we have experienced difficulties using it for our rather simple tests. We have therefore developed a simpler, more robust program that is tailored to our test needs and written in the Visual BASIC language.

III. SPECIMENS

Tensile test specimens have been machined from HT9 rod stock. The 0.343 in.-dia rods were fabricated for Argonne National Laboratory by Carpenter Technology from heat No. 96648. Carpenter Technology's chemical analysis [4] of the alloy is shown in Table I.

TABLE I. Chemical Analysis of HT9 Tensile Specimens

Chemistry (Ingot/Checks)											
	<u>C</u>	<u>Mn</u>	<u>Si</u>	<u>P</u>	<u>S</u>	<u>Cr</u>	<u>Ni</u>	<u>Mo</u>	<u>Al</u>	<u>V</u>	<u>W</u>
INGOT:	0.23	0.47	0.26	0.012	0.004	11.79	0.62	1.03	0.002	0.33	0.50
CHECKS:											
1C	0.20	0.70	0.28	0.013	0.003	11.79	0.61	1.01	0.002	0.32	0.50
1D	0.20	0.69	0.29	0.012	0.003	11.76	0.62	1.02	0.002	0.32	0.50
1X	0.21	0.70	0.29	0.012	0.003	11.82	0.62	1.02	0.003	0.32	0.50
Note: Less than 0.02 Nb; Balance, Fe											

Figure 8 shows the tensile specimen geometry. The 0.220 in. diameter of the uniform gauge section was dictated by the dimensions of the rod stock and the need to cut threads on the ends to attach the specimens to the load train. The 60° grooves near the ends of the specimens were machined to accommodate the ATS extensometer. We decided not to proportion the dimensions according to those of standard ASTM/E-8 tensile specimens because this would have resulted in a smaller diameter in the gauge section and reduced accuracy in the load measurements. Since our primary interest is the stress-strain behavior of HT9 and not the ultimate strength, we are less concerned that the failure of the specimens could occur outside of the gauge section. Furthermore, the high temperatures of our tests will tend to lessen the effects of stress concentrations, which could cause the specimens to fail at locations other than those with the minimum diameter.

All Dimensions are inches

Fig. 8. Tensile Specimen Geometry

The HT9 specimens were fabricated by grinding in the as-received condition. They are heat treated to the condition specified for fuel rod cladding prior to testing: austenitized at 1038°C for 5 min, air cooled, then tempered at 760°C for 30 min and again air cooled. This heat treatment was conducted in a horizontal split tube furnace, within a quartz tube. The tube was alternately evacuated and purged with helium prior to the heat treatment. A flow of helium was maintained during heat treatment to reduce specimen oxidation.

The specimen temperatures were monitored during the heat treatment with a Type K thermocouple spot welded to a shoulder of the specimen. The thermocouple output was displayed on a digital thermometer, and recorded on a strip-chart recorder. Control and uniformity of temperature histories during the anneals were very good. The microstructure of the specimens is believed to have been very uniform prior to tensile testing.

In addition to the HT9 specimens, we have had specimens of identical geometry fabricated from Type 316 stainless steel. The Type 316 stainless steel tensile specimens were used to perform tests to check out the equipment and to compare our results with previous work.

IV. TEST RESULTS

Some preliminary testing was carried out first with a 316SS specimen to test the furnace system and the mechanical testing/computer software systems. Subsequent tests were directed to provide data in the intermediate regime between short-term tensile tests and longer-term creep tests, and to compare results to earlier data in the short-term test regime. Seven tests were completed in the first series, all under computer control using the Instron FLAPS software. A second series of four tests was carried out later under control of our Visual Basic software.

We selected conditions in the mid-range of conditions of interest for the first test. The temperature was 600°C. The effect of temperature below about 400°C is expected to be small, and temperatures above about 700°C are not sustainable with the furnace system used in these tests. The strain rate was selected to result in 10% true strain in about one day. The results of the first test, shown in Fig. 9, indicate some slippage or misalignment of the extensometer during the test. There are several

Fig. 9. Test N901, at 600°C at a Strain Rate of 1.38×10^{-6} per Second for 25 h

possible causes of this behavior, but the most likely is simply that the clamping bolts were not sufficiently tight. There was also one drop in stress that occurred when a software error caused the test to be stopped temporarily. The test was repeated under the same conditions, but with the ATS extensometer attached more firmly, in the N902 test. The results are shown in Fig. 10. The results were very similar to the results of the N901 test, but without the irregularities.

Fig. 10. Test N902; Same Conditions as Test N901

Figure 11 shows the combined results from both tests. Except for the irregularities, the results are the same. In addition to determining the saturation stress of the HT9 alloy under these test conditions, these first tests also demonstrated the reproducibility of the test results.

The observed saturation stress in the first two tests was considerably higher than would have been predicted by the flow law for this material. Although we had hoped to observe some strengthening during the test, no such increase in flow stress was observed. We therefore selected conditions for the next test to explore the effect of a slower strain rate (1.5×10^{-7} per second) and lower temperature (575°C). These conditions also are in the intermediate regime of interest in our modeling effort. The results of this test are shown in Fig. 12. There are some gaps in the data caused by loss of data buffers at the ends of intermediate segments of the test. The FLAPS software was limited in the number of hours that a test segment can be run, so the test was partitioned into

Fig. 11. Superposition of Results from Tests N901 and N902

Fig. 12. Test N903, at 575°C at a Strain Rate of 1.5×10^{-7} per Second (11 Days)

segments of 96 h. Errors encountered during transfer between segments caused the loss of a buffer of data at the end of each segment, and required the test to be restarted.

We next ran three tests in the more common short-term tensile test regime, to benchmark the behavior of our test material to other data. We applied the same strain rate, 1×10^{-4} per second, for all three tests. The temperatures were held constant at 400, 500, and 600°C for specimens N904, N905, and N907, respectively. The results are combined in Fig. 13, which shows the variation in load with time for the tests. Figure 14 shows the corresponding true stress-true strain results for these tests.

Fig. 13. Load Histories for Tests N904, N905, and N907

The last test in this series was carried out with specimen N908 at a temperature of 650°C and a nominal strain rate of 1.5×10^{-7} per second. The test duration was planned to be 12 days, which required three 4-day test stages. The gaps in the data were caused by the necessity to run the test in multiple segments. The results are shown in Fig. 15.

Fig. 14. Variation of True Stress with True Strain for the Tests Shown in Fig. 13

Fig. 15. Results for Test N908, at 650°C and $1.57 \times 10^{-7} \text{ s}^{-1}$

The temperature for this test was the highest in the first test series. We found it difficult to adjust the control parameters to eliminate fluctuations in load during this test. The parameters were altered several times as the test was continued in segments over the 12-day period. In general, the data appear to fluctuate about a smooth curve, although the stress is somewhat higher than expected in the range of 0.06 to 0.09 true strain.

At the end of this test, several modifications were made to the test procedure. First, the new specimen holders shown in Fig. 4 were designed and fabricated. The holders used in the foregoing tests were badly oxidized, and the N908 sample could not be removed. The new holders were designed in two pieces that can be separated for specimen removal. We also converted to Type N thermocouples, because of the potential for degradation of the accuracy of Type K thermocouples at temperatures near 500°C. The Type N thermocouples were calibrated against a platinum thermocouple (see Appendix A). The Type K thermocouples were not calibrated; instead, we relied on the manufacturer's specifications for "Special Limits of Error" thermocouple wire. We calibrated the Type N wire because it was not available in Special Limits of Error grade. We also purchased a new LVDT for use with the ATS extensometer. This LVDT, obtained from Instron, has a smaller range of ± 100 mils. The LVDT used in the previous series of tests covered a range of ± 500 mils, although it had been calibrated for a reduced range of ± 200 mils. We selected this LVDT to increase the sensitivity of strain control in our tests, and to improve compatibility with the Instron hardware. Calibration of this LVDT is described in Appendix B. Finally, we developed software to replace the FLAPS software. We used the Visual Basic for DOS programming system to develop interactive software to meet our needs. One of the features of this software is that the output file is closed and re-opened each time data is acquired from the Instron system. We particularly wanted to avoid the type of data loss that we had experienced with the FLAPS software.

The first test in the second series was a room-temperature test at a strain rate of 1×10^{-4} per second. The purpose of this test was to provide a further check on the low-temperature mechanical properties of the steel. We also used this test to check out the modified hardware and software. The test was completed in about 40 s. The results for this test, designated N906, are shown in Fig. 16. The maximum true strain with the new LVDT is about 16%.

Fig. 16. Results for Test N906; Room Temperature at a Strain Rate of $1.0 \times 10^{-4} \text{ s}^{-1}$

Fig. 17. Results for Test N909, at 516°C and a Strain Rate of $4.0 \times 10^{-5} \text{ s}^{-1}$

The next two tests in the series were designed to provide data in the transition region between low-temperature behavior and higher-temperature, strain-rate dependent behavior. Test N909 was carried out at 516°C at a strain rate of 4×10^{-5} per second, with a test duration of a little over an hour. The results are shown in Fig. 17.

We increased the temperature to 621°C for test N910, and increased the strain rate to 4×10^{-3} per second. This test was the most rapid that we have run; the test duration was only 40 s. Figure 18 shows the results.

The final test in this second series was planned to further detail the strain behavior in the intermediate regime between tensile tests and creep tests. The results for Test N911 are shown in Fig. 19. The temperature for this test was 700°C, which is the highest temperature that we have used, and is near the upper limit of capability of the current furnace enclosure system. A strain rate of 1.38×10^{-6} per second was maintained over the test duration of 32 h.

Fig. 18. Results for Test N910, at 621°C and Strain Rate of $4.0 \times 10^{-3} \text{ s}^{-1}$

Fig. 19. Results for Test N911 at 700°C and Strain Rate of $1.38 \times 10^{-6} \text{ s}^{-1}$

V. SUMMARY AND CONCLUSION

This report describes the results of a series of 11 mechanical tests of HT9 steel. The uniaxial tensile tests were carried out using an Instron 8500 series digital testing machine, under computer control. None of the specimens failed during the tests.

The experimental conditions were selected to cover a range of conditions of interest in modeling the off-normal behavior of HT9 cladding material. Figure 20 summarizes the tests, characterizing them according to the strain rate and actual test temperature. Selected test results are shown in Table II.

Fig. 20. Summary of Test Conditions

TABLE II. Summary of Test Results

Test ID	Temp, °C	Strain Rate, in./in./s	Test Time	"Peak" True Stress, MPa	Final True Stress, MPa	Ratio, Final:Peak
N901	600	1.4x10 ⁻⁶	25 h	242	251	1.04
N902	600	1.4x10 ⁻⁶	30 h	244	233	0.95
N903	575	1.5x10 ⁻⁷	11 d	263	247	0.94
N904	400	0.0004	4 min	736	837	1.14
N905	500	0.0004	7 min	596	665	1.12
N906	20	0.0004	7 min	957	1129	1.18
N907	600	0.0004	14 min	387	456	1.18
N908	650	1.5x10 ⁻⁷	15 d	107	73.8	<0.69>
N909	516	0.00004	67 min	534	546	1.02
N910	620	0.004	40 s	437	428	0.98
N911	700	1.4x10 ⁻⁶	32 h	105	78.4	<0.75>

The true stress, as shown in the preceding figures, diminishes after reaching a peak value. The decrease is due either to a net reduction in the flow stress (material softening), or to the onset of necking (or at least a nonuniform reduction in cross-sectional area). The peak value of the true stress is thus representative of the flow stress before necking may have begun. The final true stress can be calculated from the final load values and measurements of the specimens after testing. The ratio of these values provides a good indication of whether the specimen strengthens or weakens during the later part of the test. The last column in Table II shows this ratio. Although pronounced necking occurred in only a few samples, none of the specimens showed a reduction in cross-section that was uniform over the entire gauge length. Most of the specimens tapered gradually to a minimum near the middle.

Significant strengthening, of the order of 10% or more (shown in bold in Table II), occurred only in the tests conducted at a strain rate of 0.0004 in./in./s. Only two tests (indicated in the table by <...>), those conducted at the highest temperatures, showed significant reduction in the flow stress during the test. The remaining 5 of the 11 tests showed no significant change.

ACKNOWLEDGMENTS

Helpful discussions, encouragement, and support from J. M. Kramer and R. J. DiMelfi are gratefully acknowledged.

REFERENCES

1. R. J. DiMelfi and J. M. Kramer, [REDACTED]
[REDACTED] Argonne National Laboratory Report, [REDACTED] (April-September 1985).
2. R. J. DiMelfi et al., "Microstructural Evolution in a Ferritic-Martensitic Stainless Steel and Its Relation to High-Temperature Deformation and Rupture Models," Argonne National Laboratory Report, ANL-IFR-176 (August 1992).

3. R. J. DiMelfi and J. M. Kramer, Argonne National Laboratory, personal communication (May 17, 1991).
4. "Certificate of Tests," Carpenter Technology Corporation, Reading, PA, Document sent to D. Erickson, Argonne National Laboratory, September 25, 1986.

APPENDIX A. TEMPERATURE CALIBRATION

A precision digital thermometer, model DP41, and a Type R platinum thermocouple probe were acquired from Omega Engineering, Inc. Together, this probe and thermometer were used as a standard to calibrate the Instron furnace controller system, using Type N thermocouple wire that was also purchased from Omega. The thermocouple probe and wire were provided with "CAL-1" certification that they were manufactured in accordance with instructions and specifications published in Omega Engineering handbooks, and that all materials conform to ANSI standard MC96.1. The digital thermometer was certified (CAL-3 certification) to have been calibrated and tested to meet or exceed the published specifications; the NIST test numbers were provided for reference.

Preliminary tests indicated errors in specimen temperatures within the Instron furnace in the range of -1 to -5°C at temperatures between 300 and 700°C . These errors were generally smaller during heating to a test-temperature plateau, and became larger as the temperature stabilized. In the preliminary tests, however, the results indicated that we had not allowed adequate time for the temperatures to stabilize at their asymptotic values. In addition, a discontinuity was observed in the results at 300°C . The high- and low-temperature portions of the Type R conversion from millivolts to temperature, in the DP41 instrument, were discontinuous at 300°C , causing an abrupt change in error of 9°C when this temperature was traversed. The instrument was returned to Omega for repair. The calibration tests were repeated following the return of the instrument. The preliminary tests had been carried out by heating a stainless-steel cylinder in the furnace chamber, as sketched in Fig. A1. Two Type N thermocouples, one from each end of the wire spool, were spot welded to a stainless-steel bolt that rested inside the cavity in the thermal block. The platinum thermocouple probe was inserted alongside the bolt. The thermal block was modified for the final calibration by slightly enlarging the hole in the center, and inserting a copper cylinder with a central hole for the platinum probe. The Type N thermocouples were spot-welded to heavy steel "foil", which was wrapped around the copper slug and inserted in the thermal block. The influence of heating and cooling on the measured temperature differences was reduced by these modifications, but not entirely eliminated.

Fig. A1. Sketch of Thermal Block

The calibration test was designed to calibrate the entire Instron controller system, complete with the thermocouples to be used during testing (Type N). The controller was set up to use the "primary" thermocouple for control. This thermocouple is a platinum Type R thermocouple probe placed in the wall of the furnace. The Type N thermocouples outputs were monitored with the Instron controller. All times and temperatures were recorded with an Everex PC, using software written for the purpose. The overall results of the calibration test are shown in Fig. A2.

The test was carried out by stabilizing the temperature at successively higher or lower values. During each heating step, the error curve moves upward (toward smaller errors). The opposite effect is observed when cooling is initiated (near 9 and 11 hours, for example).

The temperature was varied by entering the desired "SET" temperature to control the "primary" furnace temperature. A steady temperature was achieved more quickly in the furnace body than in the thermal block, which was suspended inside a quartz tube in a flowing helium atmosphere. The temperature control and stabilization are more clearly illustrated in the expanded plot shown in Fig. A3, which shows the results for the 700°C calibration. Some estimation is required to select the initial set point, to establish the desired calibration temperature. Further, the

Fig. A2. Overall Data from Calibration Test

Fig. A3. Results of Calibration Test at 700°C

set point must be altered slightly to maintain the desired temperature as the overall system temperature rises. The curves shown in Fig. A3 illustrate how quickly the primary temperature reaches the set point, and how the "standard" Type R thermocouple and the two Type N thermocouples follow.

The "error" curve plots the difference E between the standard Type R thermocouple reading and the average of the two Type N thermocouple readings:

$$E = R - \frac{1}{2}(N_1 + N_2).$$

The error varies over a range of 2.5°C during this portion of the test, but becomes stable near -6.5°C as the furnace temperature is held constant.

Similar expanded plots were examined for all temperatures of interest. The results are summarized in Table A1, and the errors are plotted as a function of temperature in Fig. A4. The thermocouple errors are accurate to the limits of error of the digital thermometer ($\pm 0.2^{\circ}\text{C}$) and of the Type R thermocouple ($\pm 1.5^{\circ}\text{C}$ or 0.25% of indicated temperature, whichever is greater). Since the errors from the plots can be estimated within about $\pm 0.1^{\circ}\text{C}$, the maximum error in the tabulated thermocouple errors should be less than $\pm 2^{\circ}\text{C}$.

TABLE A1. Variation of Error
with Temperature

Nominal Temperature, $^{\circ}\text{C}$	Error, $^{\circ}\text{C}$
400	-5.5
500	-5.7
600	-6.0
650	-6.0
675	-6.5
700	-6.5

Fig. A4. Summary of Calibration Results

These error values must be added to the temperatures indicated by the Type N thermocouples. If the indicated temperature is 650°C, for example, the true temperature is 644°C $\pm 2^\circ\text{C}$. In our experiments, these thermocouples are used for control. In this case, if the desired specimen temperature is 650°C, the set temperature for these Type N thermocouples should be 656°C.

APPENDIX B. CALIBRATION OF THE INSTRON 8562 TENSILE TEST EQUIPMENT

The Instron 8562 load cell and position actuator were calibrated by Instron services on March 18, 1993. Certificates of calibration were provided by Instron for these services; copies of the results are shown in Tables B1 and B2, respectively.

The strains during testing of the HT9 tensile specimens are determined by means of an extensometer that measures the deformation within the gauge length. Our test setup requires an extensometer with rods transmitting the displacement in the gauge length to a transducer below the furnace and beyond the bottom end of the quartz tube. We have available two extensometers for high-temperature measurements. The JVK model (developed at Chalk River Nuclear Laboratories in Canada) can be clamped directly to the reduced gauge section of the tensile specimens. This extensometer is clamped with spring-actuated knife edges. Annealing of the springs may cause them to loose their gripping force at temperatures in excess of 550°-600°C. The ATS extensometer (manufactured by Applied Test Systems, Inc.) is clamped into grooves in the specimen shoulders with knife edges secured by bolts, and is less subject to slippage. The ATS extensometer was fabricated from MAR-M246 nickel alloy and has a maximum operating temperature of 1000°C. Current testing is being conducted with the ATS extensometer. Both extensometers use Linear Variable Differential Transformers (LVDT's) to measure extension, and have been calibrated against a micrometer head that was calibrated in March 1993 by the Argonne Inspection Department (SSD-IN). Table B3 is a copy of the certificate issued by SSD-IN for the micrometer-head calibration.

The Schaevitz LVDT (SN 2024) that was used in our 1992 tests was last calibrated in February 1992. The calibration was carried out in a fixture developed to calibrate the LVDT response when mounted in the JVK extensometer. The data obtained in that calibration are plotted in Fig. B1. Although the range of this LVDT is ± 0.5 in., the Instron calibration software was optimized for ± 0.2 in. to improve linearity, sensitivity, and control. The principal source of error is in the LVDT, provided the extensometer is properly attached to the specimen. This calibration should therefore be valid for the tests carried out in 1992, even though the LVDT was used with a different extensometer (the ATS model) in those tests.

Fig. B1. Calibration Results Obtained in February 1992

The base results, shown by the curves labeled "Run#1" and "Run#2", show a maximum difference of 0.1 mil. The "Reverse #1" curve indicates the effect of backlash in the micrometer head. The results differ from the base results by 0.1 to 0.2 mils, with one difference of 0.3 mils. The "Reset" curves show results after shifting the extensometer relative to the micrometer head. Only relative changes in error are significant, since the base value is shifted. All of these results are consistent, and show a maximum deviation of only 0.5 mils over the 200-mil range (except for "Reset #2", which shows a deviation of 0.6 mils).

This LVDT was retested in 1993 with the micrometer head mounted in the calibration fixture, without the use of an extensometer. Tests were carried out several times, with slight changes in the "zero" position. The results of the last test, on September 3, 1993, are shown in Fig. B2. The symbols show the results of three separate test series, with the mean values indicated by the heavy line. The maximum deviation over the 400-mil range of interest has increased from 0.5 mils to 1.7 mils. The reason for this change from the earlier calibration test is not clear. This calibration is the one currently loaded in the Instron software for the Schaevitz LVDT.

Fig. B2. Current Calibration Results for Schaevitz LVDT

A new LVDT (Schlumberger M814330-01) was procured from Instron, in an effort to improve compatibility with the Instron instrumentation system. This LVDT has a smaller range (± 0.1 in.) than the older LVDT (± 0.5 in.), and employs an integral core moved by a spring-loaded plunger that presses against a bearing surface. The calibration fixture was modified so that the LVDT could be mounted in a position similar to that employed during a tensile test. The plunger then rested against the end of the micrometer anvil. Calibration tests were carried out in the "unload" direction, starting with the plunger in the fully inserted position. This direction corresponds to that experienced during a tensile test. The results are shown in Fig. B3.

The maximum variation in error observed in this calibration test was less than 0.7 mils, which is considerably better than the 1.7 mils obtained for the current calibration of the Schaevitz LVDT. However, over the smaller span of ± 100 mils, the maximum deviation for the Schaevitz LVDT was similar. To place the results in perspective, however, it should be noted that errors in extensometer output affect the strain rate in proportion to the slope of the error curve. Thus if the error varies by 1 mil in 100 mils, the error in the strain rate is only 1%. When strain rates are varied by orders of magnitude, this error must be considered negligible.

Fig. B3. Calibration Results for Schlumberger LVDT

TABLE B1. Certificate of Calibration for Instron Load Cell

Page 1

**** LOAD WEIGHING VERIFICATION ****
**** ARGONNE NATL.LAB. ****

INDICATED LOADS: from Machine GPIB
MACHINE: MODEL #8562 SERIAL #HO 604
LOAD CELL: MODEL #2518-601 SERIAL #UK 514 CAPACITY 5620 lb
DATE: 03-18-1993

VERIFICATION PERFORMED WITH MACHINE SYSTEM IN "AS FOUND" CONDITION

**** TENSION RESULTS ****				**** COMPRESSION RESULTS ****			
%	IND LD	STD LD	%ERROR	%	IND LD	STD LD	%ERROR
1	55.524	55.356	0.3029	1	56.743	56.806	-0.1105
2	112.850	112.739	0.0990	2	112.000	112.025	-0.0222
4	223.386	223.376	0.0046	4	223.435	223.460	-0.0114
6	336.981	336.875	0.0316	6	336.911	336.882	0.0086
8	449.054	448.927	0.0283	8	448.952	448.880	0.0160
10	562.033	561.732	0.0537	10	561.952	561.928	0.0043
20	1123.968	1123.580	0.0346	20	1122.459	1121.911	0.0488
40	2247.826	2246.719	0.0493	40	2247.774	2247.486	0.0128
60	3371.841	3370.290	0.0460	60	3371.833	3373.161	-0.0393
80	4495.767	4493.660	0.0469	80	4495.593	4496.684	-0.0243
100	5599.930	5596.565	0.0601	100	5599.674	5603.259	-0.0640

(errors calculated using load values with 6 decimal place precision)

THIS VERIFICATION IS PERFORMED ACCORDING TO ASTM STANDARD E4-89
'Standard Methods of LOAD VERIFICATION OF TESTING MACHINES'

TESTED USING INSTRON STANDARDS ACCORDING TO ASTM E74-91 & TRACEABLE TO NIST(NBS)

STANDARD(S) USED						
RANGE	SERIAL #	DIR(S)	% (S) OF RANGE	LOWER LIMITS (Units=lb)		CALIBRATED
100%	511283	T	1,2	T: 10	C: 10	01/29/93
	396971	T	4,6,8,10,20,40,60,80,100	T: 200	C: 292	10/07/92
	511283	C	1,2,4	T: 10	C: 10	01/29/93

VOLT INDICATOR FOR STANDARD LOAD CELL(S): HBM DK38, SERIAL #27774

VERIFIED BY: DALE NELSON

SIGNED:

TABLE B2. Certificate of Calibration for Instron Stroke Position

**** STROKE POSITION VERIFICATION ****
 **** ARGONNE NATL. LABATORY ****

MACHINE: MODEL # 8562 SERIAL # H0 604 F/S STROKE 2.0 in

**** TENSION RESULTS ****				**** COMPRESSION RESULTS ****			
%	STD POS	MACH POS	%ERR-FULL TRVL	%	STD POS	MACH POS	%ERR-FULL TRVL
10	0.2005	0.2000	-0.01500	10	0.1996	0.2000	0.00039
20	0.4014	0.4000	-0.03499	20	0.3986	0.4000	0.03499
30	0.6021	0.6000	-0.05249	30	0.6020	0.5999	-0.05249
40	0.8024	0.8000	-0.06000	40	0.8044	0.8000	-0.10999
50	1.0020	1.0000	-0.05000	50	1.0050	0.9999	-0.12750
60	1.2048	1.2000	-0.12000	60	1.2070	1.2000	-0.17500
70	1.4031	1.4000	-0.07750	70	1.4112	1.4000	-0.27999
80	1.6016	1.5999	-0.04250	80	1.6154	1.6000	-0.38499
90	1.8008	1.7999	-0.02249	90	1.8162	1.7999	-0.40750
100	1.9998	2.0000	0.00499	100	2.0142	2.0000	-0.35500

STANDARD(S) USED:

STANDARD	SERIAL #	% OF RANGE	CALIBRATED
Boeckeler Indicator	5095	ALL	10/13/92

INSTRON CERTIFIES
 THAT ITS STANDARDS ARE ACCURATE RELATIVE TO NIST(NBS) STANDARDS.

VERIFIED BY: DALE NELSON

DATE: 03-18-1993

SIGNED:

TABLE B3. Certificate of Calibration for Micrometer Head

ARGONNE NATIONAL LABORATORY

SSD-IN

Certificate of Calibration

Date: 5/28/93

page 1 of 1

Instrument Type: 0-1,000 Micrometer Head

ID No 1

SSD-IN Calibration Procedure No 4-128

Traceable Standard(s) 208-82-1596

Nominal Size	Actual Reading	Deviation
.000	.000	
.100	.100	
.200	.20005	+ .00005
.300	.3001	+ .0001
.400	.39995	- .00005
.500	.500	
.600	.6001	+ .0001
.700	.69995	- .00005
.800	.800	
.900	.900	
1.000	1.00005	+ .00005

* Accuracy = ± 0.002 " TRB

- Out of tolerance conditions will be indicated with an asterisk (*)

- Deviations are determined by comparative measurement

- Results are traceable to the National Institute of Standards and Technology (NIST)

Inspected by Vivian Rhodes
(SSD-IN Inspector)

Procedure Approved by Lynne E. Gruber
(Project/Customer)