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DP-MS-89-9

RAPID HEATING TENSILE TESTS OF HIGH-ENERGY-RATE-FORGED 316L STAINLESS STEEL CHARGED WITH HYDROGEN AND TRITIUM*

By

W. C. Mosley

Westinghouse Savannah River Company
Savannah River Site
Aiken, South Carolina 29808

A paper proposed for presentation
IMS-AIME 1989 Fall Meeting
Indianapolis, Indiana
October 1-5, 1989

*This paper was prepared in connection with work done under Contract No. DE-AC09-76SR00001 (now Contract No. DE-AC09-88SR18035) with the U.S. Department of Energy. By acceptance of this paper, the publisher and/or recipient acknowledges the U.S. Government's right to retain a nonexclusive, royalty-free license in and to any copyright covering this paper, along with the right to reproduce and to authorize others to reproduce all or part of the copyrighted paper.

June 20, 1989

DP-MS-89-9

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W. C. MOSLEY
WESTINGHOUSE SAVANNAH RIVER COMPANY
SAVANNAH RIVER LABORATORY
P. O. BOX 616
AIKEN, SOUTH CAROLINA 29802

ABSTRACT

Rapid heating tensile tests of uncharged, hydrogen-charged, and tritium-charged-and-aged stainless steels are being used to determine effects of internal hydrogen and helium on fracture modes and mechanical properties at elevated temperatures. Testing of high-energy-rate-forged (HERF) 316L stainless steel at temperatures up to near 1200°C revealed that internal hydrogen has only slight effects on mechanical properties whereas internal helium from radioactive decay of tritium causes severe embrittlement at temperatures above 500°C.

Derivative Classifier J. P. Howell

DP-MS-89-9

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INTRODUCTION

316L stainless steel is a candidate material for construction of equipment that will be exposed to tritium. This austenitic stainless steel is frequently used in the high-energy-rate-forged metallurgical condition to take advantage of increased strength produced by cold work introduced by the HERF process. Proper design of tritium-handling equipment will require an understanding of how tritium and its decay product, helium-3, affect mechanical properties. This report describes results of elevated-temperature tensile testing of uncharged, hydrogen-charged and tritium-charged and six-months-aged specimens of HERF 316L stainless steel using a special apparatus to heat to test temperatures up to 1200°C within one minute.

SUMMARY

Rapid heating tensile tests of uncharged, hydrogen-charged, and tritium-charged and six-months-aged specimens of HERF 316L stainless steel revealed effects of internal hydrogen and helium on fracture modes and mechanical properties at temperatures up to near 1200°C. Tests of uncharged specimens displayed the normal mechanical behavior and showed how annealing of cold work introduced by the HERF process influenced mechanical properties. Specimens charged with hydrogen exhibited mechanical behaviors very similar to uncharged samples and established that internal hydrogen has only slight effects on mechanical properties. Specimens containing helium-3 from radioactive decay of tritium exhibited severe embrittlement during tests at temperatures above 500°C. Strengths were similar to those of uncharged and hydrogen-charged specimens but ductilities as measured by reduction-in-area and nonuniform elongation at temperatures above 500°C were greatly reduced. Embrittling effects of internal helium changed the failures at elevated temperatures from transgranular ductile (cup-and-cone or plastic attenuation) fractures to mixed-mode shear-type or intergranular brittle fractures. Helium embrittlement was ameliorated somewhat at 720°C by soaking for five minutes and was enhanced at 892°C by a very slow strain rate.

DISCUSSION

Experimental

Round bar specimens were machined from high-energy-rate-forged 316L stainless steel. The gage diameter was 0.11 inch (2.9 mm) and the gage length was 0.87 inch (22 mm). Some specimens were charged with hydrogen by heating them for eight days at 350°C in a vessel pressurized with hydrogen at 1200 psi. Calculations made with a finite-difference program for diffusion of hydrogen into stainless steels indicated that the hydrogen concentration was uniform along the specimen radius at 3.1 cc per cc of metal.[1] Rapid heating tensile testing of these hydrogen-charged specimens was used to determine the effects of internal hydrogen on mechanical properties. Other specimens were charged with a mixture of tritium and deuterium. After charging, these specimens were stored at 0°C to prevent loss of the tritium and to allow ingrowth of helium-3 from the radioactive decay of the tritium. After six months of storage, some of the specimens were removed for testing. Figure 1 shows the calculated profiles of tritium, deuterium and helium-3 concentrations along the specimen radius at the time of testing.[1] Average concentrations of tritium, deuterium and helium-3 at the time of testing were 1.9, 0.6 and 0.3 cc per cc of metal, respectively. Rapid heating tensile testing of these tritium-charged and six-months-aged specimens was used to determine the combined effects of internal hydrogen isotopes and helium-3 on mechanical properties.

Rapid heating tensile tests up to about 1200°C were performed on an Instron tensile testing machine equipped with an environmental chamber connected to an off-gas exhaust system to remove evolved tritium. A quartz lamp heater in the environmental chamber was used to heat the specimens. Temperatures were controlled and monitored with small thermocouples spot-welded to the specimens. In routine tests, specimens were heated in air to the desired test temperatures within about a minute and held at constant temperature (within 20°C) for testing. Routine tests were conducted at an extension rate of 0.5 inch per minute (0.21 mm/sec). Special tests were performed to determine effects of temperature, time at temperature and extension rate on mechanical properties.

Ultimate tensile strength, 0.2% offset yield strength, total elongation, uniform elongation and nonuniform elongation were determined from load-time recordings. Strength values are reported in thousands of pounds per square inch (ksi). Uniform elongation was considered to occur under uniaxial tension up to the point of maximum load where necking usually begins. Necking introduces triaxial stresses that cause nonuniform elongation until failure occurs. Low magnification photography and scanning electron microscopy were used to record images of the fractures. High magnification images of fracture surfaces were recorded with a scanning electron microscope (SEM). Reduction-in-area values were determined from measurements of specimen diameters at the points of fracture made with an optical microscope or from SEM images. Reduction-in-area and elongations are reported as percentages.

Results

Uncharged HERF 316L Stainless Steel

Routine Tests

Routine rapid heating tensile tests were performed on uncharged specimens of HERF 316L stainless steel from room temperature to 1178°C to establish the normal mechanical behavior. Results were as expected for an austenitic stainless steel.[2] All specimens fractured in a ductile manner as shown in Figure 2. Cup-and-cone fractures occurred for tests from room temperature up to 717°C. Extensive necking (plastic attenuation) occurred at higher temperatures. For all routine tests, fracture surfaces exhibited dimpled, transgranular rupture (Figure 3). The size of the dimples generally increased as the test temperature increased.

In tests between 523 and 717°C, serrations were noted in the uniform elongation portions of the load-time recordings. This phenomenon is an indication of dynamic strain-aging (the Portevin-LeChatelier effect) and is caused by repeated stages of yielding and aging during testing.[3]

Figure 4 shows the variations in ultimate tensile strength and offset yield strength with increasing test temperature for uncharged specimens of HERF 316L stainless steel. Between room temperature and 578°C, ultimate tensile strength decreases from 100.9 to 63.1 ksi. Over this same temperature range offset yield strength exhibits an almost parallel decrease with increasing temperature from 61.7 to 30.3 ksi. Between 578°C and 814°C, ultimate tensile strength continues to decrease sharply with increasing test temperature to 37.1 ksi while offset yield strength decreases only slightly to 28.5 ksi (a local maximum of 33.0 ksi actually occurs at 718°C). Above 814°C, values for ultimate tensile strength are only slightly larger than those for offset yield strength and values for both strength parameters decrease to about 5 ksi at 1178°C.

Figure 5 shows the variations in the ductility parameters with increasing test temperature for uncharged specimens of HERF 316L stainless steel. Reduction-in-area values are in the range of 78.1-86.4% and exhibit little variation with increasing test temperature between room temperature and 814°C. At higher test temperatures, values for reduction-in-area approached 100% as extensive necking occurred. Total elongation decreased from 34.0% at room temperature to a minimum value of 21.1% at 665°C and then increased to 40.8% at 1178°C. Such a minimum is a characteristic of austenitic stainless steels.[2] For HERF 316L stainless steel, this minimum corresponds to a transition from predominantly uniform elongation (12.3-26.2%) below about 750°C to predominantly nonuniform elongation (13.1-33.4%) at higher temperatures.

Special Tests

Several special tests on uncharged specimens of HERF 316L stainless steel were performed to determine how heating affects mechanical properties determined in subsequent tests at room temperature and how time at temperature affects mechanical properties at elevated temperatures. Specimens fractured in these special tests are shown in Figure 6. Figure 7 shows images of the fracture surfaces. Results of the special tests along with results expected for comparison routine tests are given in Table 1.

Room Temperature After Five Minutes At 1000°C

Specimens tested at room temperature after heating for five minutes at 1000°C exhibited cup-and-cone failures and dimpled, transgranular fracture surfaces like those tested without heating (Figures 6 and 7). However, heating at 1000°C did cause changes in strength and ductility parameters. There was a small decrease in ultimate tensile strength from 100.9 to 94.1 ksi but a large decrease in offset yield strength from 61.7 to 45.2 ksi. This heating also caused increases in total elongation from 34.0 to 40.9% and in uniform elongation from 26.2 to 31.2%. No significant changes in reduction-in-area or nonuniform elongation were caused by heating at 1000°C for five minutes. Decreases in strength and increases in ductility are effects normally attributed to annealing of cold work introduced by the HERF process.

717°C After Soaking For Five Minutes

A specimen tested after soaking at 717°C for five minutes underwent cup-and-cone failure and exhibited dimpled, transgranular fracture surfaces like a specimen from a routine test at the same temperature Figures 6 and 7). The test after soaking resulted in higher values for ultimate tensile strength (60.1 ksi) and offset yield strength (42.4 ksi) compared to values of 52.4 and 33.0 ksi, respectively, determined in the routine test. However, there were no statistically significant differences in the ductility parameters for the two tests. The increases in the strength values and the unchanged values of the ductility parameters after soaking for five minutes at 717°C are unexpected. Annealing of cold work introduced by the HERF process would be expected to cause decreases in strength and increases in ductility. Further testing will be required to confirm or explain these effects caused by soaking.

907°C After Soaking For Five Minutes

A specimen tested at 907°C after soaking for five minutes underwent extensive necking as observed for specimens from routine tests at 814-1178°C (Figure 6). The fracture surface exhibited large dimples like those observed for specimens fractured in routine tests near the same temperature (Figure 7). Strength values after soaking were not statistically different from those expected for a routine test at the same temperature. The only effect of the soaking at 907°C on ductility was to increase nonuniform elongation to 27.1% compared to an expected value of 22.5%. This increase in nonuniform elongation resulted in a corresponding increase in total elongation from an expected value of 29.5% to 35.4%. The results of this special test at 907°C and the one at 717°C suggest that cold work introduced by the HERF process influences strength and ductility of 316L stainless steel in different ways. Effects of annealing appear to be functions of both temperature and time at temperature.

Hydrogen-Charged HERF 316L Stainless Steel

Routine Tests

Photographs of fractured specimens of hydrogen-charged HERF 316L stainless steel from routine tests are shown in Figure 8. All the specimens failed in a ductile manner. Specimens tested at room temperature, 346 and 631°C exhibited cup-and-cone fractures. Extensive necking (plastic attenuation) occurred for specimens tested at 829 and 1017°C. SEM images of fracture surfaces are shown in Figure 9. All fracture surfaces exhibited dimpled, transgranular rupture. (The specimen tested at 1017°C had been drawn almost to a point which was damaged during subsequent handling. However, a region about 50 micrometers across had dimpled topography.)

Figure 10 shows the variations in strength values with temperature. At room temperature, values of 98.1 and 62.2 ksi were determined for ultimate tensile strength and offset yield strength, respectively. Values of ultimate tensile strength and offset yield strength exhibit parallel decreases with increasing temperature to 62.2 and 32.9 ksi, respectively, for the test at 631°C. Between 631 and 829 °C, ultimate tensile strength decreases significantly to 32.4 ksi while offset yield strength decreases only slightly to 29.3 ksi. For the test at 1017°C, ultimate tensile strength (10.9 ksi) was only slightly higher than offset yield strength(10.0 ksi).

Changes in ductility with temperature as indicated by values of reduction-in-area, total elongation, uniform elongation and nonuniform elongation are shown in Figure 11. Reduction-in-area remains fairly constant at about 80% between room temperature and 631°C but increases to near 100% at 829 and 1017°C. Total elongation, which has values of about 33% for tests at room temperature and 1017°C, has a minimum value of 21.3% for the 631°C test. Uniform elongation decreases from 24.1% to 15.1% between room temperature and 631°C and is the major contributor to total elongation in this temperature range. Between 631 and 829°C, uniform elongation decreases sharply to 4.0% and remains low at 5.1% up to 1017°C. In contrast, nonuniform elongation, which has low values of 5.9-8.8% below 631°C, increases sharply to 19.3% at 829°C and 28.1% at 1017°C and is the main component of total elongation above 829°C.

Load-time recordings for the test at 631°C exhibited serrations in the uniform elongation portions caused by dynamic strain-aging (the Portevin-LeChatelier effect).[3]

Special Tests

No special tests were performed on hydrogen-charged specimens of HERF 316L stainless steel.

Tritium-Charged and Six-Months-Aged Specimens

Routine Tests

Photographs of specimens of tritium-charged and six-months-aged HERF 316L stainless steel fractured in routine tests are shown in Figure 12. Cup-and-cone fractures occurred in tests at room temperature and 418°C. Shear-type fracture occurred in tests at 536 and 643°C. Brittle fracture was observed in specimens tested between 768 and 1075°C. Figure 13 shows scanning electron micrographs of fracture surfaces. Dimpled, transgranular rupture was observed for specimens tested at room temperature and 418°C. The specimen that underwent shear-type failure at 536°C exhibited a mixture of dimpled, transgranular rupture and intergranular rupture. In contrast, the specimen that underwent similar shear-type failure at 643°C exhibited only dimpled, transgranular rupture. Specimens that failed in a brittle manner between 768 and 1075°C exhibited totally intergranular rupture.

Serrations indicative of dynamic strain-aging were noted in the uniform elongation portion of the load-time recording for the test at 536°C but not for tests at higher or lower temperatures.

Figure 14 shows the variations in strength values with temperature. At room temperature, values of 99.5 and 70.2 ksi were determined for ultimate tensile strength and offset yield strength, respectively. Values of ultimate tensile strength and offset yield strength exhibit parallel decreases with increasing temperature to 64.9 and 41.2 ksi, respectively, for the test at 536°C. Between 536 and 768°C, ultimate tensile strength decreases significantly to 40.4 ksi while offset yield strength decreases to 30.9 ksi. For the test at 924°C, ultimate tensile strength (16.4 ksi) was only slightly higher than offset yield strength(16.0 ksi). The two strength values were identical at 16.3 ksi for the test at 1075°C.

Variations in ductility with temperature as indicated by values of reduction-in-area, total elongation, uniform elongation and nonuniform elongation are shown in Figure 15. Reduction-in-area remains fairly constant at about 80% between room temperature and 418°C but decreases sharply with increasing temperature above 418°C to 12% at 924°C. A further small decrease to 9.5% was determined for the 1075°C test. Total elongation, which has a value of 31.0% at room temperature, decreases moderately to 24.1% at 536°C with an intermediate minimum of 22.1% at 418°C. Above 418°C , total elongation decreases sharply with increasing temperature to 2.5% at 1075°C. Uniform elongation displays a trend with increasing temperature similar to that of total elongation. The value for the test at 536°C of 18.5% is moderately lower than the value of 23.8% determined for room temperature tests and there is an intermediate minimum of 17.3% at 418°C. Above 418°C, uniform elongation decreases sharply with increasing temperature to 1.3% at 1075°C. Nonuniform elongation decreases slightly from 7.2% at room temperature to 4.8-5.6% at 418-768°C and then decreases sharply to 1.2-1.3% at 924-1075°C.

Special Tests

Results of special tests on specimens of tritium-charged and six-months-aged HERF 316L stainless steel are given in Table 2. Figure 16 shows the fractured specimens and SEM images of their fracture surfaces are shown in Figure 17.

Room Temperature After Heating At 1000°C

Specimens tested at room temperature after heating to 1000°C exhibited ductile cup-and-cone fractures (Figure 16). The fracture surfaces (Figure 17) showed dimpled, transgranular rupture but the dimples were very small. Heating to 1000°C and immediately cooling to room temperature caused only a slight decrease in ultimate tensile strength from 99.9 to 94.7 ksi but a significant decrease in offset yield strength from 70.2 to 55.2 ksi. Soaking for five minutes at 1010-1036°C caused further slight decreases to 94.4 and 52.6 ksi for ultimate tensile strength and offset yield strength, respectively. Heating to 1000°C had no statistically significant effects on any of the ductility parameters but soaking for five minutes at 1010-1036°C caused a small increase in uniform elongation from 23.8 to 29.5% and a possible slight increase in nonuniform elongation from 7.2 to 9.0% corresponding to an increase in total elongation from 31.0 to 38.5%.

720°C After Soaking For Five Minutes

A specimen tested at 720°C after a five minute soak failed in a ductile manner with a cup-and-cone fracture (Figure 16) in contrast to the shear-type fracture observed for the routine test at 643°C and the brittle-type fracture observed for the routine test at 768°C (Figure 12). The dimpled, transgranular fracture surface had dimples about the same size as those observed for specimens tested at room temperature without heating (Figure 13). The five minute soak at 720°C had significant effects on strengths and reduction-in-area but not on any of the elongation parameters (Table 2). This special test resulted in values for ultimate tensile strength (55.4 ksi) and offset yield strength (43.4 ksi) that are moderately higher than values of 46 and 33 ksi, respectively, expected from routine tests without the soak. The value for reduction-in-area (78.4%) is considerably larger than the 39% that would be anticipated for a routine test at the same temperature. However, the 15.7, 9.3 and 6.4% values for total, uniform and nonuniform elongation, respectively, are as anticipated for a 720°C routine test.

893°C After Soaking For Five Minutes

A specimen tested at 893°C after a five minute soak failed in a brittle manner (Figure 16) with intergranular rupture (Figure 17) and had tensile property values (Table 2) as expected for a routine test at this temperature. Since this brittle failure is attributed to the internal helium, the failure of the five minute soak to cause any effects shows that the kinetics of the helium embrittlement phenomenon at 893°C are rapid.

892°C At A Very Slow Strain Rate Of 0.005 Inch/Minute

In a special test at the very slow strain rate of 0.005 inch/minute at 892°C, the specimen exhibited brittle failure (Figure 16) with intergranular rupture (Figure 17) as observed for other specimens from routine and special tests at temperatures above 768°C. Values for ultimate tensile strength (12.4 ksi), offset yield strength (11.3 ksi) and reduction-in-area (9.0%) were significantly lower than values anticipated for a routine test (Table 2). Elongation values were not affected, probably because they were already so low. This special test indicates that a low strain rate enhances helium embrittlement.

Comparisons of Results

Effects of internal hydrogen and helium on mechanical behavior of HERF 316L stainless steel were discerned by comparing results for routine and specials tests of uncharged, hydrogen-charged and tritium-charged and six-months-aged specimens. Figures 18 through 23 compare results for specific mechanical properties determined in routine rapid heating tensile tests.

Comparisons Of Results Of Routine Tests Of Hydrogen-Charged Specimens With Those For Uncharged Specimens

Failure Mode

The ductile fractures (Figure 8) and the transgranular, dimpled rupture surfaces (Figure 9) observed for hydrogen-charged specimens were almost identical to those observed for uncharged specimens (Figures 2 and 3). Thus, internal hydrogen had no noticeable effect on the fracture mode.

Strain-Aging

Dynamic strain-aging in hydrogen-charged HERF 316L stainless steel occurred in a test at 631^{O}C (indicated by serrations in the uniform elongation portion of the load-time recording) which is within the 523-717^{O}C temperature range where it was observed for uncharged specimens. This result indicates that internal hydrogen does not influence dynamic strain-aging (the Portevin-LeChatelier effect).[3]

Offset Yield Strength

The decrease in offset yield strength with increasing temperature is the same for both hydrogen-charged and uncharged specimens as shown in Figure 18. The higher value of 43.8 ksi for the hydrogen-charged specimen tested at 346^{O}C compared to 38.5 ksi for the uncharged specimen tested at 315^{O}C may be statistically significant and represent a strengthening effect of hydrogen.

Ultimate Strength

Figure 19 shows a comparison of ultimate tensile strength versus temperature for hydrogen-charged and uncharged HERF 316L stainless steel. For room temperature tests, the slightly lower value of 98.1 ksi for hydrogen-charged specimens compared to 100.9 ksi for uncharged specimens is considered statistically significant. In tests at higher temperatures, the decrease in ultimate tensile strength with increasing temperature is the same for both hydrogen-charged and uncharged specimens.

Reduction in Area

Figure 20 shows a comparison of reduction-in-area versus temperature for hydrogen-charged and uncharged specimens. Between room temperature and 600^{O}C, values for reduction-in-area determined for hydrogen-charged specimens were generally several percent lower than values determined for uncharged specimens. Although standard deviations are probably on the order of several percent (based on multiple determinations at room temperature), these differences may be real and represent a decrease in ductility caused by the hydrogen. The increase in reduction-in-area to near 100% for tests above 800^{O}C appears to be nearly identical for both hydrogen-charged and uncharged specimens.

Elongation

Variations in total elongation, uniform elongation and nonuniform elongation with increasing temperature for hydrogen-charged and uncharged specimens are shown in Figures 21, 22 and 23, respectively. Hydrogen appears to have no statistically significant influence on any of these ductility parameters over the temperature range of the tests.

Comparisons of Results of Routine Tests Of Tritium-Charged And Six-Months-Aged Specimens With Those For Uncharged and Hydrogen-Charged Specimens

Failure Mode

Effects of internal helium on fracture mode were determined by comparing photos of fractured tritium-charged and six-months-aged specimens (Figure 12 and 13) with similar photos for uncharged specimens (Figure 2 and 3) and hydrogen-charged specimens (Figure 8 and 9). Tritium-charged and six-months-aged specimens of HERF 316L stainless steel tested at room temperature and 418°C failed in a ductile manner with cup-and-cone fractures in the same manner as observed for uncharged and hydrogen-charged specimens tested in this temperature range. Fracture surfaces of all three types of specimens exhibited indistinguishable dimpled, transgranular fracture. Thus, internal helium appears to have no affect on fracture mode at temperatures between room and 418°C. However, different behaviors occurred at elevated temperatures. Tritium-charged and six-months-aged specimens failed by shear at 500-700°C and by brittle rupture above 700°C while uncharged and hydrogen-charged specimens exhibited cup-and-cone fracture to above 700°C and extensive necking (plastic attenuation) above 800°C. Uncharged and hydrogen-charged specimens had dimpled, transgranular fracture surfaces while the tritium-charged and six-months-aged specimens that failed by shear exhibited a mixture areas with dimpled, transgranular fracture and intergranular fracture and those that failed by a brittle mode exhibited only intergranular fracture surfaces. These different fracture modes observed for tritium-charged and six-months-aged specimens tested at elevated temperatures are attributed to embrittling effects of the internal helium.

Strain-Aging

Dynamic strain-aging detected for a tritium-charged and six-months-aged specimen tested at 536°C occurred in the temperature range where this phenomenon was detected for uncharged and hydrogen-charged specimens. Thus, internal helium appears to have no effect on dynamic strain-aging.

Offset Yield Strength

Figure 18 compares offset yield strength versus test temperature for tritium-charged and six-months aged HERF 316L stainless steel with results for uncharged and hydrogen-charged specimens. The values for tritium-charged and six-months-aged specimens between room temperature and 600°C are consistently 5-10 ksi higher than those determined for uncharged and hydrogen-charged specimens over this temperature range. These differences may represent a small strengthening effect caused by internal helium in addition to a similar small strengthening effect attributed to internal hydrogen as suggested by the results for hydrogen-charged specimens. Above 700°C, all three types of specimens exhibited essentially identical decreases in offset yield strength with increasing temperature. Thus, neither internal helium nor internal hydrogen appear to influence offset yield strength above 700°C.

Ultimate Tensile Strength

Routine rapid heating tensile testing of uncharged, hydrogen-charged and tritium-charged and six-months-aged specimens yielded essentially identical variations of ultimate tensile strength with temperature from room temperature to above 1000°C as shown in Figure 19. No effects of internal helium are indicated by these results.

Reduction-in-Area

Comparisons of reduction-in-area versus test temperature shown in Figure 20 reveal the pronounced effect that internal helium in the tritium-charged and six-months-aged specimens has on this ductility property of HERF 316L stainless steel at elevated temperatures. Internal helium apparently inhibits the necking process and causes the sharp decrease in reduction-in-area with increasing temperature exhibited by tritium-charged and six-months-aged specimens tested above 418°C.

Elongation

Figures 21, 22 and 23 compare the temperature-related variations in total elongation, uniform elongation and nonuniform elongation determined from routine tests of uncharged, hydrogen-charged, and tritium-charged and six-months-aged specimens. These comparisons show that internal helium has no significant effect on elongation from room temperature to about 600°C. Above 600°C, internal helium influences primarily nonuniform elongation. This nonuniform component, which normally increases sharply with increasing temperature above 600°C and gives rise to the characteristic minimum in total elongation, is prevented from doing so by the presence of internal helium. Values of nonuniform elongation, which normally exceed 20% above 900°C, fall to less than 2% when internal helium is present. A secondary effect of internal helium is observed for uniform elongation at temperatures above 800°C where normally low values of 5-8% are reduced to 1-2%. Thus, the reduction in total elongation above 600°C results from the combined effects of internal helium on both the uniform and nonuniform components.

Comparisons of Results of Special Tests Of Tritium-Charged and Six-Months-Aged Specimens With Those For Uncharged Specimens

Ductile, cup-and-cone failures and dimpled, transgranular fracture surfaces were observed for specimens of both uncharged (Figures 6 and 7) and tritium-charged and six-months-aged (Figure 16 and 17) HERF 316L stainless steel tested at room temperature after heating at 1000°C. The much smaller dimple size for the tritium-charged and six-months-aged specimens is attributed to the internal helium. Decreases in strength values and increases in values of ductility parameters for the tritium-charged and six-months-aged specimen (Table 2) determined in room temperature tests after heating for five minutes near 1000°C are like those determined for similar special tests of uncharged specimens (Table 1) and are attributed to annealing of cold work introduced by the HERF process. The higher value for offset yield strength (52.6 ksi) for the tritium-charged and six-months-aged specimen compared to corresponding value for the uncharged specimen of 45.2 ksi is attributed to a strengthening effect of the internal helium. Values of all the ductility parameters for the tritium-charged and six-months-aged specimen are slightly lower than those for the uncharged specimen and represent a slight embrittling effect of the internal helium.

Comparison of results for the tritium-charged and six-months-aged specimen tested after five minutes at 720°C (Table 2) and the uncharged specimen tested after five minutes at 717°C (Table 1) reveals very similar behavior which is unexpected. Both underwent ductile cup-and-cone failure (Figures 6 and 16) and had dimpled, transgranular fracture surfaces but the dimples for the tritium-charged and six-months-aged specimen (Figure 17) were smaller than those for the uncharged specimen (Figure 7). Both specimens had higher values of ultimate tensile strength and offset yield strength than companion specimens from routine tests at the same temperature. These increases in strength caused by the five minute soaks are unexpected since annealing of cold work in a metal usually causes strength values to decrease. This phenomenon does not appear to be related to internal helium since it happened for both types of specimens. It may be related to partial annealing of cold work introduced by the HERF process. Soaking for five minutes at 720°C resulted in a much higher value of 78.4% for reduction-in-area for the tritium-charged and six-months-aged specimen compared to the 39% expected for a routine test at that temperature. This increase in the value of reduction-in-area correlates with a change in fracture mode from shear or brittle to cup-and-cone. In contrast, soaking for five minutes at 717°C caused no statistically significant change in the value of reduction-in-area (79.1-82.9%) for uncharged specimens and the fracture mode remained cup-and-cone. Values of the elongation parameters for both types of samples were unchanged by soaking for five minutes. These special tests at 720°C have revealed a complicated interaction between the cold work introduced by the HERF process and internal helium introduced by tritium decay that is a function of time at temperature. The 720°C test temperature falls in the transition range where extensive necking (plastic attenuation) normally begins to occur, nonuniform elongation begins to dominate over uniform elongation, annealing of cold work is beginning, and internal helium may become mobile. Soaking appears to ameliorate most of the embrittling effects of internal helium at this temperature.

Comparison of results of a special test in which a tritium-charged and six-months-aged specimen was tested at 893°C after soaking for five minutes (Figures 16 and 17)) with a similar test of an uncharged specimen at 907°C (Figures 6 and 7) revealed the same brittle and ductile failures, respectively, as observed or expected for routine tests at these temperatures. Soaking for five minutes caused no statistically significant changes in values of the tensile parameters of the brittle, tritium-charged and six-months-aged specimen but did increase the value of nonuniform elongation for the ductile, uncharged specimen from 22.5 to 27.1% with a corresponding increase in the value of total elongation from 29.5 to 35.4%. The increases in values of these elongation parameters for the uncharged specimen caused by the five minute soak are attributed to annealing of cold work from the HERF process. Similar annealing of cold work may have occurred during the five minute soak of the tritium-charged and six-months-aged specimen but the effects are masked by the embrittlement caused by internal helium.

CONCLUSIONS

Uncharged Specimens

Uncharged specimens of HERF 316L stainless steel exhibited mechanical behaviors typical of an austenitic stainless steel in routine rapid heating tensile tests from room temperature to 1178°C. Specimens failed in a ductile manner with cup-and-cone fractures from room temperature to 717°C and plastic attenuation at higher temperatures. Values of ultimate tensile strength and offset yield strength generally decreased with increasing test temperature. Both exhibited almost parallel decreases with increasing test temperature from room temperature to about 600°C. Between 600°C and 800°C, ultimate tensile strength decreased sharply while offset yield strength remained almost constant with increasing test temperature. Above 800°C, ultimate tensile strength values were only slightly higher than those for offset yield strength and both decreased with increasing test temperature. Variations in values of ductility parameters with increasing test temperature were influenced by necking of the specimens. Values of reduction-in-area, which were almost constant from room temperature to 717°C, increased sharply with increasing test temperature at higher temperatures. Total elongation exhibited a characteristic minimum value near 700°C corresponding to a transition from predominately uniform elongation at lower temperatures to predominately nonuniform elongation at higher temperatures.

In special tests, lower values of ultimate tensile strength and offset yield strength and higher values of total and uniform elongation determined at room temperature after heating for five minutes at 1000°C compared to values determined at room temperature without heating are attributed to annealing of cold work introduced by the HERF process. However, soaking for five minutes at 717°C before testing unexpectedly produced higher values for ultimate tensile strength and offset yield strength and essentially the same values for the ductility parameters compared to values determined for a specimen tested without soaking. This unusual effect is attributed to an intermediate stage of annealing of cold work. Annealing of cold work during a five minute soak at 907°C resulted in higher values for total and nonuniform elongation compared to values expected for a specimen tested without soaking. Thus, effects of annealing of cold work from HERF processing of 316L stainless steel appear to be functions of both temperature and time at temperature.

Hydrogen-Charged Specimens

Routine rapid heating tensile tests of hydrogen-charged specimens of HERF 316L stainless steel yielded results very similar to those determined for uncharged specimens. Internal hydrogen appears to have only slight effects on both strength and ductility parameters. Statistically significant effects attributed to internal hydrogen include a slight decrease in the value for ultimate tensile strength at room temperature, a slight increase in the value for offset yield strength near 350°C, and a small decrease in values for reduction-in-area between room temperature and 600°C.

Tritium-Charged and Six-Months-Aged Specimens

The primary effect of internal helium from radioactive decay of tritium in HERF 316L stainless steel was to reduce ductility during routine testing at elevated temperatures. Instead of cup-and-cone and plastic attenuation failures as observed for uncharged and hydrogen-charged specimens, tritium-charged and six-months-aged specimens underwent mixed-mode, shear-type failures between 536 and 643°C and intergranular, brittle failures at and above 768°C. Embrittlement was manifested in greatly decreased values for reduction-in-area above 418°C and for nonuniform elongation above 643°C. Internal helium apparently interferes with the necking process which influences these two ductility parameters. Thus, embrittlement may involve interactions between the internal helium and the complex triaxial stress state that arises when necking starts. Therefore, helium embrittlement may involve notch sensitivity. Routine rapid heating tensile tests also revealed several secondary effects attributed to the presence of internal helium. A small increase in offset yield strength from room temperature to 600°C was noted for tritium-charged and six-months-aged specimens.

Special tests revealed that cold work introduced by the HERF process and internal helium introduced by the radioactive decay of tritium affect tensile parameters in complex ways that are functions of temperature, time at temperature and strain rate. While routine tests at temperatures above 418°C revealed severe embrittlement caused by internal helium, a similar helium-bearing specimen heated for five minutes at 1000°C retained its ductility in subsequent tests at room temperature. The indication is that helium embrittlement occurs at elevated temperatures during straining. At 720°C, helium embrittlement was ameliorated somewhat by soaking for five minutes before testing. At 892°C, embrittlement by internal helium was enhanced by a very slow strain rate.

FUTURE STUDIES

Rapid heating tensile tests will be performed on additional tritium-charged specimens of HERF 316L after they have aged for 18 and 36 months to allow higher concentrations of helium-3 to form. These tests should reveals how the embrittlement effects are influenced by helium concentration. Samples of several of the six-months-aged specimens have been submitted for helium analyses to confirm calculations of concentrations made by the finite-difference program for diffusion of hydrogen into stainless steels.[1]

ACKNOWLEDGEMENTS

Initial stages of sample preparation and equipment development were supervised by P. E. Zapp, K. E. Kain-Slaughter and A. K. Birchenall. The rapid heating tensile testing equipment was developed by L. J. Harpring and E. R. Selden. M. J. Morgan calculated the tritium, deuterium and helium-3 profiles in the tensile bars. Special appreciation is express to R. L. Cone for assembly and checkout of the equipment and performance of the tests.

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1. K. E. Kain, Finite-Difference Program for Hydrogen Diffusion, DP-1738, March 1987.

2. J. D. Whittenberger and M. V. Nathal, Elevated/Low Temperature Tension Testing, Metals Handbook,Ninth Edition, Volume 8. (American Society for Metals, Metals Park, Ohio, 1985), pp34-37.

3. G. E. Dieter, Mechanical Metallurgy. (McGraw-Hill, New York, 1970).

TABLE 1. Results of Special Tests on Specimens of Uncharged HERF 316L Stainless Steel

TESTED AT ROOM TEMPERATURE AFTER INDICATED HEATING

PROPERTY	5 MINUTES at 1000 ^{o}C	
Ultimate Strength, ksi	94.1	(100.9)
Offset Yield Strength, ksi	45.2	(61.7)
Reduction-in-Area, %	84.0	(82.4)
Total Elongation, %	40.9	(34.0)
Uniform Elongation, %	31.2	(26.2)
Nonuniform Elongation, %	9.7	(7.8)

TESTED AFTER SOAKING AT TEMPERATURE FOR 5 MINUTES

PROPERTY	717 ^{o}C		907 ^{o}C	
Ultimate Strength, ksi	60.1	(52.4)	23.3	(23.5)
Offset Yield Strength, ksi	42.4	(33.0)	18.1	(20.0)
Reduction-in-Area, %	79.1	(82.9)	99.2	(98.8)
Total Elongation, %	20.3	(21.6)	35.4	(29.5)
Uniform Elongation, %	10.9	(12.3)	8.3	(7.0)
Nonuniform Elongation, %	9.4	(9.3)	27.1	(22.5)

Values in parentheses are expected for routine tests at indicated temperatures.

TABLE 2. Results of Special Tests on Specimens of Tritium-Charged and Six-Months-Aged HERF 316L Stainless Steel

TESTED AT ROOM TEMPERATURE AFTER INDICATED HEATING

PROPERTY	HEATED TO 1000 ^{o}C	5 MINUTES 1010-1036 ^{o}C
Ultimate Strength, ksi	94.7 (99.5)	94.4 (99.5)
Offset Yield Strength, ksi	55.2 (70.2)	52.6 (70.2)
Reduction-in-Area, %	75.9 (78.0)	76.6 (78.0)
Total Elongation, %	32.5 (31.0)	38.5 (31.0)
Uniform Elongation, %	26.0 (23.8)	29.5 (23.8)
Nonuniform Elongation, %	6.5 (7.2)	9.0 (7.2)

TESTED AFTER SOAKING AT TEMPERATURE FOR 5 MINUTES

PROPERTY	720 ^{o}C	893 ^{o}C
Ultimate Strength, ksi	55.4 (46)	22.5 (21)
Offset Yield Strength, ksi	43.4 (33)	20.5 (19)
Reduction-in-Area, %	78.4 (39)	15.6 (16)
Total Elongation, %	15.7 (14)	4.7 (5)
Uniform Elongation, %	9.3 (8)	3.5 (3)
Nonuniform Elongation, %	6.4 (6)	1.2 (2)

TESTED AT A STRAIN RATE OF 0.005 INCH/MINUTE

PROPERTY	892 ^{o}C
Ultimate Strength, ksi	12.4 (21)
Offset Yield Strength, ksi	11.3 (19)
Reduction-in-Area, %	9.0 (16)
Total Elongation, %	5.3 (5)
Uniform Elongation, %	3.1 (3)
Nonuniform Elongation, %	2.2 (2)

Values in parentheses are expected for routine tests at indicated temperature.

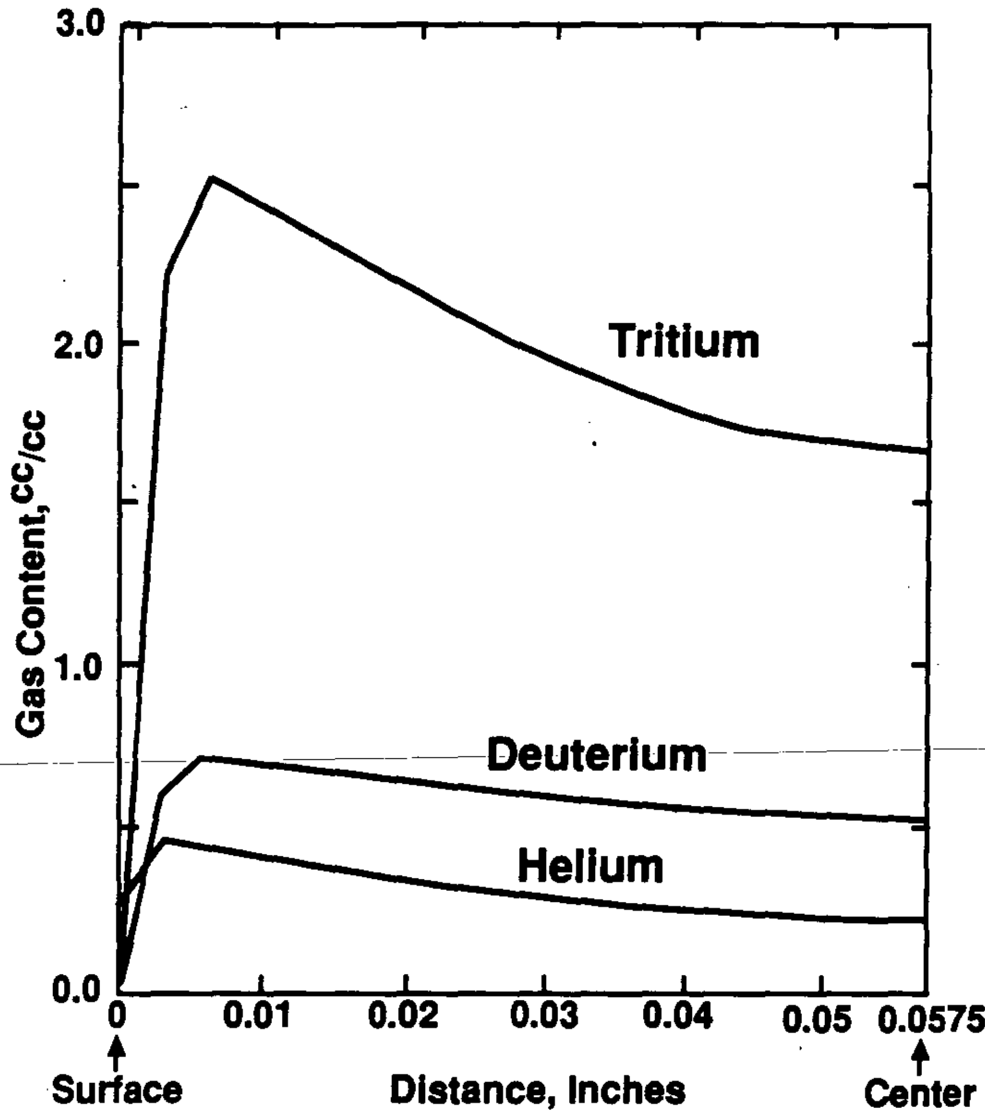


FIGURE 1. Calculated Concentration Profiles for Tritium, Deuterium and Helium-3 in Specimens of Tritium-Charged and Six-Months-Aged HERF 316L Stainless Steel

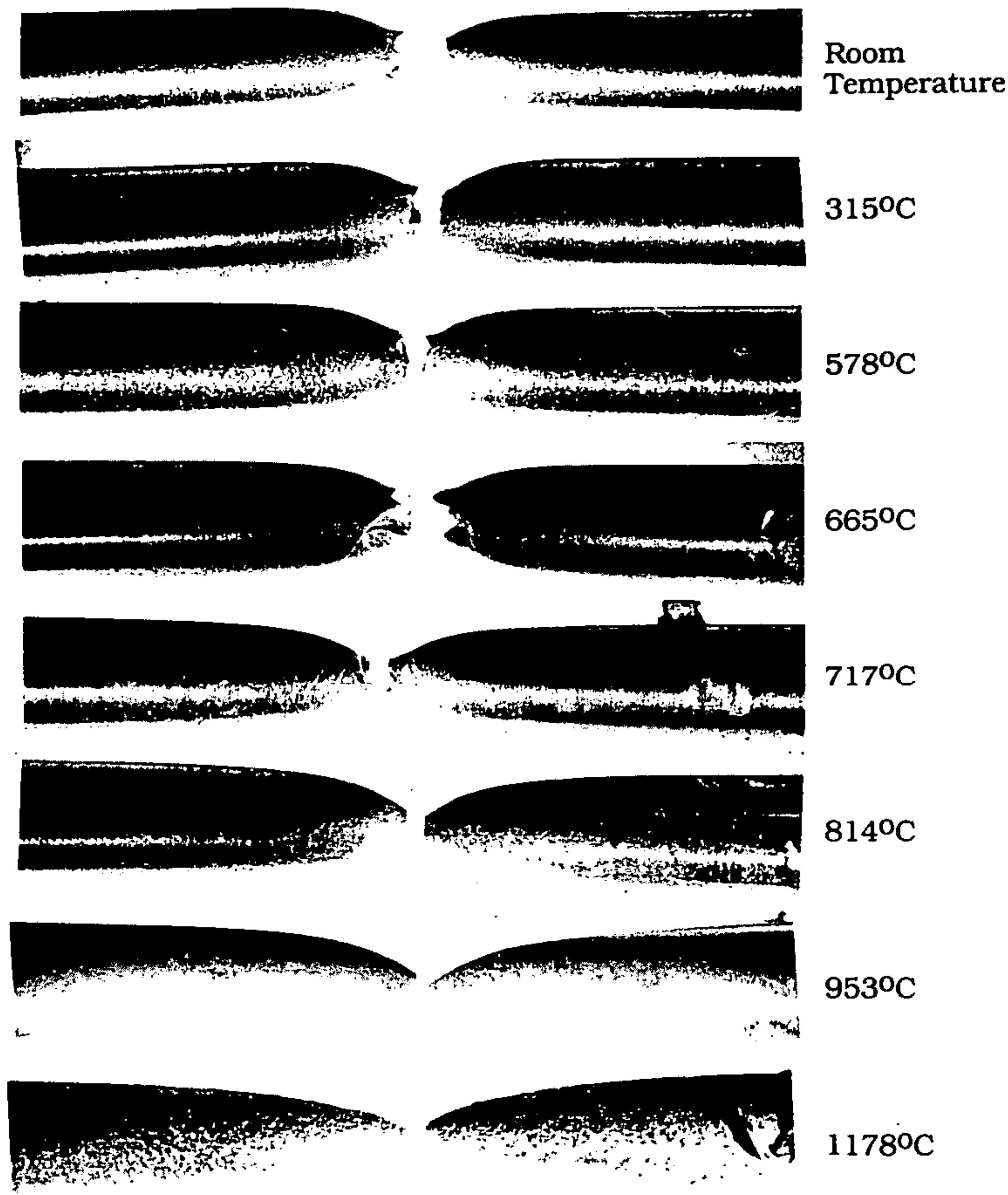
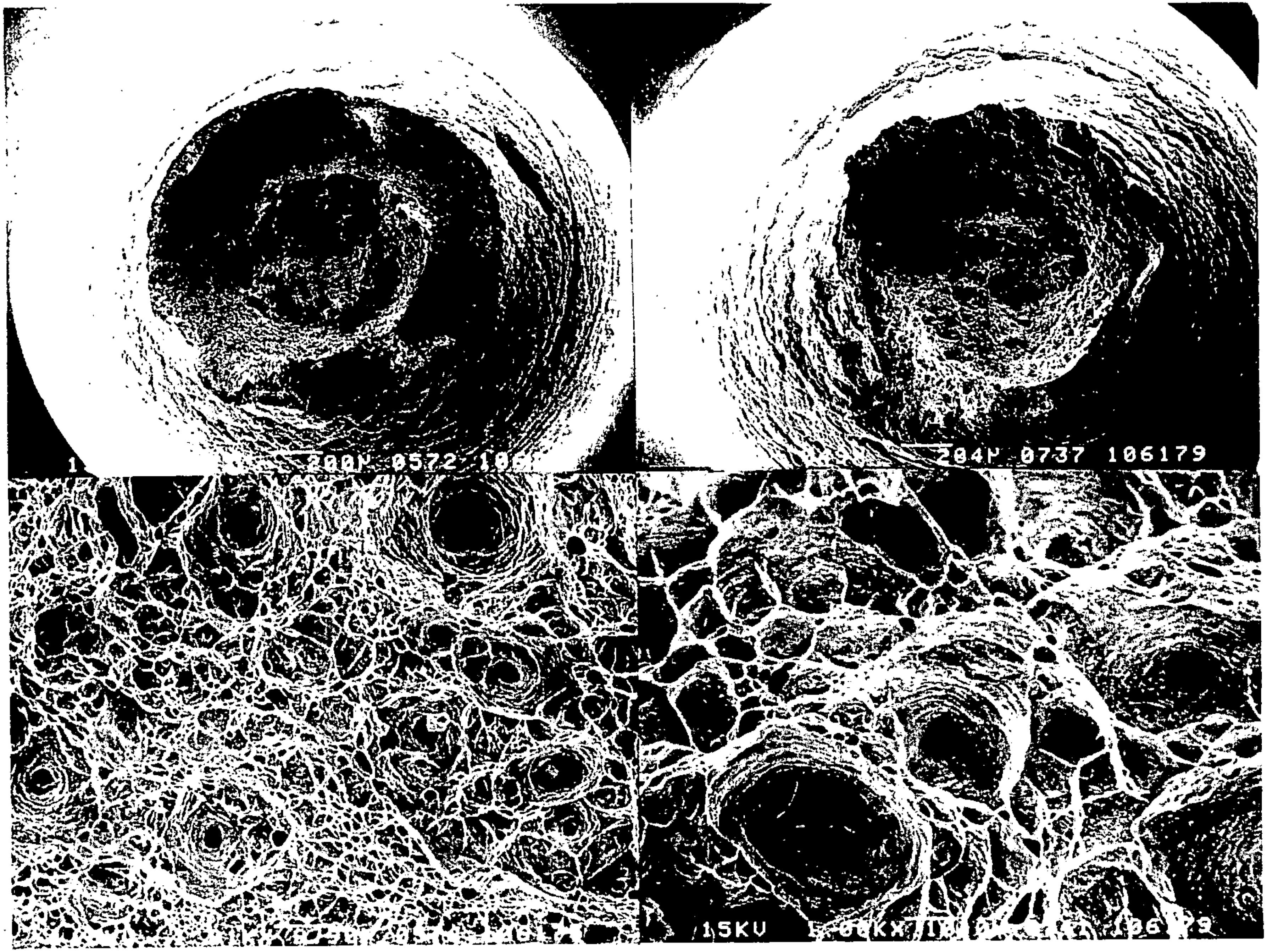


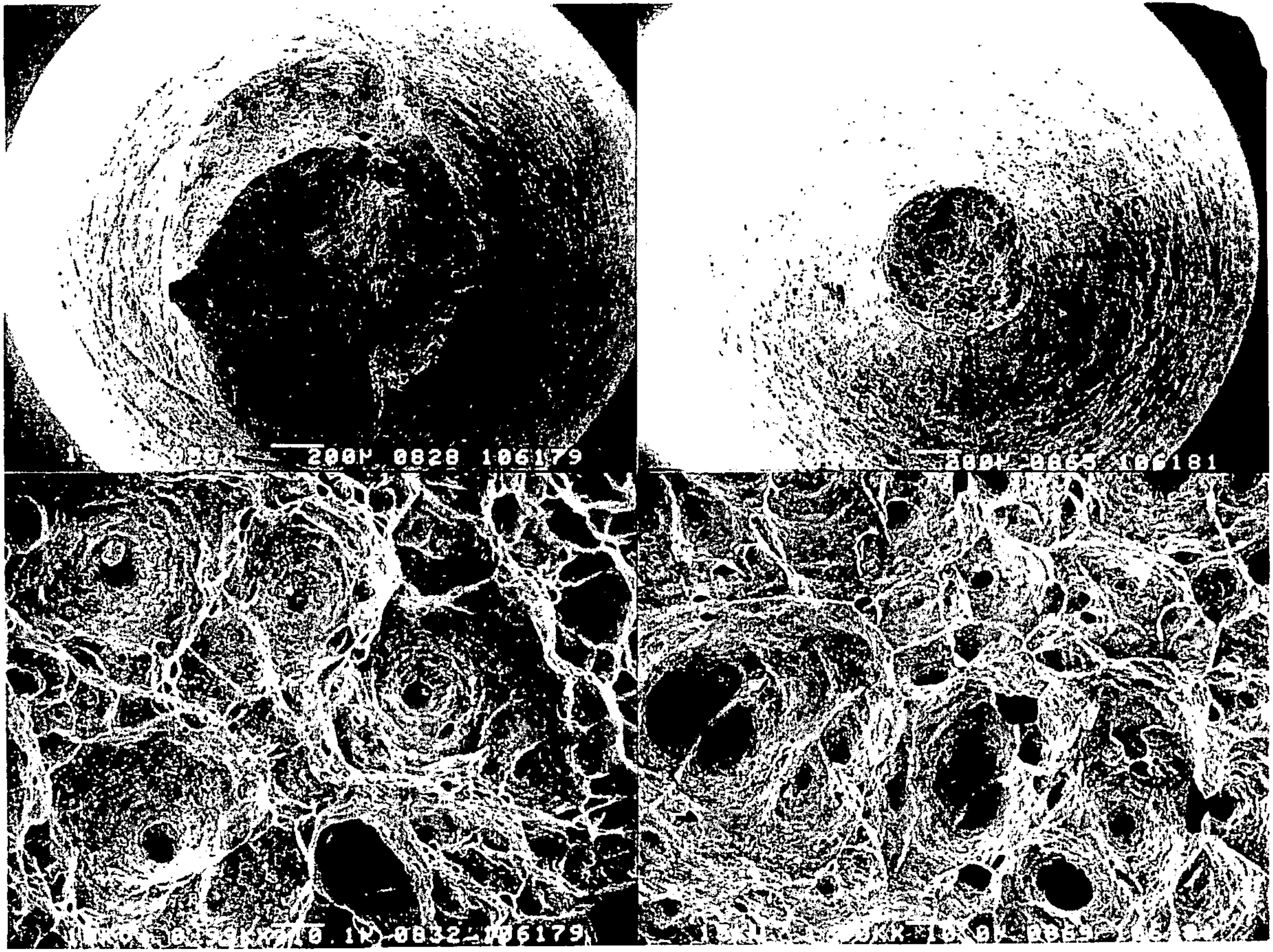
FIGURE 2. Specimens of Uncharged HERF 316L Stainless Steel from Routine Rapid Heating Tensile Tests



Room Temperature

578^{0}C

FIGURE 3. Fracture Surfaces of Specimens of Uncharged HERF 316L Stainless Steel from Routine Rapid Heating Tensile Tests



717°C

814°C

FIGURE 3 (Continued). Fracture Surfaces of Specimens of Uncharged HERF 316L Stainless Steel from Routine Rapid Heating Tensile Tests

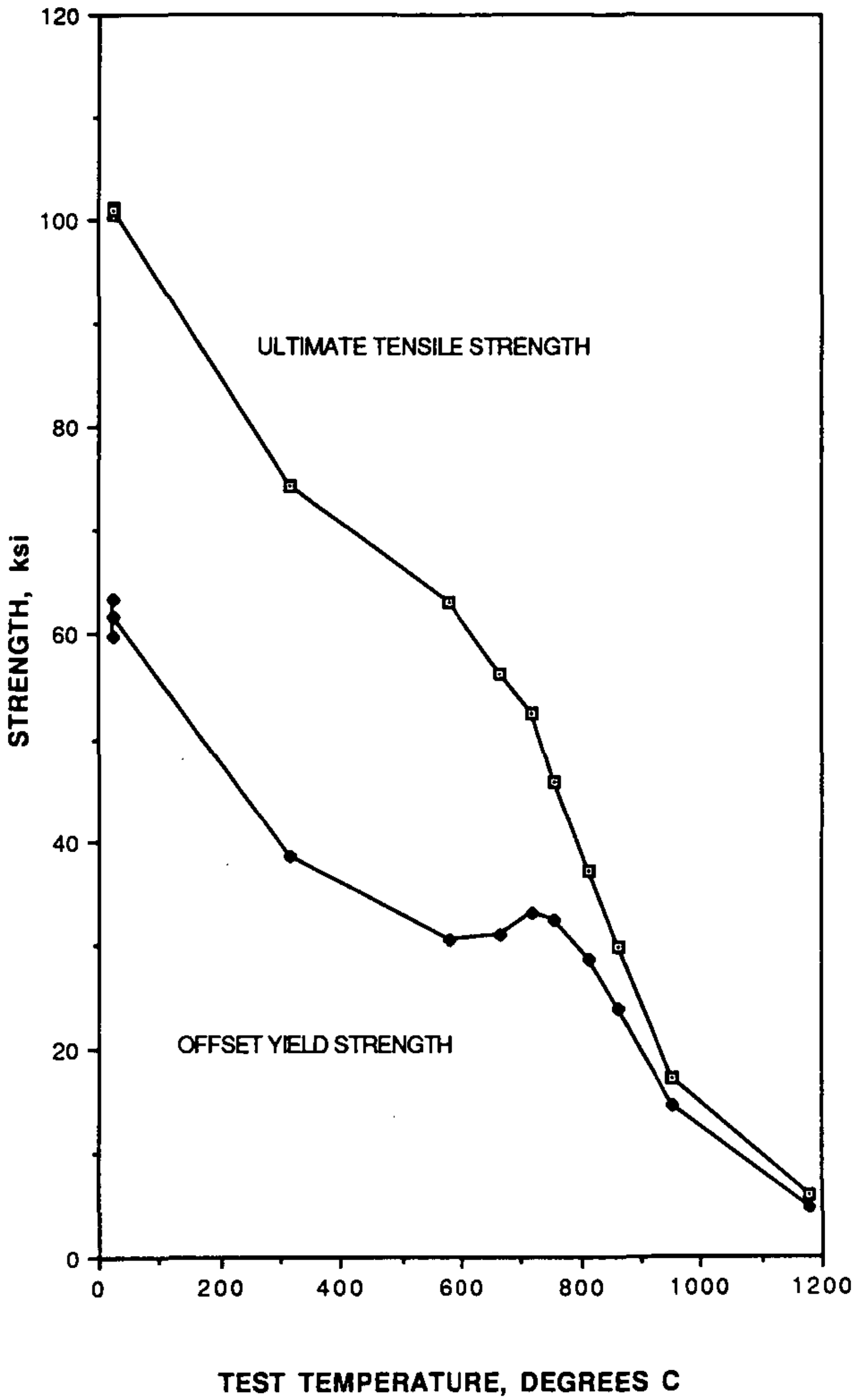


FIGURE 4. Strength Versus Temperature Determined in Routine Rapid Heating Tensile Tests of Uncharged Specimens of HERF 316L Stainless Steel

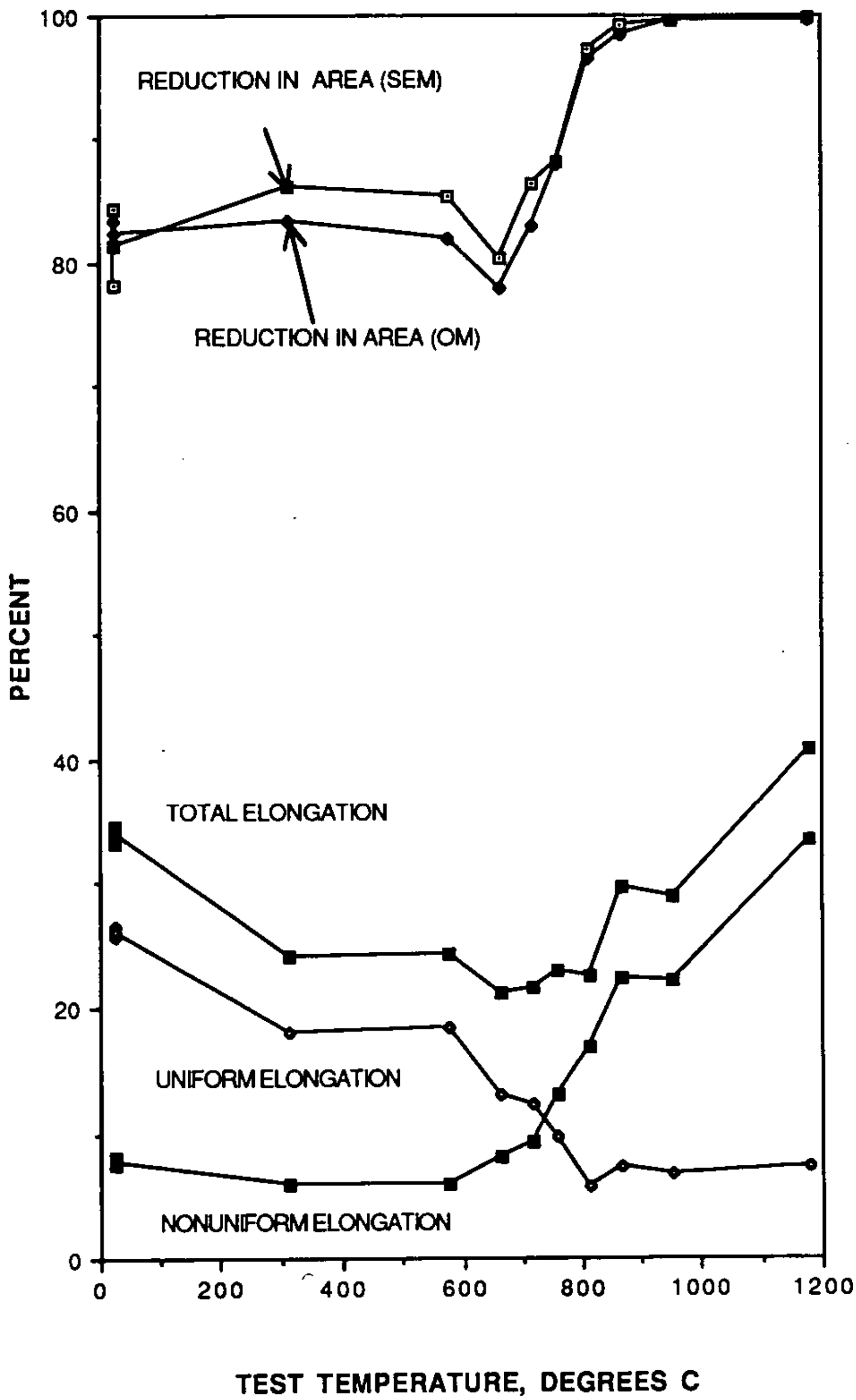
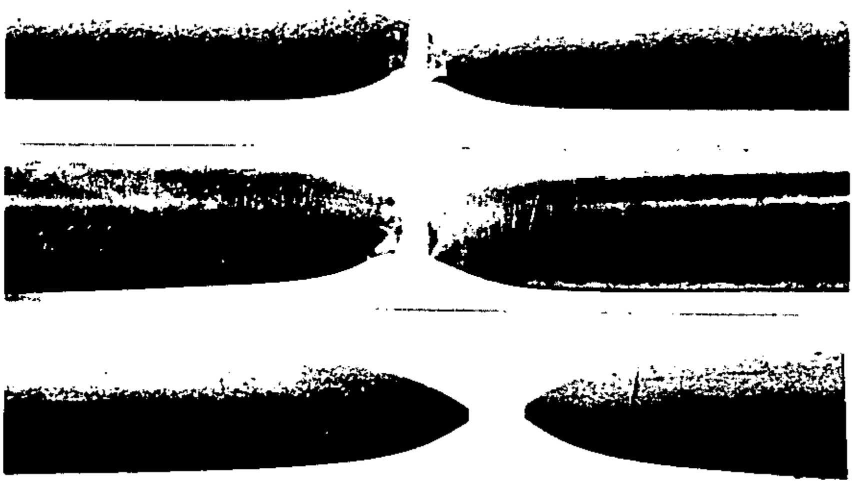


FIGURE 5. Ductility Versus Temperature Determined in Routine Rapid Heating Tensile Tests of Uncharged Specimens of HERF 316L Stainless Steel

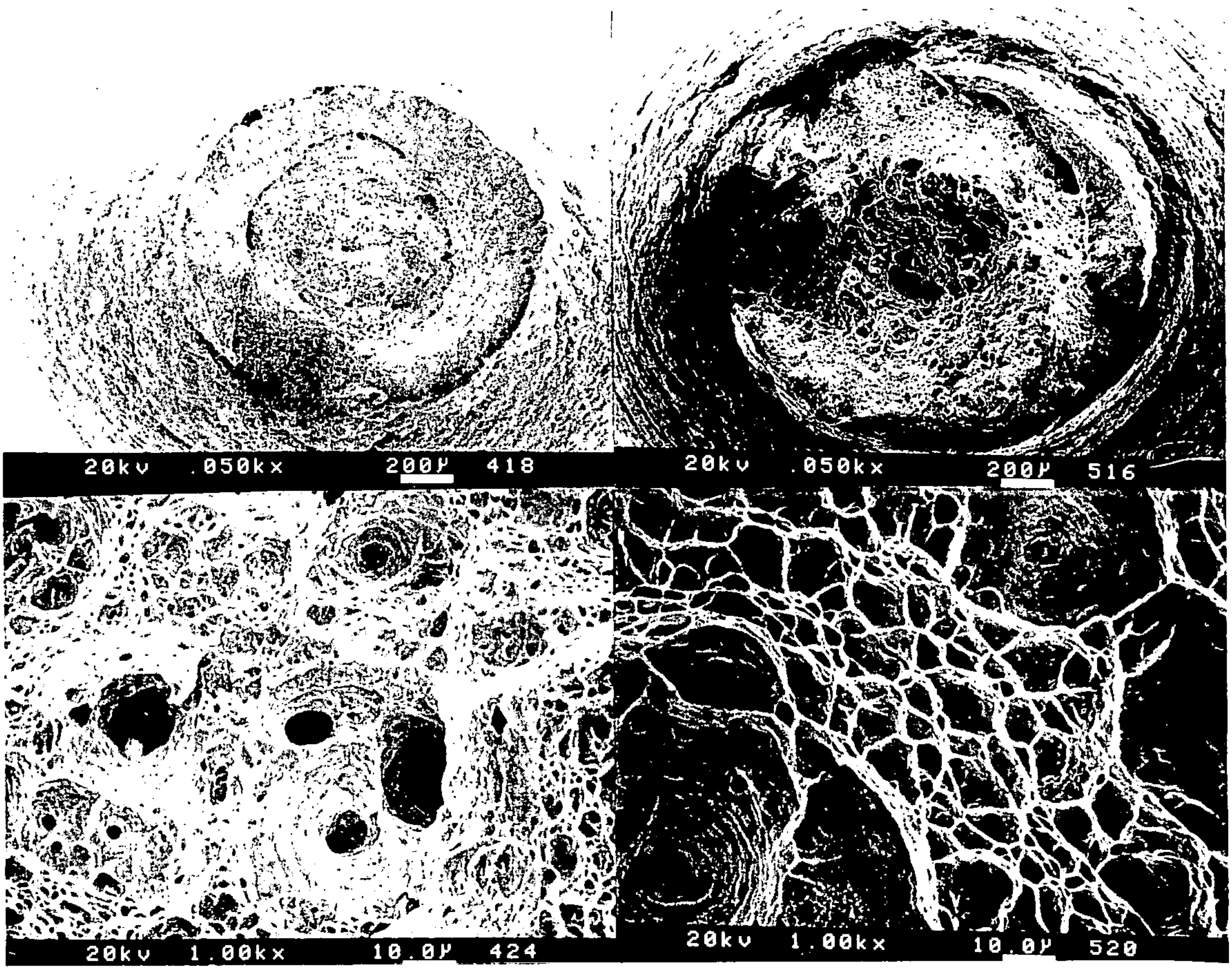


Room Temperature After Soaking For 5 Minutes at 1000°C

717°C After Soaking For 5 Minutes

907°C After Soaking For 5 Minutes

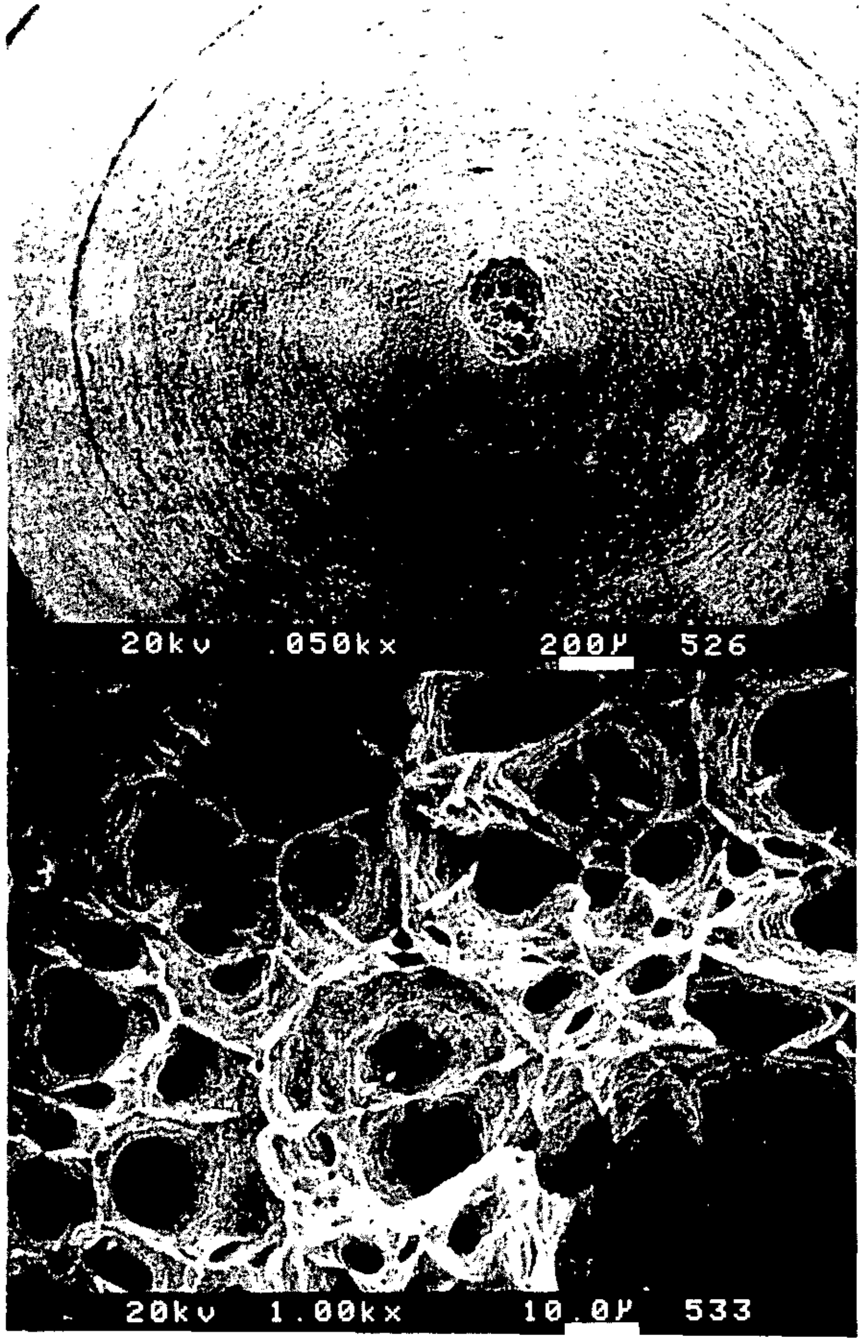
FIGURE 6. Specimens of Uncharged HERF 316L Stainless Steel from Special Tests



Room Temp. After 5 Minutes at 1000°C

717°C After 5 Minute Soak

FIGURE 7. Fracture Surfaces of Specimens of Uncharged HERF 316L Stainless Steel from Special Tests



907°C After 5 Minute Soak

FIGURE 7 (Continued). Fracture Surfaces of Specimens of Uncharged HERF 316L Stainless Steel from Special Tests

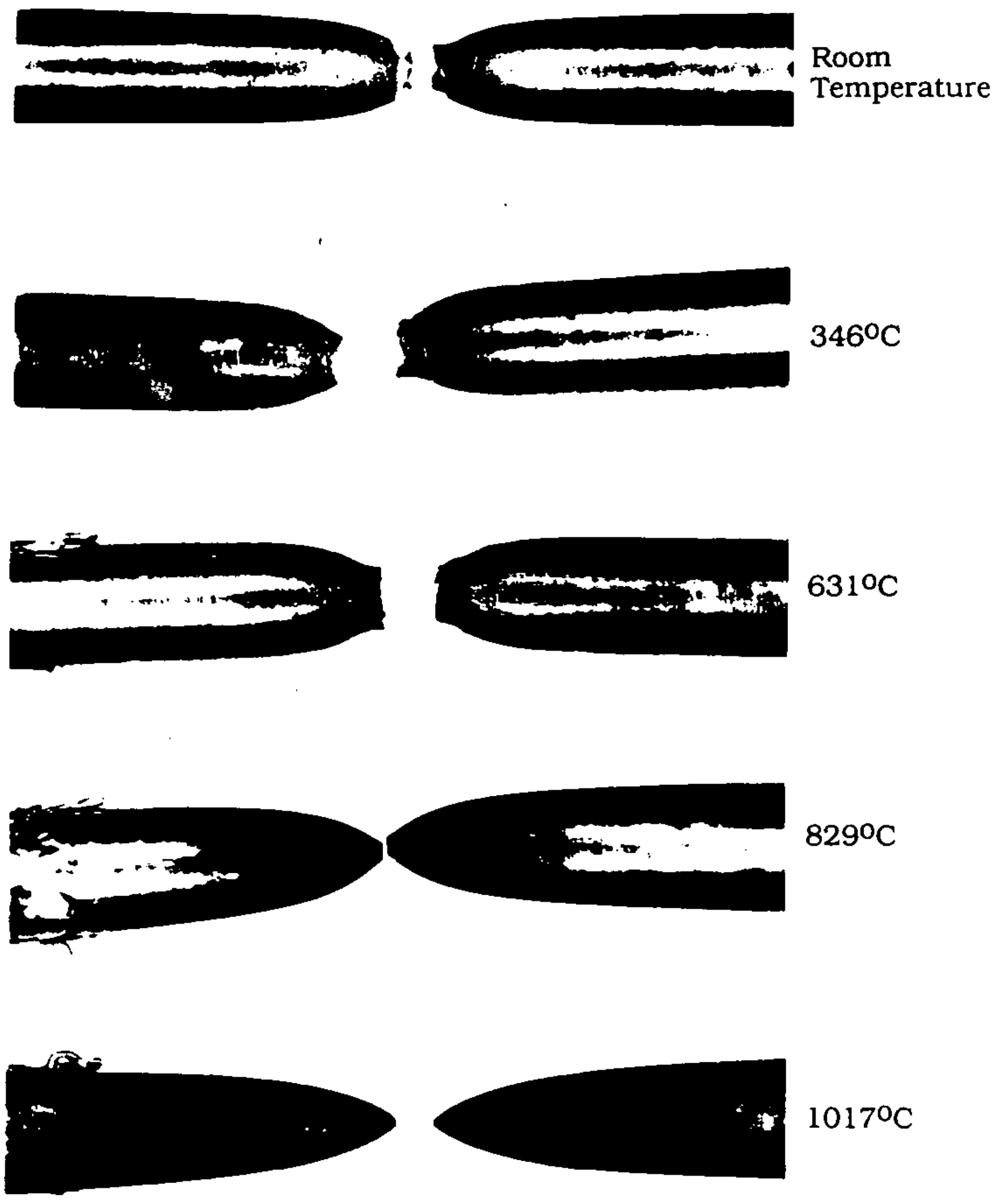
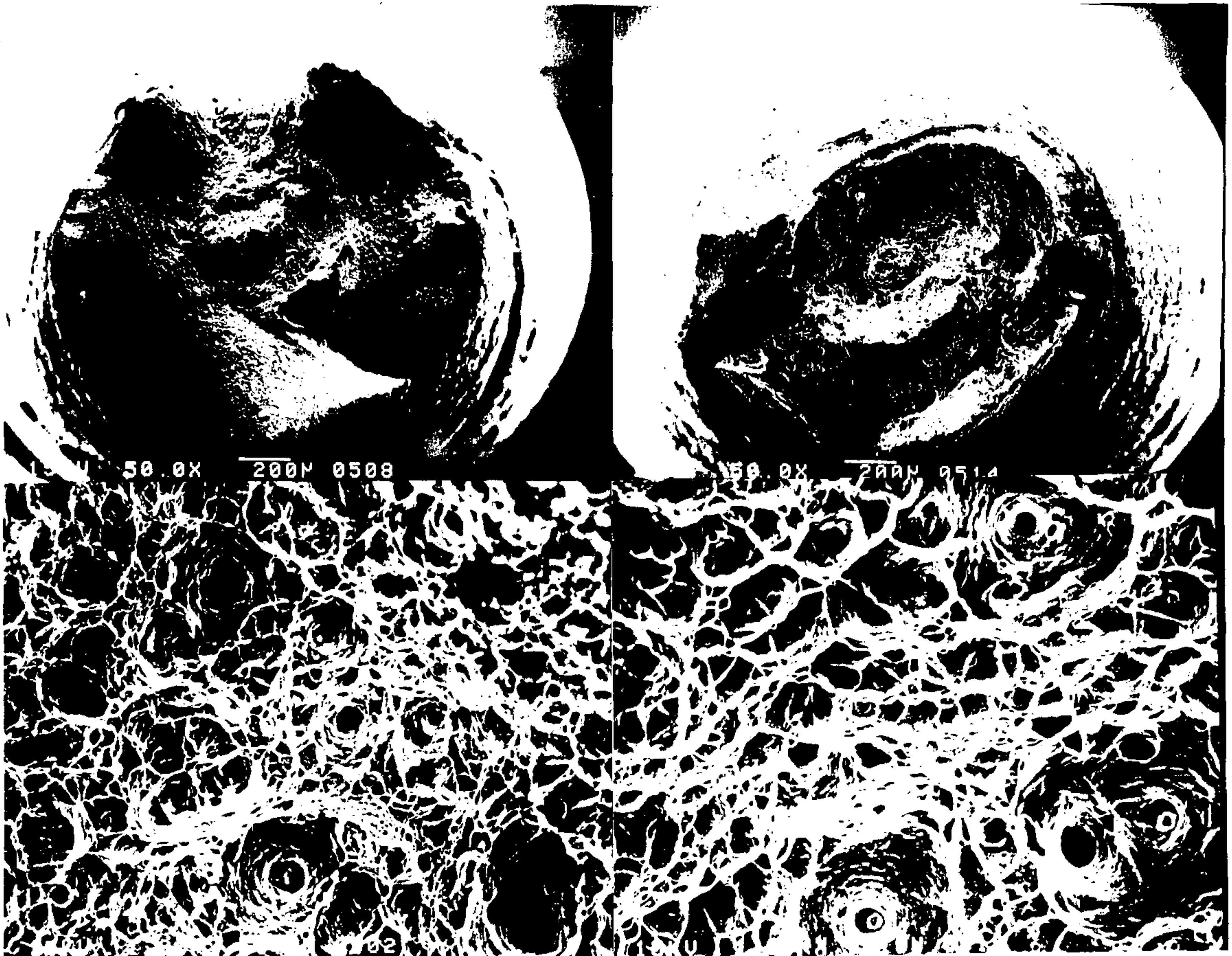


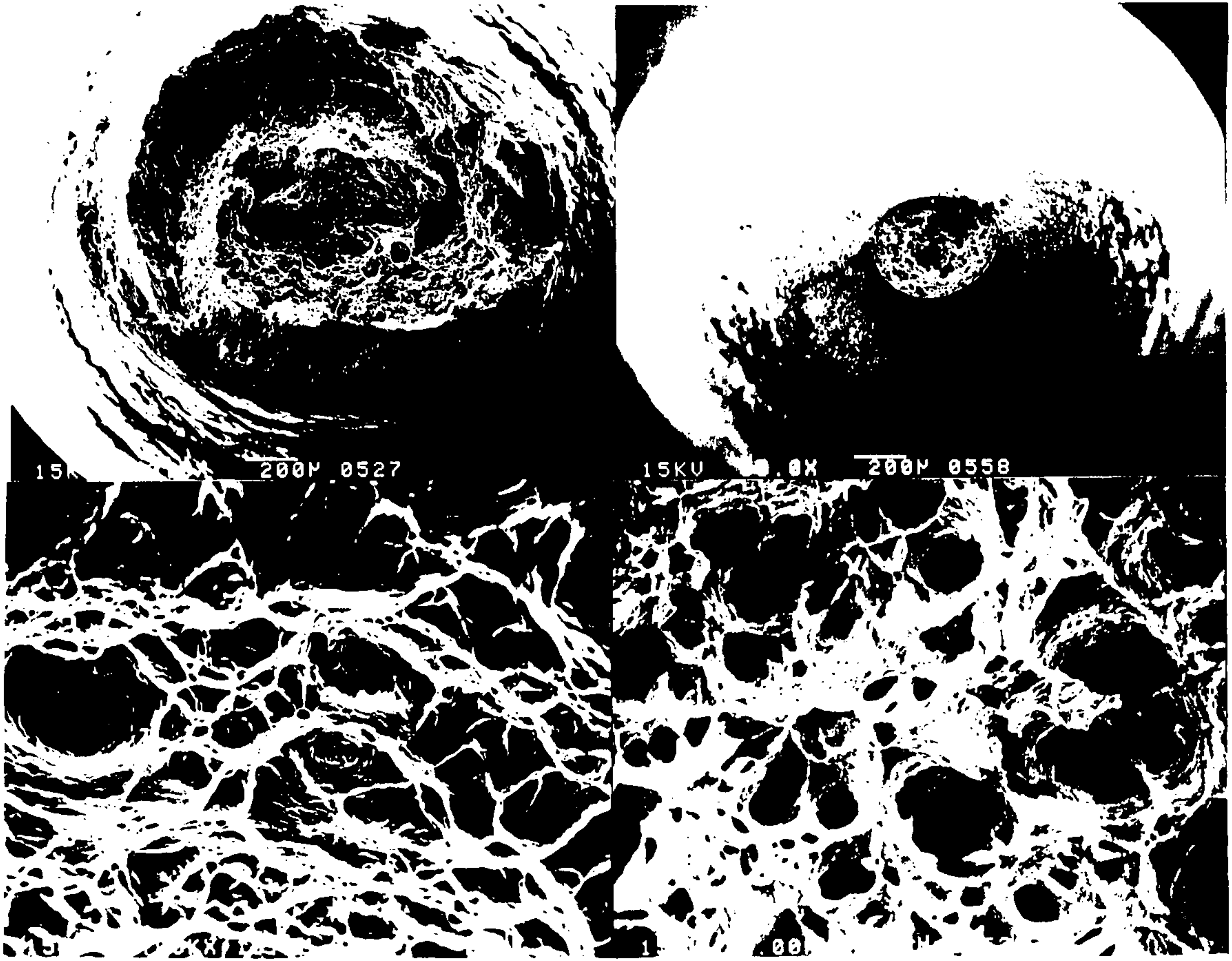
FIGURE 8. Specimens of Hydrogen-Charged HERF 316L Stainless Steel from Routine Rapid Heating Tensile Tests



Room Temperature

346°C

FIGURE 9. Fracture Surfaces of Specimens of Hydrogen-Charged HERF 316L Stainless Steel from Routine Rapid Heating Tensile Tests



631°C 829°C

FIGURE 9 (Continued). Fracture Surfaces of Specimens of Hydrogen-Charged HERF 316L Stainless Steel from Routine Rapid Heating Tensile Tests

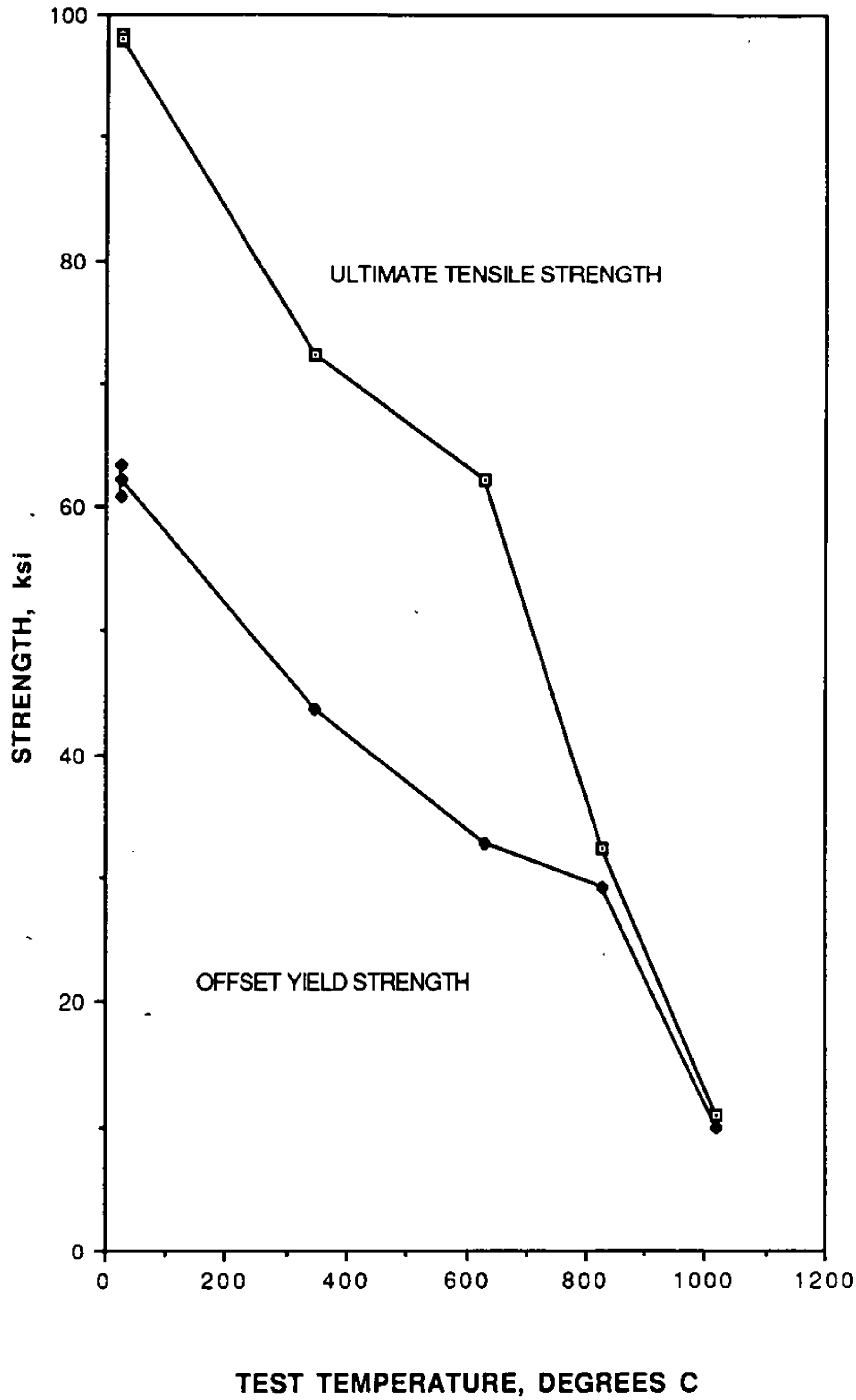


FIGURE 10. Strength Versus Temperature Determined in Routine Rapid Heating Tensile Tests of Hydrogen-Charged Specimens of HERF 316L Stainless Steel

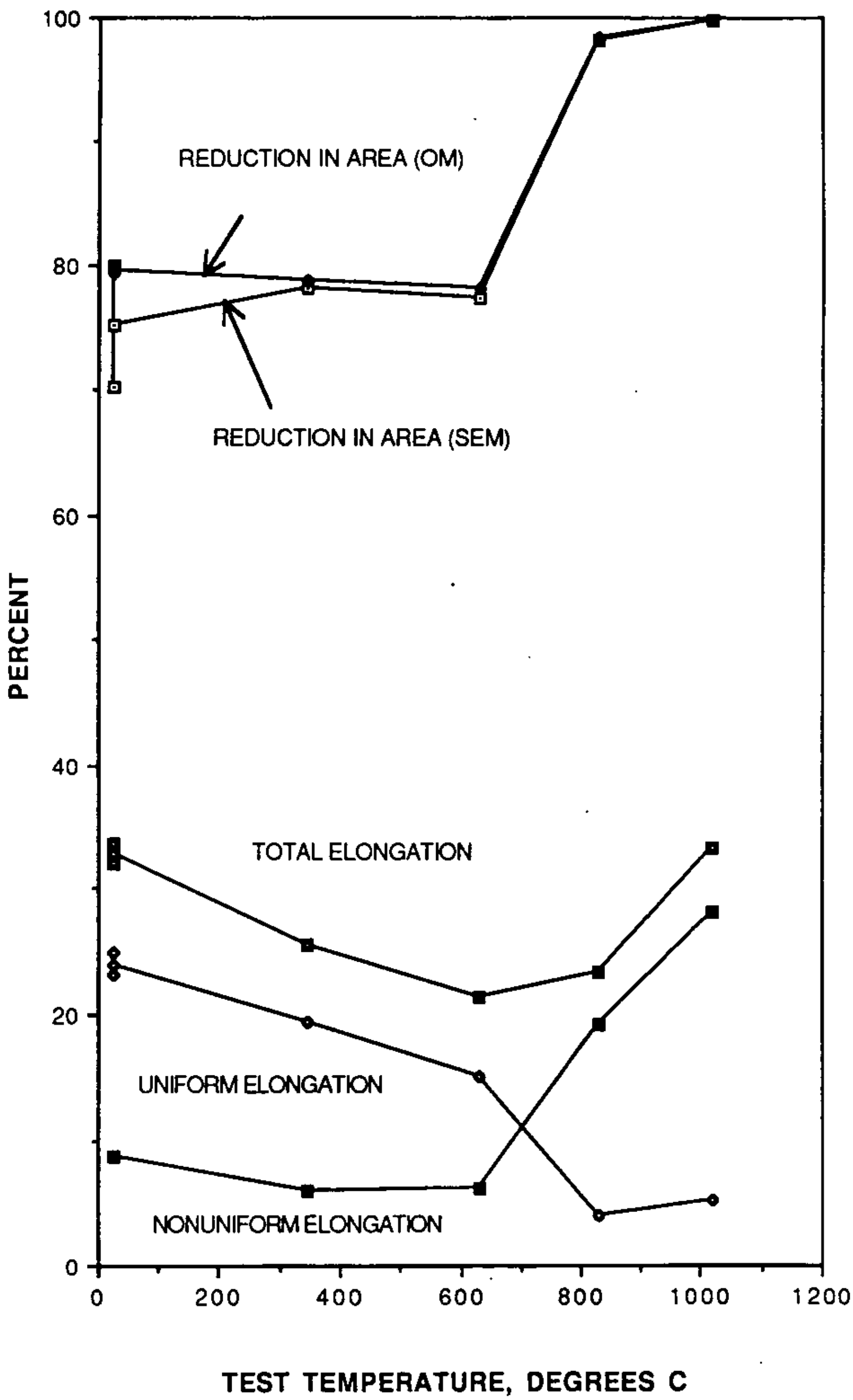


FIGURE 11. Ductility Versus Temperature Determined in Routine Rapid Heating Tensile Tests of Hydrogen-Charged Specimens of HERF 316L Stainless Steel

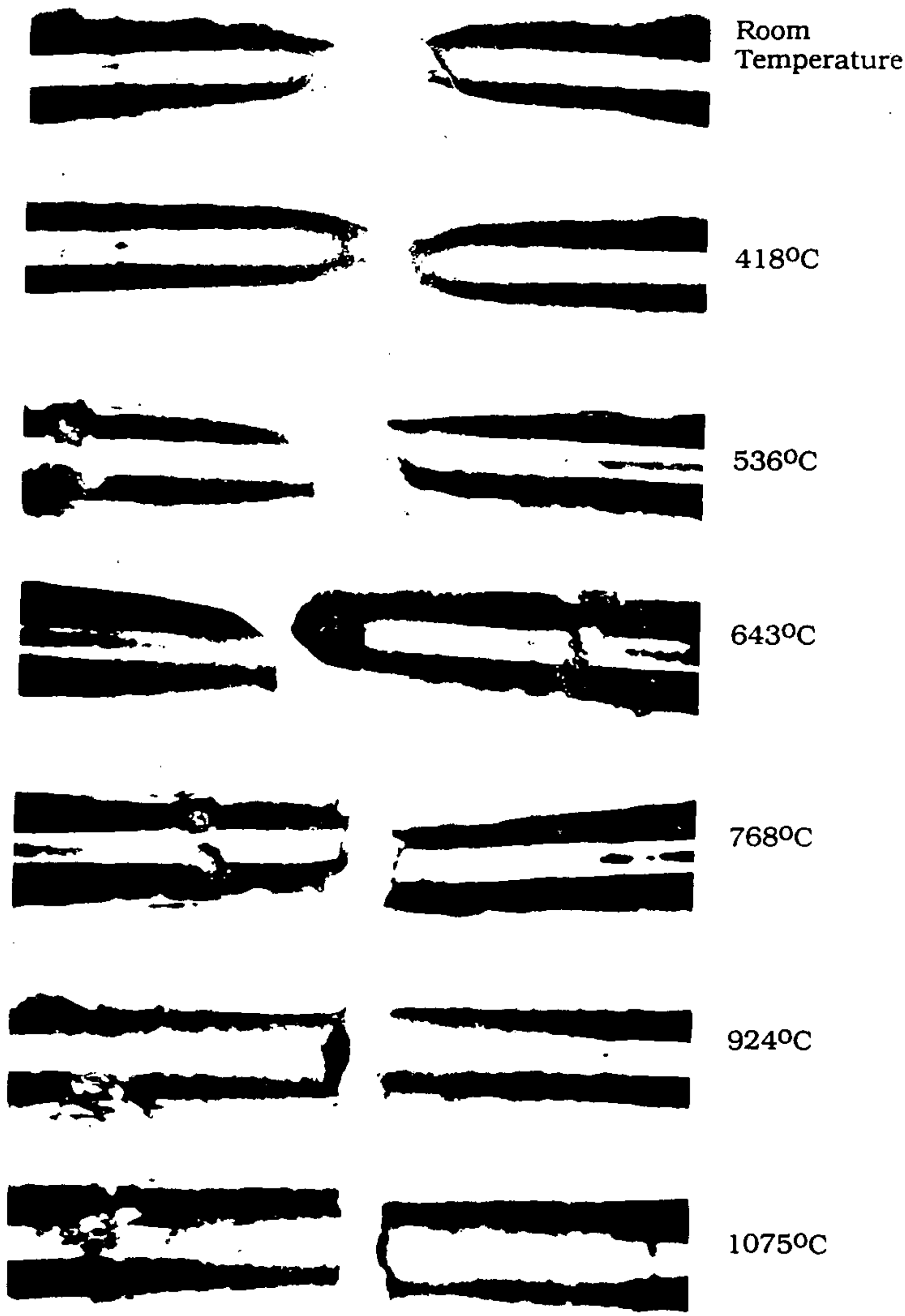
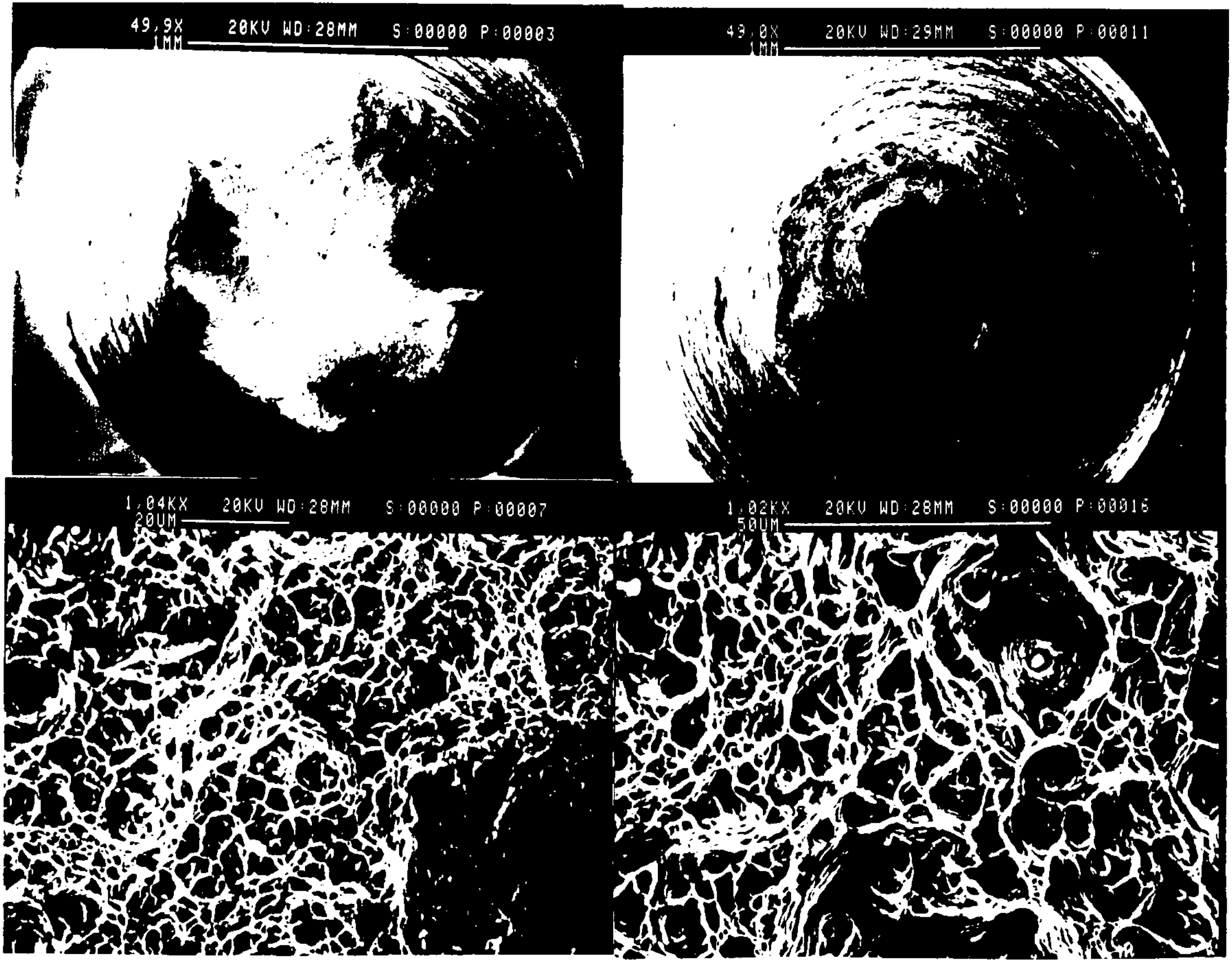
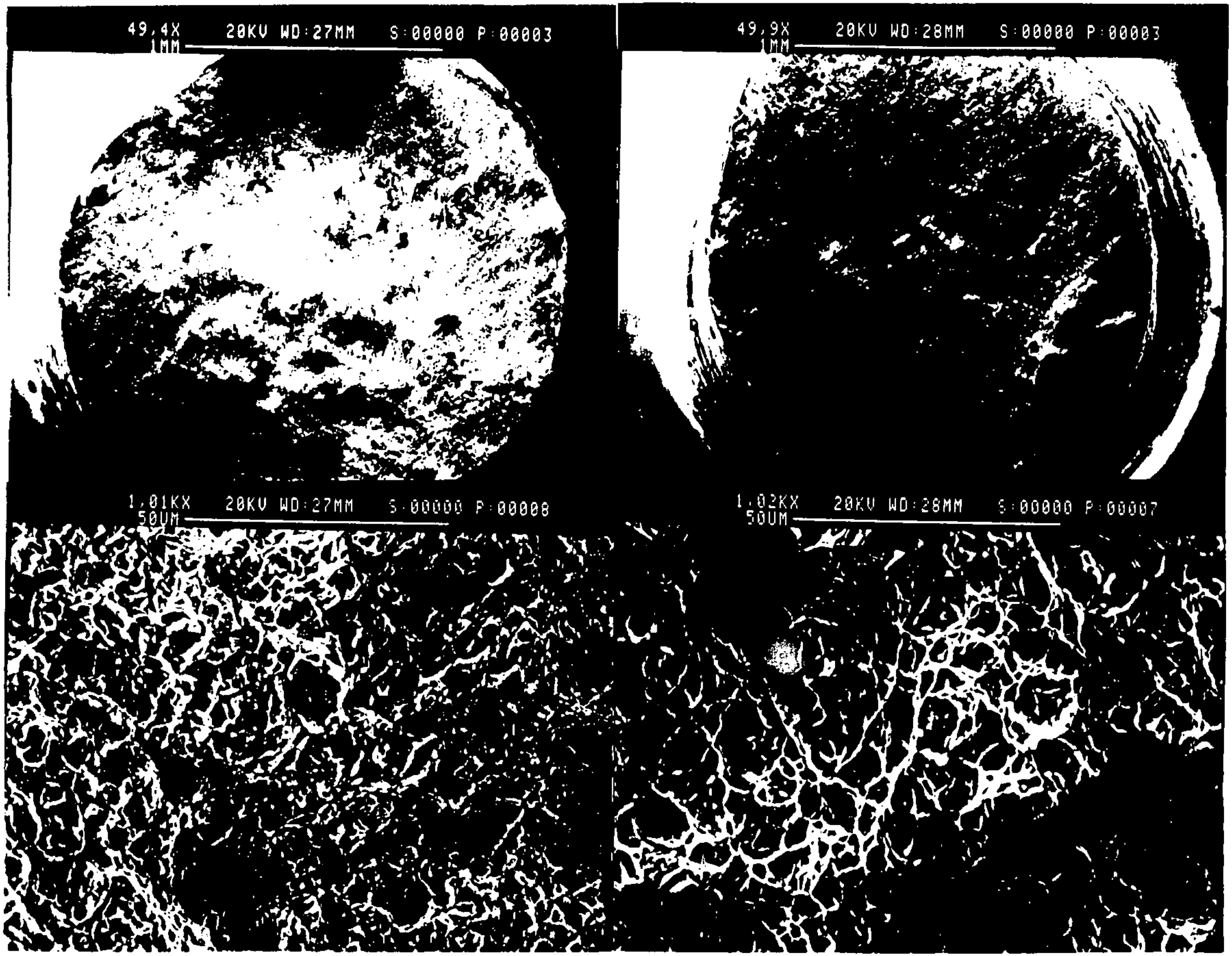


FIGURE 12. Specimens of Tritium-Charged and Six-Months-Aged HERF 316L Stainless Steel from Routine Rapid Heating Tensile Tests



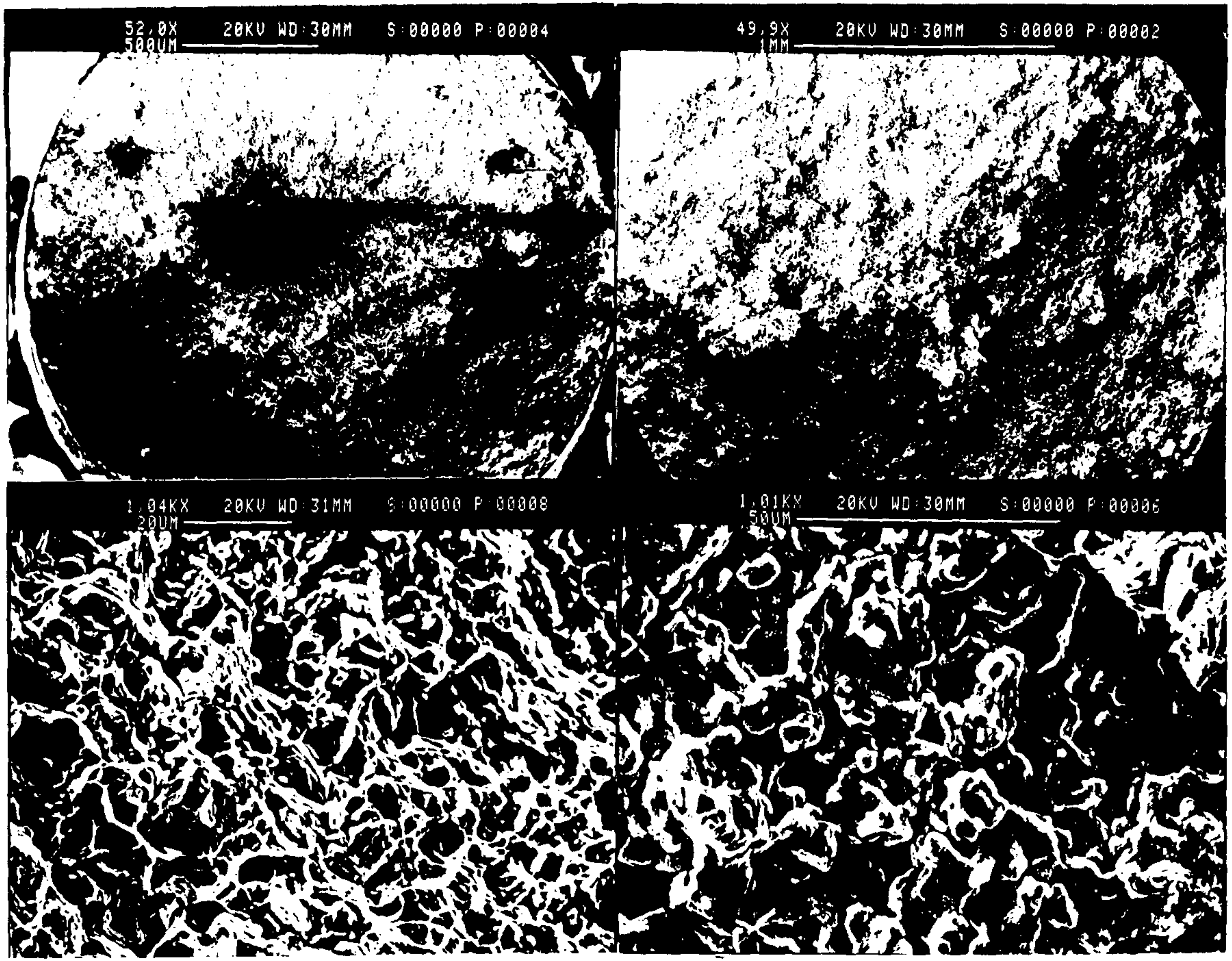
Room Temperature 418°C

FIGURE 13. Fracture Surfaces of Specimens of Tritium-Charged and Six-Months-Aged HERF 316L Stainless Steel from Routine Rapid Heating Tensile Tests



536°C 643°C

FIGURE 13 (Continued). Fracture Surfaces of Specimens of Tritium-Charged and Six-Months-Aged HERF 316L Stainless Steel from Routine Rapid Heating Tensile Tests



768°C

924°C

FIGURE 13 (Continued). Fracture Surfaces of Specimens of Tritium-Charged and Six-Months-Aged HERF 316L Stainless Steel from Routine Rapid Heating Tensile Tests

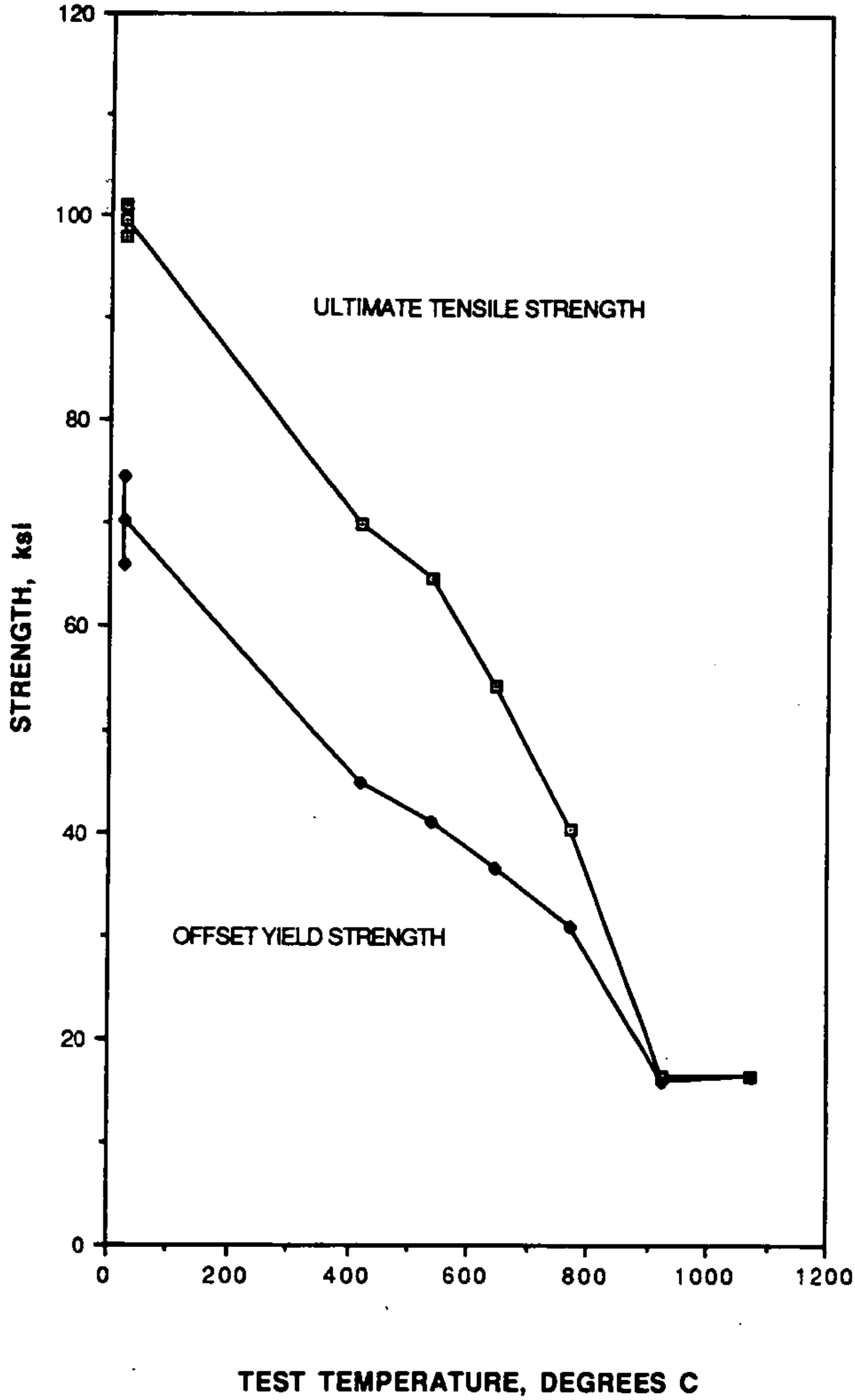


FIGURE 14. Strength Versus Temperature Determined in Routine Rapid Heating Tensile Tests of Tritium-Charged and Six-Months-Aged Specimens of HERF 316L Stainless Steel

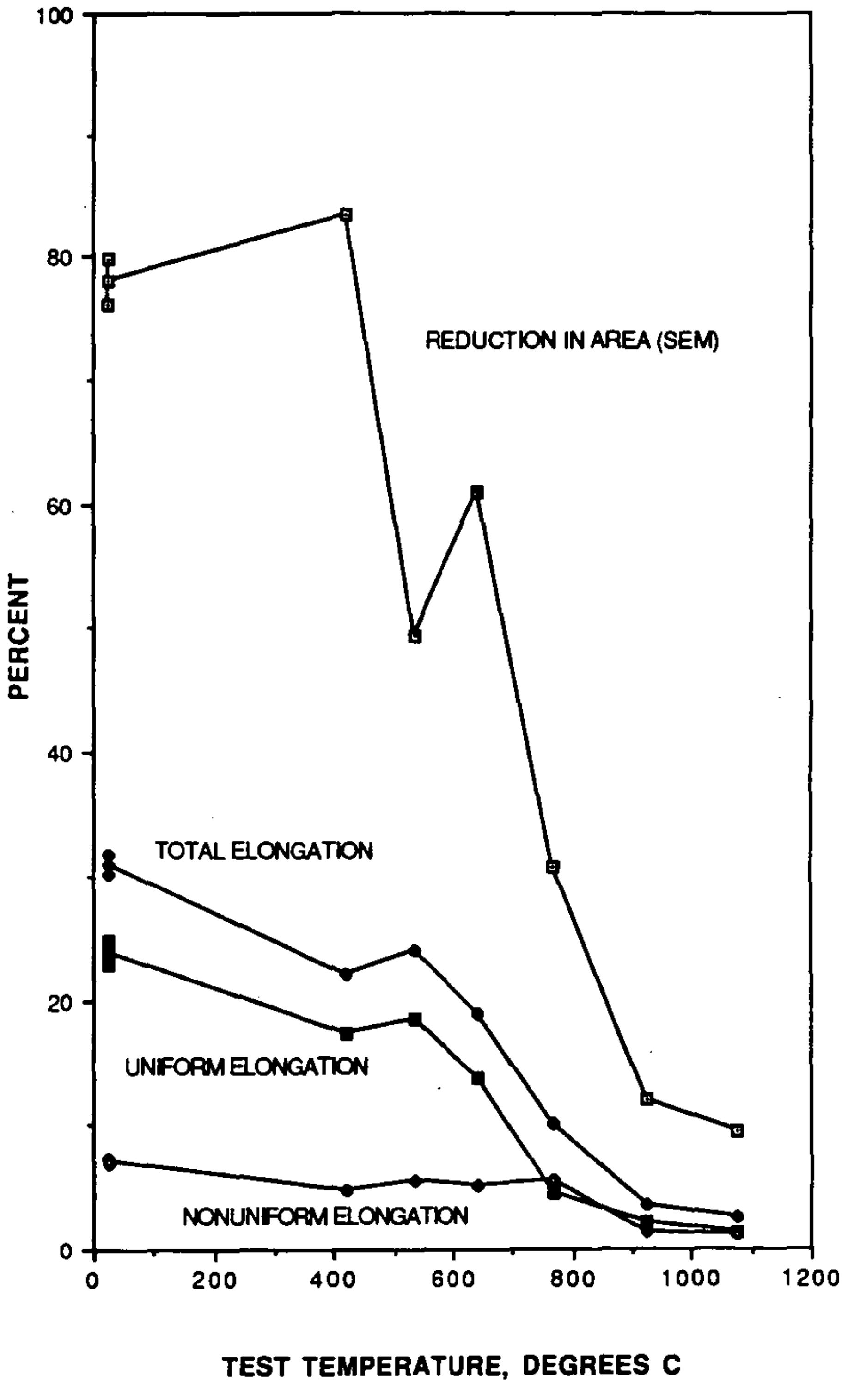


FIGURE 15. Ductility Versus Temperature Determined in Routine Rapid Heating Tensile Tests of Tritium-Charged and Six-Months-Aged Specimens of HERF 316L Stainless Steel

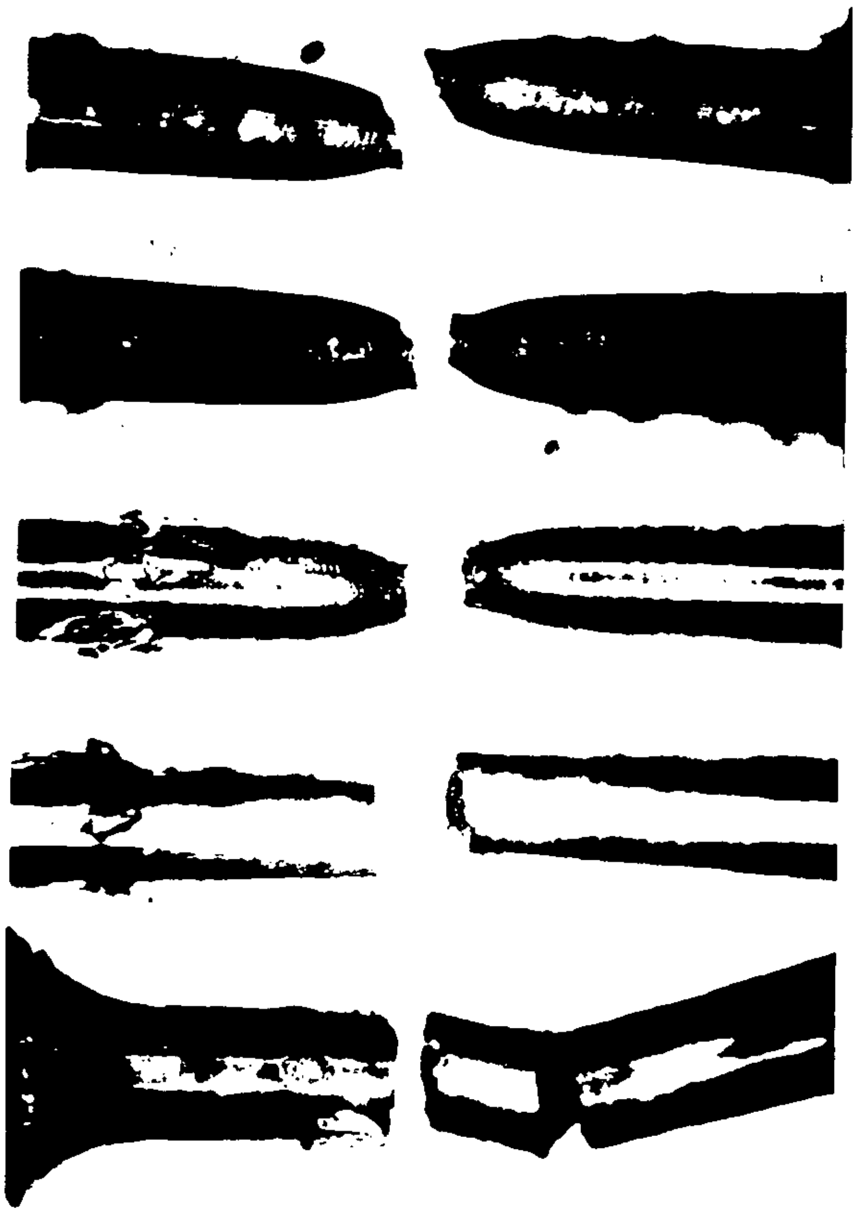
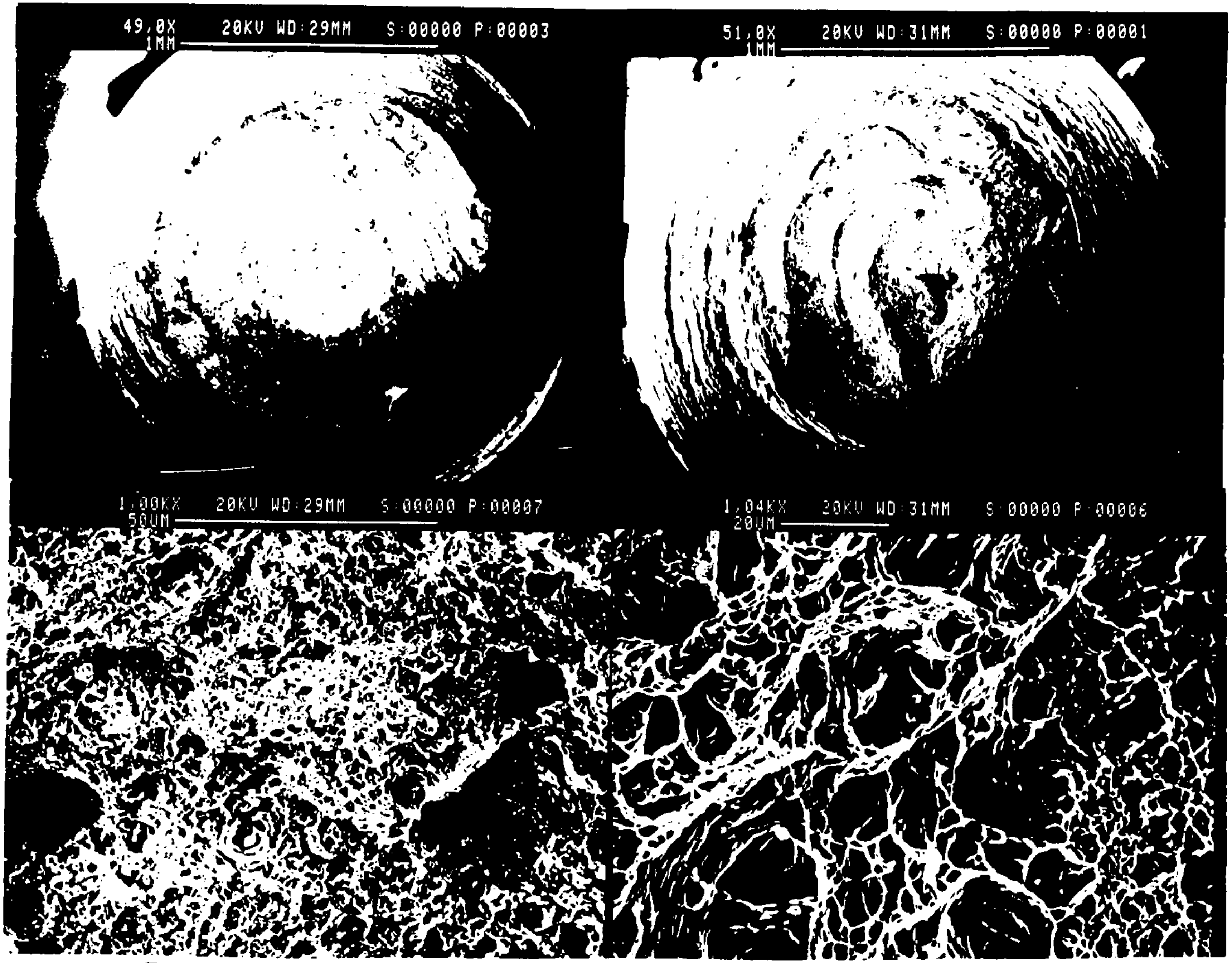


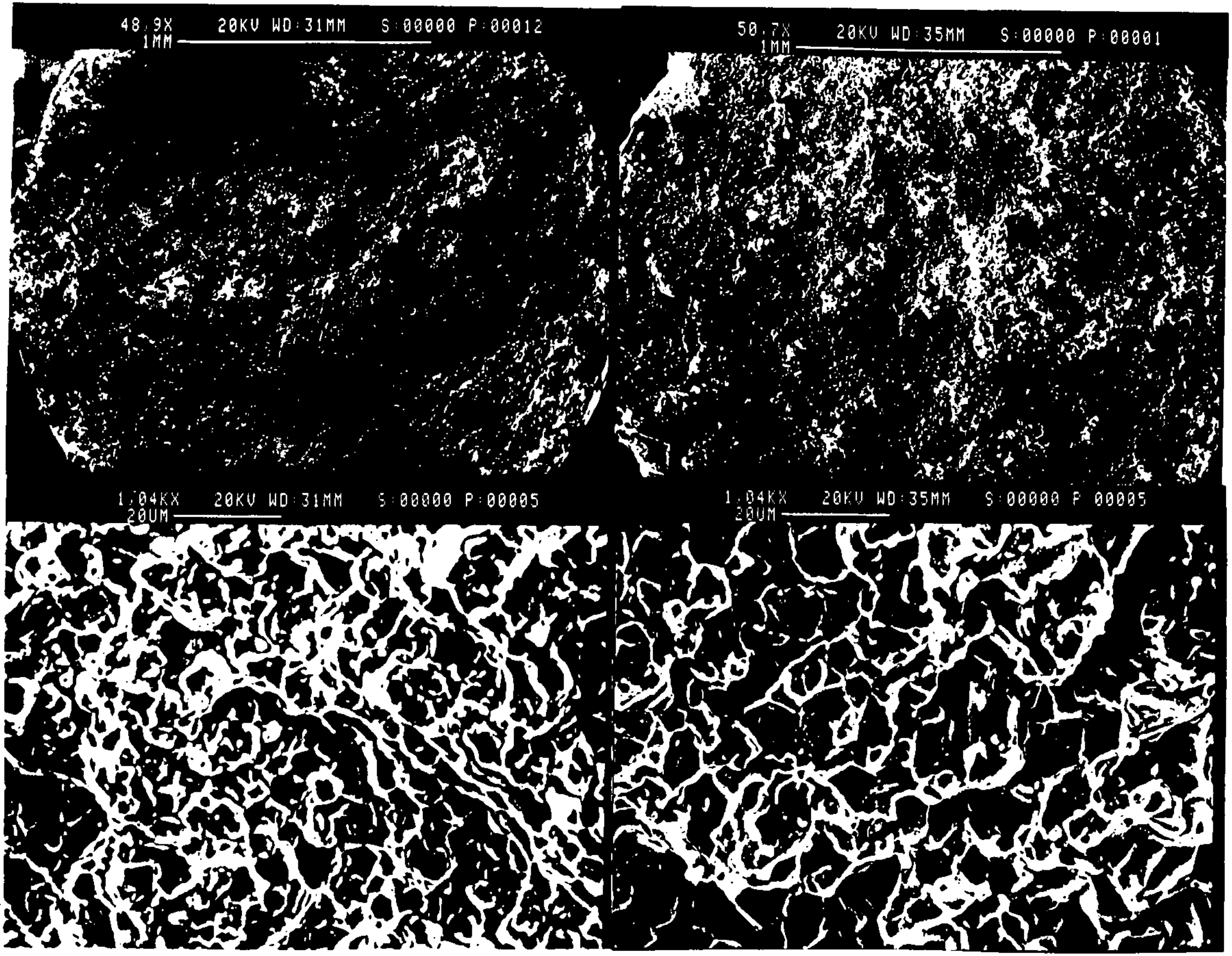
FIGURE 16. Specimens of Tritium-Charged and Six-Months-Aged HERF 316L Stainless Steel from Special Tests



Room Temp. After 5 Minutes At 1025°C

720°C After 5 Minute Soak

FIGURE 17. Fracture Surfaces of Specimens of Tritium-Charged and Six-Months-Aged HERF 316L Stainless Steel from Special Tests



893°C After 5 Minute Soak

892°C 0.005 Inch/Minute

FIGURE 17 (Continued). Fracture Surfaces of Specimens of Tritium-Charged and Six-Months-Aged HERF 316L Stainless Steel from Special Tests

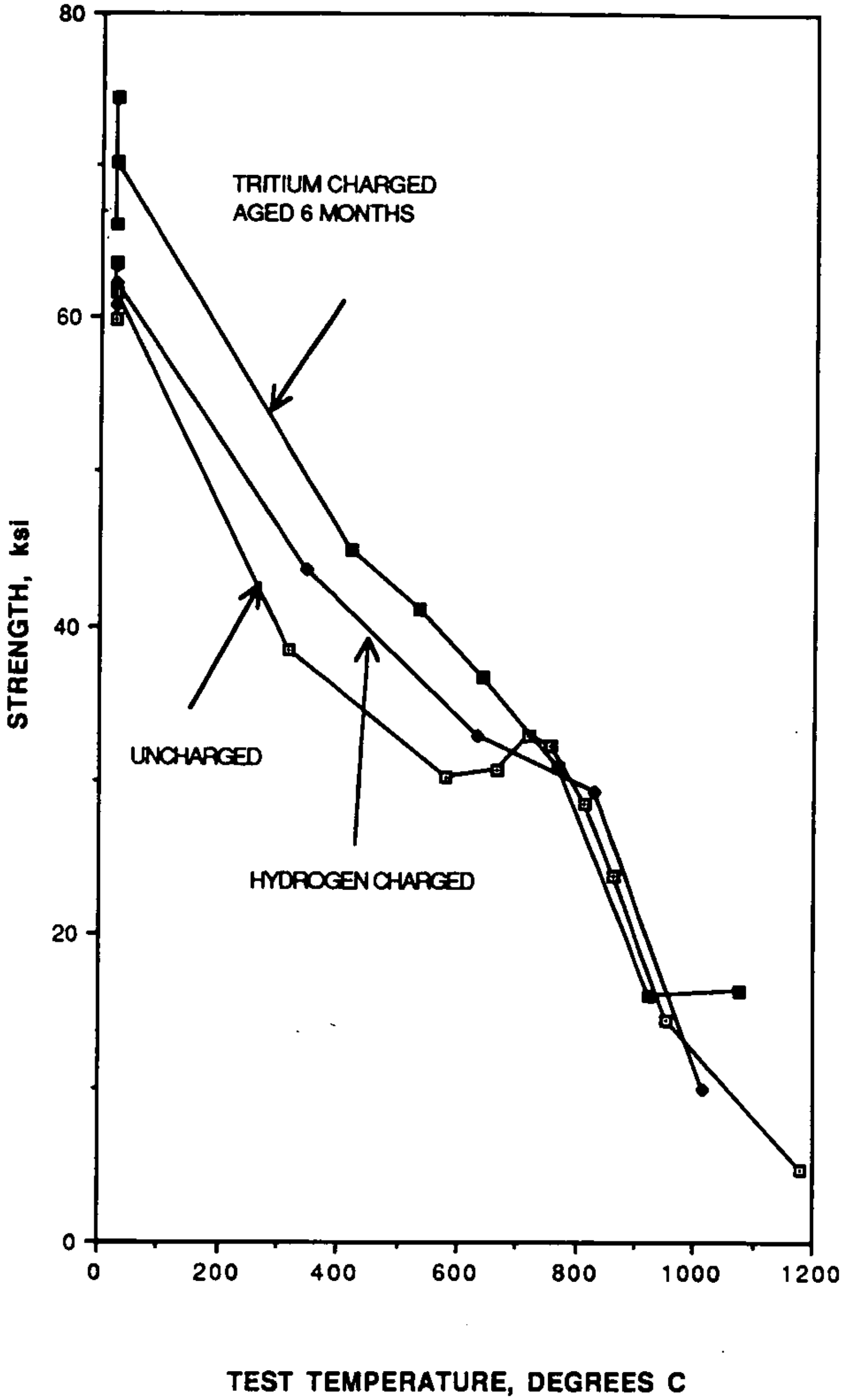


FIGURE 18. Offset Yield Strength Versus Temperature Determined in Routine Rapid Heating Tensile Tests of HERF 316L Stainless Steel

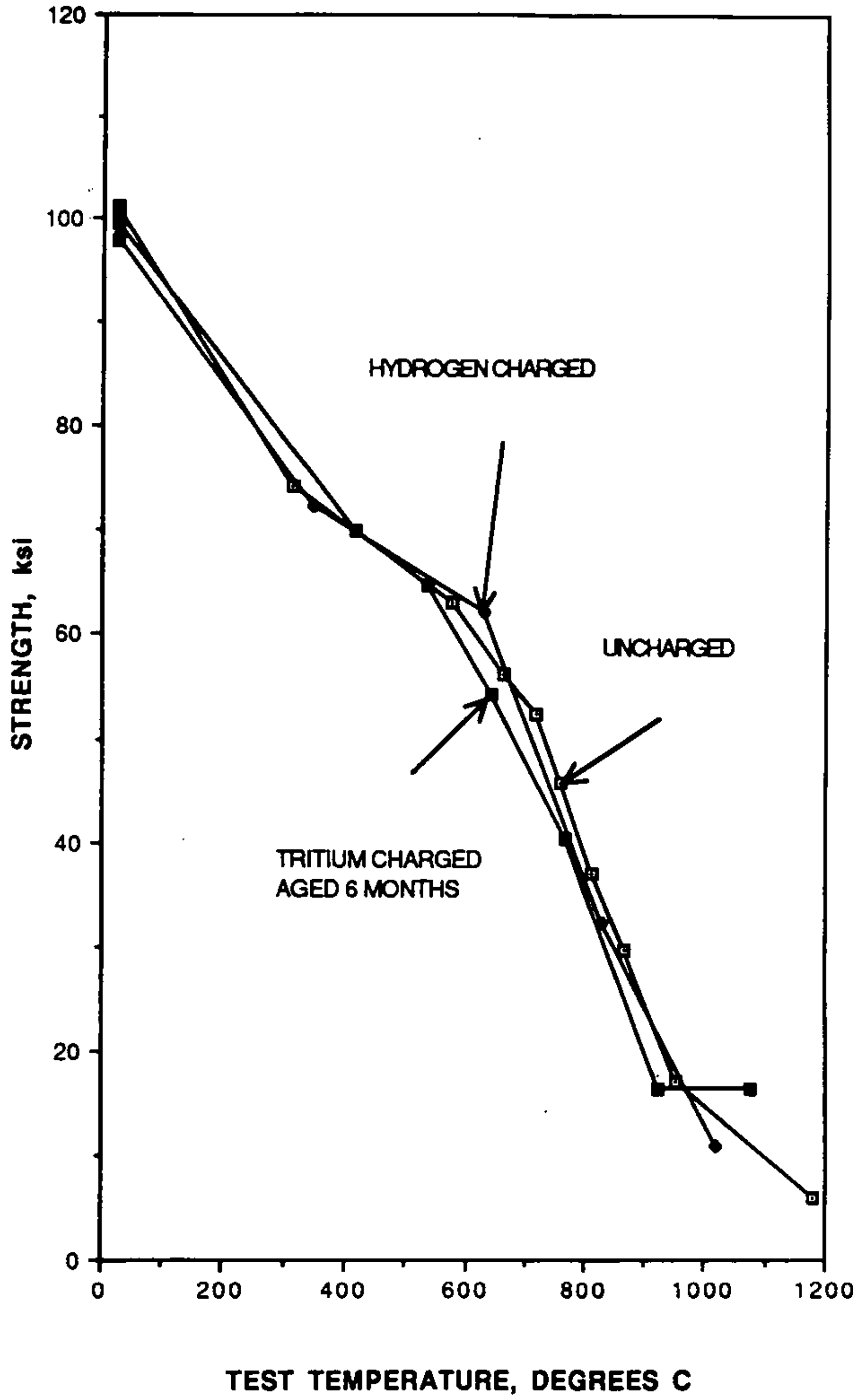


FIGURE 19. Ultimate Tensile Strength Versus Temperature Determined in Routine Rapid Heating Tensile Tests of HERF 316L Stainless Steel

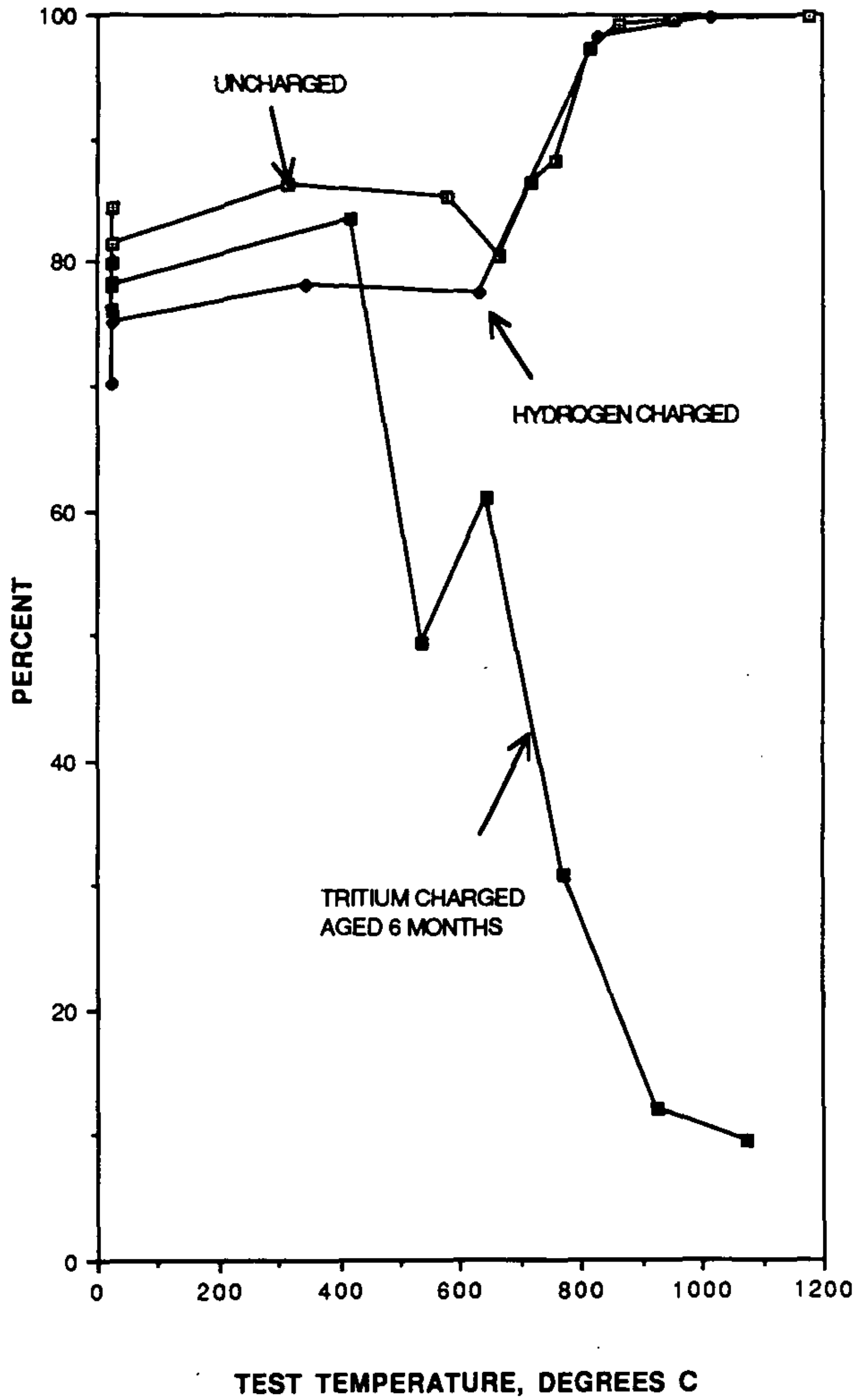


FIGURE 20. Reduction-in-Area Versus Temperature Determined in Routine Rapid Heating Tensile Tests of HERF 316L Stainless Steel

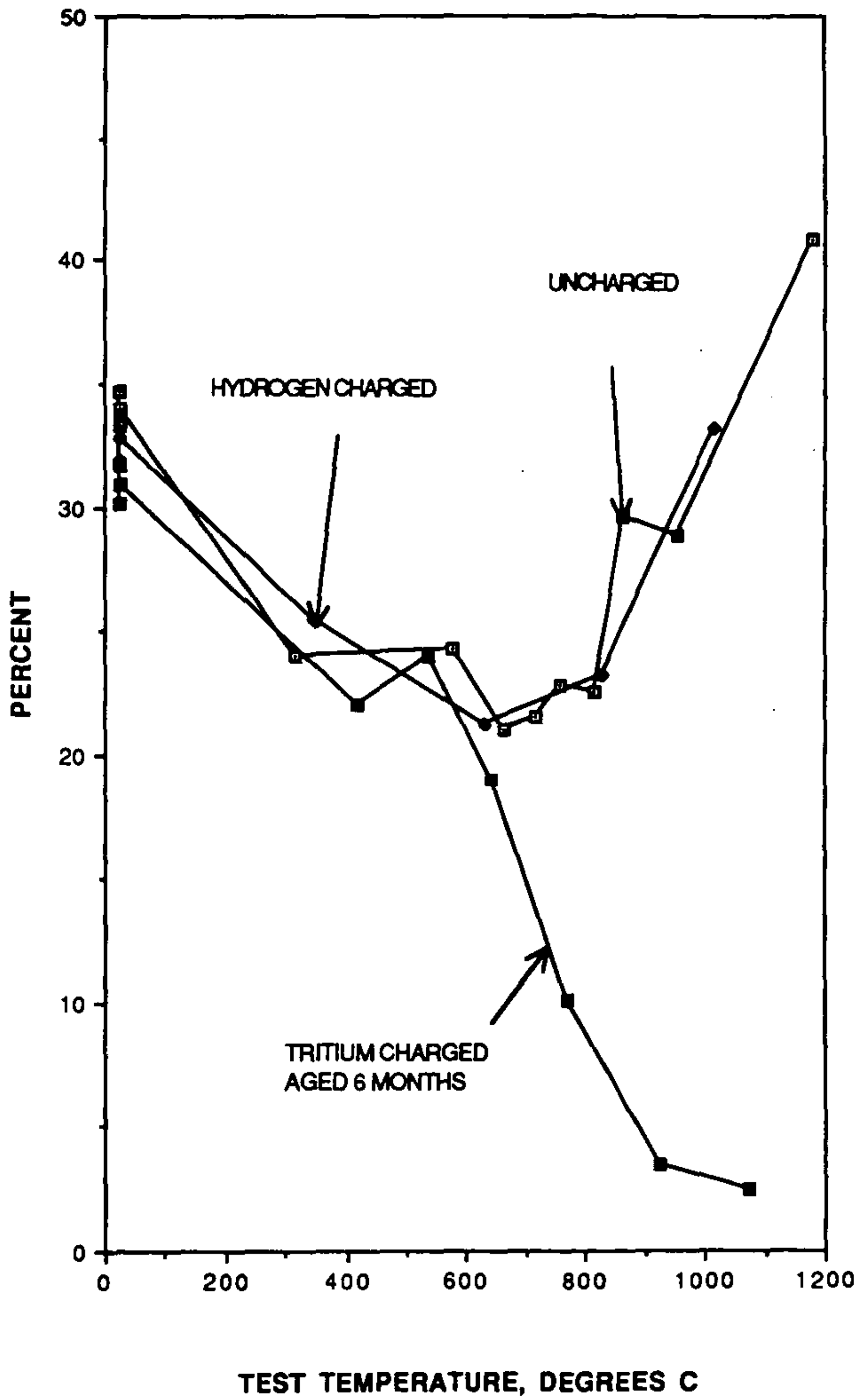


FIGURE 21. Total Elongation Versus Temperature Determined in Routine Rapid Heating Tensile Tests of HERF 316L Stainless Steel

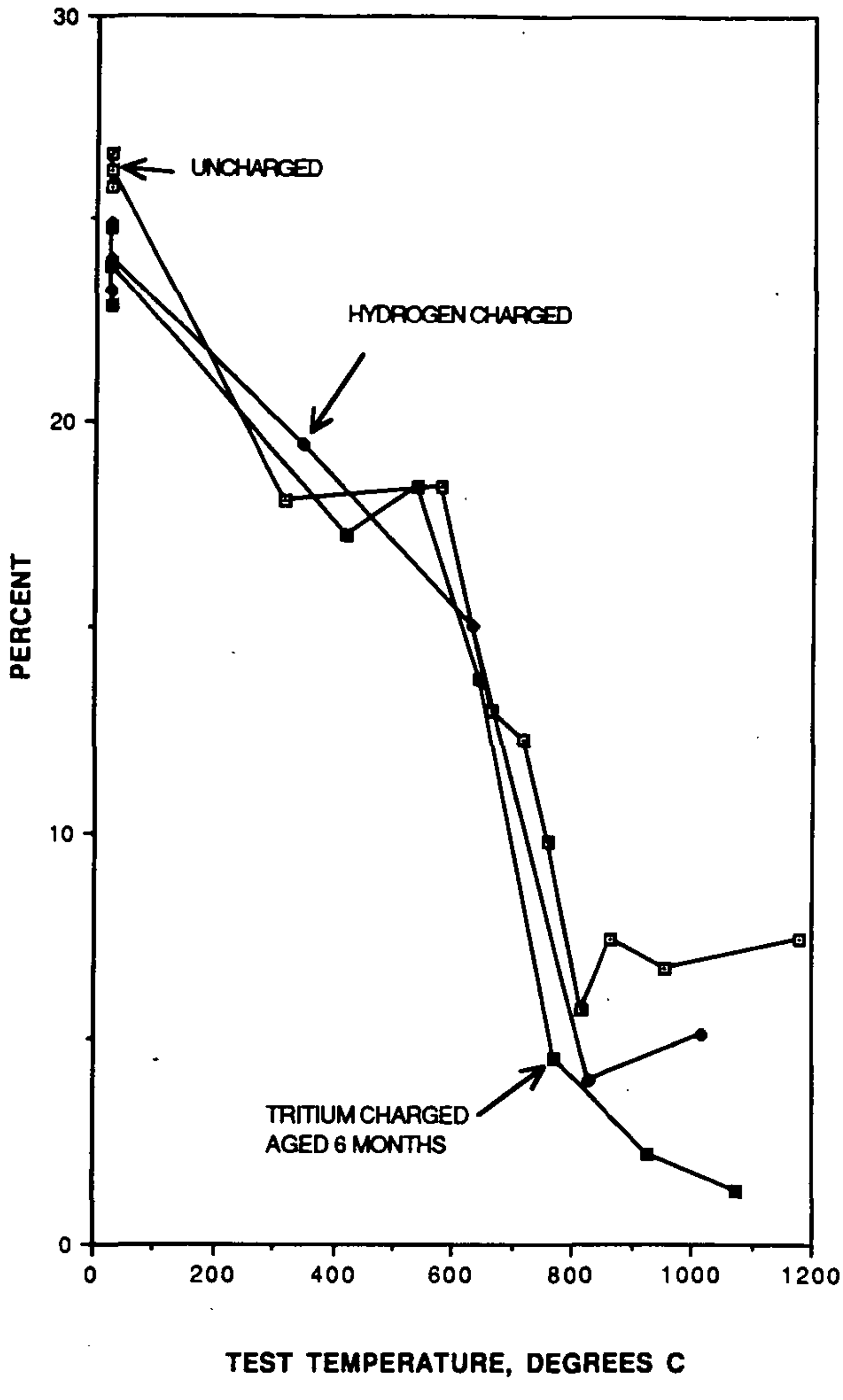


FIGURE 22. Uniform Elongation Versus Temperature Determined in Routine Rapid Heating Tensile Tests of HERF 316L Stainless Steel

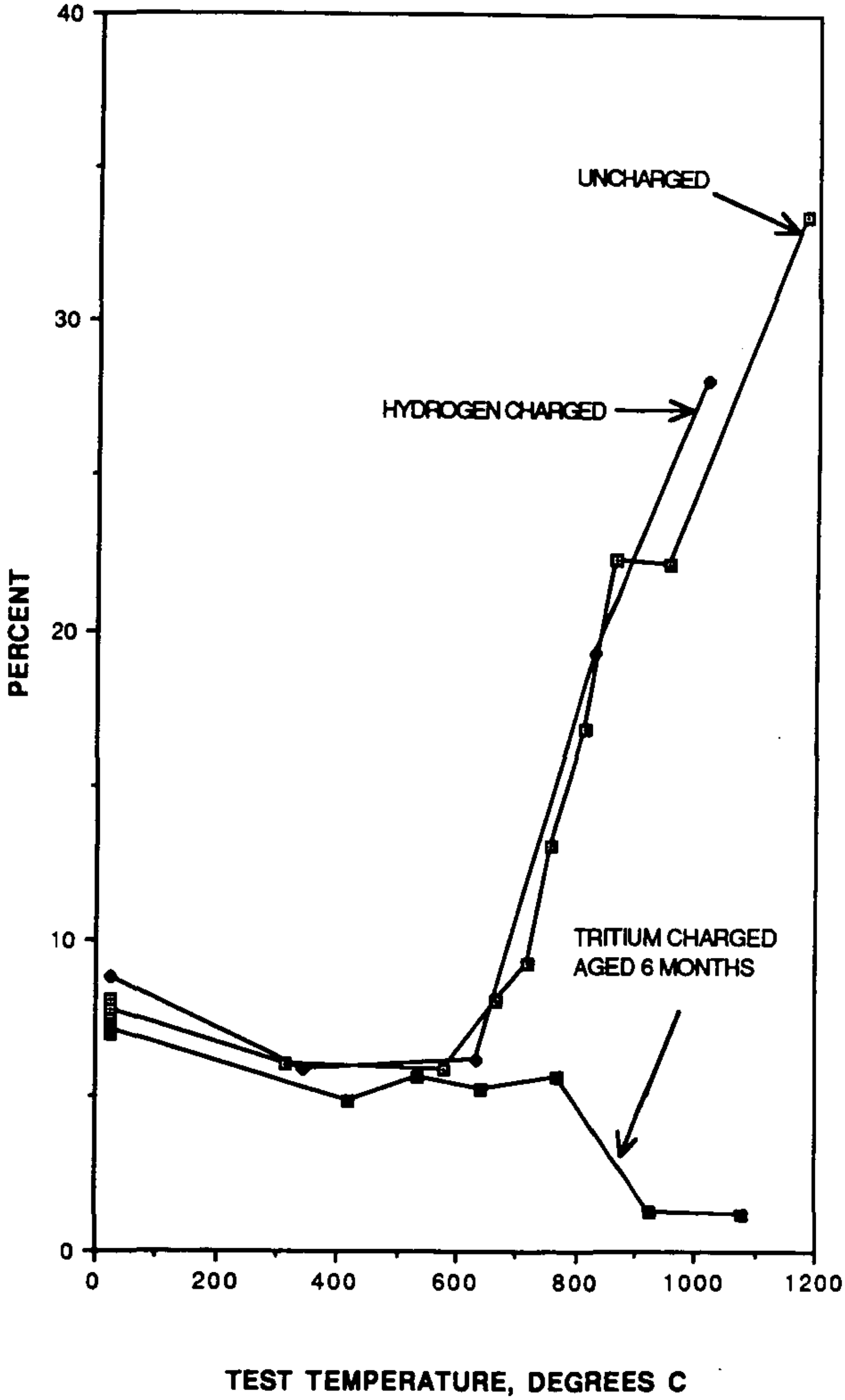


FIGURE 23. Nonuniform Elongation Versus Temperature Determined in Routine Rapid Heating Tensile Tests of HERF 316L Stainless Steel