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EMBRITTLEMENT OF NICKEL-, COBALT-, AND IRON-BASE SUPERALLOYS BY EXPOSURE TO HYDROGEN

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16. Abstract
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EMBRITTLEMENT OF NICKEL-, COBALT-, AND IRON-BASE SUPERALLOYS BY EXPOSURE TO HYDROGEN

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SUMMARY

The susceptibility of seven superalloys to embrittlement by exposure in gaseous hydrogen at elevated temperatures was determined. Five nickel-base alloys (Inconel 718, Udimet 700, Rene 41, Hastelloy X, and TD-NiCr), one cobalt-base alloy (L-605), and one iron-base alloy (A-286) were tested in various cold-rolled (CR) and heat-treated (HT, HT-1,...) conditions. Specimens were exposed in 0.1-MN/m^2 (15-psi) hydrogen at several temperatures in the range 430° to 980° C for as long as 1000 hours. Embrittlement was determined by mechanically testing the specimens at a low strain rate at room temperature after the hydrogen exposure.

Moderate embrittlement after exposure in hydrogen at 650° C for 1000 hours was observed for Rene 41/HT-1, Inconel 718/HT, and A-286/HT. Substantial embrittlement was observed for Hastelloy X/HT, Rene 41/HT-2, TD-NiCr/HT, and Udimet 700/HT-3. Severe embrittlement was observed for A-286/CR, Rene 41/CR, Rene 41/HT-3, Inconel 718/CR, and L-605/CR. Although L-605/HT and Udimet 700/HT-2, HT-1, and CR were apparently not embrittled by hydrogen, their ductility after exposure in air was so low that they should be used with caution for structural applications.

Substantial concentrations of hydrogen were absorbed by all alloys during exposure in hydrogen. No changes in microstructural features were observed for any alloy, and only minor changes in fractographic features were observed.

For Inconel 718, the following observations were also made: Embrittlement was more severe for specimens tested at low strain rates than for those tested at high strain rates. Elevated-temperature outgassing treatments after exposure in hydrogen resulted in the loss of hydrogen and the recovery of ductility.

It is suggested that such strain-rate sensitivity and outgassing response is common in varying degrees for superalloys of the types studied in this investigation. These analytical and mechanical results are indicative of interstitially dissolved, diffusible hydrogen and are consistent with a mechanism of Internal Reversible Hydrogen Embrittlement.

INTRODUCTION

The compatibility of structural alloys with hydrogen and hydrogen-containing environments is becoming increasingly important to the aerospace and ground power generation and transmission industries. The problems associated with internal hydrogen embrittlement have been recognized for many years and are now reasonably well understood. Hydrogen pickup during material processing, electroplating, and corrosion can result in Internal Reversible Hydrogen Embrittlement due to diffusible hydrogen within a component (ref. 1). Hydrogen that subsequently reacts chemically within the component to form a new phase, such as gas bubbles (molecular hydrogen, methane, or water vapor) or hydrides can result in a distinctly different form of hydrogen embrittlement - Hydrogen Reaction Embrittlement (ref. 1).

Another more recent and possibly distinct form of hydrogen embrittlement has caused considerable interest and controversy within the past few years. Increasing use of high-purity, high-pressure hydrogen for space applications has resulted in several field failures of storage tanks and has stimulated extensive NASA-supported research. Much of the laboratory evidence suggests that embrittlement in a hydrogen environment may be a surface or near-surface effect (ref. 1).

Advanced auxiliary and main power generating units being evaluated by NASA will use hydrogen gas as a fuel. Nickel-base superalloys are proposed for many components of these power units. Initial laboratory research (ref. 2) demonstrated that nickel-base superalloys are severely embrittled when tensile tested at room temperature in high-pressure gaseous hydrogen. More recent research (refs. 3 to 7) has demonstrated that embrittlement and/or enhanced crack growth can occur over the temperature range -100° to 700° C and the pressure range 10^{-12} to 10^{2} MN/m^{2}. Since such embrittlement was observed only during dynamic mechanical testing, it has been suggested that significant plastic strain was required before embrittlement by hydrogen occurred. These previous investigations are in general agreement that nickel-base superalloys and high-strength ferritic and martensitic steels are extremely susceptible to Hydrogen Environment Embrittlement. The few cobalt-base superalloys that have been tested exhibit an intermediate degree of susceptibility, while stable austenitic stainless steels have been shown to be resistant to Hydrogen Environment Embrittlement (ref. 1).

For long-life design applications, structural alloys must be resistant to both the embrittlement under dynamic test conditions discussed in the preceding paragraphs (Hydrogen Environment Embrittlement) and to embrittlement resulting from the absorption of hydrogen during long-term service under expected operating conditions (Internal Reversible Hydrogen Embrittlement or, possibly, Hydrogen Reaction Embrittlement). The objective of this investigation was to determine the residual strength and ductility of typical nickel-, cobalt-, and iron-base structural alloys after long-term exposures in hydrogen at elevated temperatures. Although the experimental conditions used in this investigation simulate the expected service conditions of an aerospace auxiliary power unit, the results obtained are applicable to many types of aerospace propulsion systems and to "hydrogen economy" ground power generating systems, as well as to commercial processes such as heat treating of superalloys in hydrogen.

Five nickel-base superalloys, one cobalt-base superalloy, and one iron-base superalloy were evaluated in this study. Specimens were exposed in air and in hydrogen at 0.1 MN/m^2 (15 psi) in the temperature range 430° to 980° C for as long as 1000 hours. After exposure, specimens were tested at room temperature to determine residual mechanical properties.

MATERIALS, SPECIMENS, AND PROCEDURES

Materials

Seven commercially available superalloys were used in this investigation. Five of these alloys were nickel base: Inconel 718, Udimet 700, Rene 41, Hastelloy X, and TD-NiCr. One alloy was cobalt base, L-605; and one alloy was iron base, A-286. The chemical compositions of these alloys are presented in table I. The vendors, heat numbers, sheet thicknesses, as-received conditions, and heat treatments performed at NASA are listed in table II. Most alloys were received as cold-rolled sheets and were tested in both the cold-rolled condition and after application of a standard heat treatment for that particular alloy. After application of the heat treatment, each alloy was in the condition in which it is normally used (refs. 8 and 9). The Udimet 700 alloy was tested not only in the form of cold-rolled sheet, but also in the form of slabs which were machined from hot-rolled bar stock. These slabs of Udimet 700 were given three different heat treatments because the alloy is used in each of the heat-treated conditions. Rene 41 was given three special-purpose heat treatments to vary the size of the gamma prime precipitate particles.

The term alloy/condition will be used in this report to describe all the alloys in their various cold-rolled (CR) or heat-treated (HT) conditions: for example, Inconel 718/CR, Rene 41/HT-1.

Photomicrographs of the alloys in all conditions tested in this program are shown in figure 1. The microstructures of as-received and heat-treated alloys were typical and representative of normal material with the possible exception of TD-NiCr, which appeared not to have fully recrystallized during heat treatment. Alloys were etched with a mixture of 33-percent acetic acid, 33-percent nitric acid, 33-percent water, and 1-percent hydrofluoric acid.

Specimens

Sheet tensile specimens of the type illustrated in figure 2 were used most extensively in this investigation. The thickness of the specimens was the same as the sheet thickness except for the heat-treated conditions of Udimet 700, which were machined from bar stock to a thickness of 2.5 millimeters (0.1 in.). All alloys were tested with the specimen surfaces in the rolled, heat-treated, or machined conditions. No surface preparation, other than degreasing, was used. The longitudinal axis of all specimens was parallel to the rolling direction of the sheet or bar.

The self-stressed specimen illustrated in figure 3 was used for determining the effect of applied stress and for spot-checking selected test results of Inconel 718. The construction of these specimens involved shearing the specimen strips from the sheet alloy, bending up the ends of each strip to a predetermined bend angle, and spotwelding the ends together. Bending stresses corresponding to the uniform curvature in the specimens were calculated from geometrical relations and from the equations associated with bending (ref. 10). Stresses were increased by increasing the bend angle of the strips, which increased the distance D between strips. Self-stressed specimens of Inconel 718 were designed to stress levels of 150, 300, 450, 600, and 750 MN/m^2 (22, 44, 65, 87, and 109 ksi).

Test Procedures

Thermal exposures. - Specimens of all alloys were continuously exposed in air and hydrogen in commercial, heated furnaces at temperatures of 430°, 540°, 650°, 760°, and 980° C. Times of 100 and 1000 hours were used for alloys except Rene 41, in which case the test times were 20 and 1000 hours. Cyclic exposures (33 cycles of 3 hr each, and 10 cycles of 10 hr each) between room temperature and the various exposure temperatures were also applied for Inconel 718. A typical heating and cooling profile for the furnace used is shown in figure 4 for an exposure temperature of 650° C. The hydrogen pressure was 0.1 MN/m^2 (15 psi) at a flow rate of 8 cm^3/sec (1 ft^3/hr). The minimum purity of the bottled hydrogen was 99.0 percent.

Postexposure tensile testing. - Sheet tensile specimens were tensile tested at room temperature immediately after exposure to hydrogen. The tensile crosshead speed (strain rate) was a constant 0.1 mm/min through fracture. Both ultimate tensile strength and elongation over a 25.4-millimeter gage length were determined. All test results are tabulated in table III.

Postexposure compression-bend testing. - Self-stressed specimens were compression-bend tested at room temperature immediately after exposure. A constant crosshead speed (strain rate) of 0.1 mm/min was used. Figure 3 illustrates the test procedure and the data measurements that were made. The maximum amount of deflection that could be measured during compression testing was about 60 millimeters. The degree of embrittlement of an alloy was defined as the percentage of deflection at fracture after exposure in hydrogen compared with the deflection at fracture after exposure in air.

Special test conditions. - The following exceptions were made to the standard post-exposure mechanical test conditions for both types of Inconel 718/CR specimens. Some specimens were tested at a crosshead speed (strain rate) of 10 mm/min. Some hydrogen-exposed specimens were stored at room temperature in air for 30 days before mechanical testing. Some hydrogen-exposed specimens were outgassed at 650° C for 3 hours in air and some in vacuum before subsequent mechanical testing.

Hydrogen analyses. - Standard vacuum fusion chemical analyses for hydrogen content were made on selected specimens of each alloy by an independent laboratory. Small samples were cut from regions immediately adjacent to the fracture surfaces of the broken specimens. Repeated analyses of Inconel 718 specimens subjected to the same hydrogen exposure several times over a 2-year period showed that the vendor's precision was about ±1 ppm by weight.

Metallography. - The microstructures of all alloys were examined by light microscopy in both the as-received and heat-treated conditions (fig. 1) and after various exposures to hydrogen. Replica electron microscopy and scanning electron microscopy were used to examine the microstructures and fracture modes of selected alloys in the as-received, heat-treated, and exposed conditions.

RESULTS AND DISCUSSION

Screening Tests on Inconel 718

An extensive screening program was conducted with Inconel 718 because it is a commonly used nickel-base superalloy, because it has been extensively evaluated in high-pressure hydrogen environments in other investigations (refs. 2 to 4 and 7), and because it was readily available in large quantities and in three different cold-rolled conditions (0, 20, and 35 percent). Specimens were tested both in the cold-rolled conditions and in the cold-rolled-plus-heat-treated conditions.

Effect of hydrogen exposure temperature. - Exposures in air and hydrogen were conducted at 430°, 540°, 650°, and 760° C for 100 hours in both continuous and cyclic modes. The resultant room-temperature elongations of specimens exposed under these conditions are shown in figures 5 to 7 for the three cold-rolled conditions. It is apparent from these data that exposures in hydrogen resulted in significant reductions in subsequent ductility for all temperatures considered. Maximum embrittlement was usually observed after exposures at 650° and 760° C.

Effect of continuous and cyclic exposures. - As figures 5 to 7 also show, all cold-rolled and cold-rolled-plus-heat-treated conditions of Inconel 718 were exposed in hydrogen for 100 hours in both a continuous mode and in a cyclic mode of 33 cycles of 3 hours each. For many of the conditions and temperatures tested, cyclic exposures appeared to result in slightly more embrittlement than did continuous exposures.

Reproducibility of test data. - An important consideration in these test results is the degree of reproducibility. Several exposure conditions were used to spot-check this aspect of the investigation. The results shown in figure 6 for Inconel 718 in the cold-rolled and cold-rolled-plus-heat-treated conditions indicate that duplicate and triplicate exposures resulted in excellent reproducibility of room-temperature elongation data. Such lack of scatter justifies the single test points used to a large extent in this study.

Effect of hydrogen exposure time. - Additional specimens of all conditions of Inconel 718 were exposed in hydrogen at 650° C for 1000 hours in a continuous mode. These results, together with the 100-hour data discussed previously, are presented in figure 8. In general, increasing the exposure time from 100 to 1000 hours resulted in slight increases in the degree of embrittlement for most conditions of this alloy.

Relative Embrittlement of Alloys

Based on an evaluation of the exposure variables used for Inconel 718, the following exposure conditions were selected for subsequent testing of the other six superalloys: All alloys were exposed in air and hydrogen at 650° C. Continuous exposures of 100 and 1000 hours were used for most alloys and conditions. Resultant room-temperature elongations of these alloys are presented in figure 9 for Udimet 700, Rene 41, Hastelloy X, TD-NiCr, L-605, and A-286.

As evident from the data in figures 8 and 9, several potential schemes for ranking the relative susceptibility of these alloy/conditions are possible. They could be ranked according to the percentage loss of ductility after either 100 or 1000 hours of exposure, or they could be ranked according to the level of actual ductility after either 100 or 1000 hours. The author feels that the most meaningful method is ranking by residual ductility after 1000 hours of exposure. Such a ranking scheme is presented in table IV, which shows the elongation of the alloy/conditions after exposure in hydrogen and, for comparison, the elongation of the alloy/conditions after an identical exposure in air. It should be noted that both Inconel 718/35-percent CR and A-286/CR exhibited substantially more elongation after exposure in air for 1000 hours than for 100 hours. This effect is presumably caused by annealing treatment, which removed the effect of residual cold work.

It is evident from the results listed in table IV that alloy/condition susceptibility to hydrogen embrittlement falls into four reasonably distinct groups: Rene 41/HT-1, Inconel 718/HT, and A-286/HT were moderately embrittled by hydrogen.

Hastelloy X/HT, Rene 41/HT-2, TD-NiCr/HT, and Udimet 700/HT-3 were substantially embrittled. A-286/CR, Rene 41/CR, Rene 41/HT-3, Inconel 718/CR, and L-605/CR were severely embrittled by hydrogen. L-605/HT, Udimet 700/HT-2, Udimet 700/HT-1, and Udimet 700/CR were apparently not embrittled by hydrogen. It should be noted that Udimet 700/CR exhibited a slight amount of embrittlement after exposure in hydrogen for 100 hours (fig. 9). However, when the exposure time was extended from 100 to 1000 hours, there was no difference between the ductility of Udimet 700/CR after exposure in either air or hydrogen. Furthermore, the limited data obtained for Udimet 700/HT-2 and Udimet 700/HT-1 (only 100 hr of exposure in hydrogen) preclude a confident declaration that these heat-treated conditions of Udimet 700 are totally immune to hydrogen embrittlement for times longer than 100 hours.

Mechanism of Embrittlement

In an effort to define the embrittling role of hydrogen, the effects of variations in exposure conditions, mechanical testing, outgassing treatments, alloy strength level, and alloy microstructure were determined. Chemical analyses for hydrogen and metallographic analyses were performed.

Effect of applied stress. - The effect of stress level applied to the specimens during the exposure in hydrogen was determined by constructing self-stressed specimens of Inconel 718/35-percent CR. The specimens were fabricated to five different stress levels: 150, 300, 450, 600, and 750 MN/m^2 (22, 44, 65, 87, and 109 ksi). These specimens were then exposed in air at 650° C continuously for 100 hours and in hydrogen at 650° C. The exposures in hydrogen were (1) continuous, (2) 10 cycles of 10 hours each, and (3) 33 cycles of 3 hours each. None of the self-stressed specimens fractured during the exposure in hydrogen. After exposure the specimens were compression-bend tested at room temperature, and the amount of deflection to fracture of one or both of the specimen legs was recorded. These results are presented in figure 10 as a percentage of the deflection measured after exposure in air.

It is evident from these results that the amount of stress applied to the specimens did not significantly influence the degree of embrittlement resulting from exposure in hydrogen. For example, continuous exposure in hydrogen resulted in subsequent deflections in the range 88 to 98 percent of the air exposure deflection for all five stress levels. Likewise, there were no significant variations with applied stress for either of the cyclic hydrogen exposures: 33 cycles of 3 hours each, or 10 cycles of 10 hours each. However, both of the cyclic exposures resulted in slightly more embrittlement than did the continuous hydrogen exposure, as was noted for sheet tensile specimens in figures 5 to 7.

A comparison between the results obtained with unstressed sheet tensile specimens and highly stressed self-stressed specimens also indicates that applied stress does not significantly influence the degree of embrittlement. For example, figure 11 compares the results obtained with both tensile and self-stressed (750 MN/m^2) specimens of Inconel 718/35-percent CR and Inconel 718/CR plus HT after cyclic exposures in the temperature range 430° to 760° C. As is evident from the data in figure 11, both types of specimens produced the same trend in results, with embrittlement increasing as the hydrogen exposure temperature increased. However, poorer correlation is apparent at the lowest exposure temperature, 430° C, than at the highest exposure temperature, 760° C. A rationalization for this behavior is not apparent.

Even though these results showed that applied stress is not a critical variable during the hydrogen exposures studied, it is the author's opinion that the self-stressed specimen has been shown to be a realistic type of specimen for experimental investigations concerned with the role of stress during environmental exposures. However, limitations of this specimen type noted in this investigation are the following: Self-stressed specimens yield qualitative information on the relative effect of environmental exposures on the ductility of materials - quantitative values of elongation are not obtained. Another potential limitation is that the maximum amount of deflection that can be obtained on a ductile self-stressed specimen is limited to a maximum bend angle of 180°. This would represent a case where the specimen legs doubled back on themselves during compression-bend testing (fig. 3). Hence, for the specimen used in this study, full deflection occurred at about 60 millimeters and may indeed have masked the full extent of ductility of the very ductile specimens.

Effect of mechanical testing speed. - A classical technique used to determine the role of hydrogen after environmental exposures is to conduct postexposure mechanical testing over a range of strain rates. If embrittlement is more severe at lower strain rates than at higher strain rates, hydrogen is assumed to be diffusing within the alloy lattice and thereby controlling the degree of embrittlement. The standard testing speed (strain rate) used throughout this study for both tensile and compression-bend testing (0.1 mm/min) was relatively slow and was chosen in order to accentuate the embrittling effects of hydrogen. A few tests were also performed at a much faster testing speed (10 mm/min) to determine whether hydrogen diffused during mechanical testing.

The results obtained with these two testing speeds (strain rates) are shown in figure 12. The ductility of Inconel 718 in two cold-rolled conditions after cyclic exposure in hydrogen at 650° C does indeed depend on the testing speed. For both the tensile and self-stressed specimens, the degree of embrittlement was greater at the lower testing speed than at the higher testing speed. Such behavior is strong evidence that the hydrogen absorbed by these specimens is contained in the alloy lattice in interstitial form and diffuses during the mechanical test. At the lower strain rate, hydrogen has more time to diffuse during the test, and thus the degree of embrittlement is greater than at the higher strain rate.

Effect of outgassing treatments. - Several different outgassing treatments were performed on Inconel 718 after cyclic exposure in hydrogen at 650° C. Selected specimens were outgassed at 650° C for 3 hours in air or in vacuum immediately after 33 cycles of 3 hours each were completed in hydrogen. In effect the specimens were given one additional thermal exposure cycle, but in air or vacuum rather than in hydrogen. Other selected specimens were stored in air at room temperature for 1 month. The ductility of these selected specimens and of the baseline specimens tested immediately after hydrogen exposure is presented in figure 13. In all cases, outgassing at 650° C in air or in vacuum immediately after the last hydrogen cycle resulted in substantially less embrittlement. Such results suggest that some of the hydrogen that had been absorbed during the hydrogen exposure was removed from the samples during the subsequent 650° C outgassing exposure in air or vacuum. Hydrogen analyses support this contention, as is discussed in the next section.

The 1-month storage treatment in air at room temperature, however, did not result in any significant difference in the degree of embrittlement as compared with the baseline specimens (fig. 13). These results suggest that hydrogen does not outgas from Inconel 718 at room temperature. Hydrogen analyses made on similar specimens stored at room temperature for as long as 1 year also support this suggestion, as is discussed in the next section.

Hydrogen analyses. - A substantial number of vacuum fusion analyses of the hydrogen content of specimens were made in this study, primarily on Inconel 718, but also on selected specimens of all the alloys tested. The analyses, listed in table III, were made on specimens that had been exposed in air and hydrogen, in continuous and cyclic modes, and at temperatures ranging from 430° to 980° C.

All alloys exposed in hydrogen absorbed hydrogen to concentrations two to four times greater than the levels in the baseline, air-exposed specimens. Baseline concentrations were usually 2 to 3 weight ppm (120 to 180 atomic ppm). The hydrogen concentrations determined in the nickel-base alloys are equivalent to or greater than the equilibrium solubility data reported for unalloyed nickel (ref. 11), 5 ppm at 650° C and 10 ppm at 980° C. The hydrogen concentrations measured in all of the alloys represent considerable supersaturation with respect to the reported room-temperature solubility of less than 1 ppm for unalloyed nickel, cobalt, and iron.

No substantial differences in hydrogen concentrations were determined between specimens exposed continuously and cyclically or between specimens exposed for 100 hours and 1000 hours. Hydrogen analyses of A-286 indicated that similar concentrations of hydrogen were absorbed by both the cold-rolled and heat-treated specimens, even though the cold-rolled condition of the alloy was more severely embrittled than the heat-treated condition.

As discussed in the previous section, embrittlement of Inconel 718 could be reversed if specimens were aged at 650° C in air or in vacuum after they had been exposed in hydrogen. It was suggested that such recovery of ductility was due to hydrogen outgassing. Vacuum fusion analyses confirmed that the hydrogen concentration was reduced from 5 ppm to the baseline concentration of 2 ppm. Storage at room temperature, together with storage of selected specimens in liquid nitrogen, confirmed that no significant outgassing of hydrogen occurs in Inconel 718 and TD-NiCr at room temperature for times as long as 1 year.

Effect of alloy microstructure. - Extensive optical and replica electron microscopic examinations were conducted on all alloys after thermal exposure at 650° C. Generally, the microstructures of all alloys after exposure were quite similar to the microstructures shown in figure 1. More importantly, no differences could be detected between the microstructures of specimens exposed in air and in hydrogen. Such lack of detectable internal porosity, cracking, or newly formed phases supports a mechanism of Internal Reversible Hydrogen Embrittlement rather than Hydrogen Reaction Embrittlement.

Rene 41 was given three heat treatments to produce microstructures with three different sizes of the strengthening phase, gamma prime (fig. 14). The average size of the gamma prime precipitates for each of these conditions (HT-1, HT-2, and HT-3) was less than 0.005, 0.02, and 0.2 micrometer, respectively. The micrographs shown in figure 14 illustrate the as-heat-treated conditions of the alloy. Although some additional precipitation of gamma prime occurred during exposure at 650° C, particularly for Rene 41/HT-1, there were no significant microstructural differences between specimens exposed in air or in hydrogen.

Based on the hydrogen-exposure data in figure 9, it is evident that the degree of embrittlement increased as the average size of the gamma prime precipitates increased. This observation is directly opposite the trend suggested previously (ref. 12) for hydrogen embrittlement of Rene 41.

The role of gamma prime precipitates in Rene 41 may be similar to the role of precipitates in maraging steel (ref. 13). It has been suggested that in the latter alloy precipitate surfaces act as sinks or traps for hydrogen, thus decreasing the severity of embrittlement. For Rene 41, an increasing gamma prime size would mean a decreasing amount of precipitate surface area. With less surface area available for trapping, the amount of diffusible hydrogen would increase and result in a greater degree of embrittlement, as shown in figure 9.

A similar rationalization may be valid for the intermediate degree of embrittlement determined in this study for TD-NiCr. The inert dispersoid thoria may act as a sink for some of the hydrogen, thereby reducing the severity of embrittlement. TD-Ni has been reported to be only slightly embrittled at -79° C (-110° F) and not embrittled at room temperature (ref. 14). Although the experimental charging and testing conditions used in that prior investigation were similar to those used in the present investigation, the strength level of the TD-Ni (ref. 14) was only 50 percent of that of the TD-NiCr used in this investigation. This difference in strength levels may account for the susceptibility to embrittlement of the TD-NiCr. However, both these dispersoid-strengthened alloys are much more resistant to embrittlement than unalloyed nickel under similar conditions (ref. 11).

Effect of alloy strength level. - Attempts were made to correlate the severity of embrittlement resulting from hydrogen exposure with the strength level of the alloy/ conditions. For example, as mentioned previously, most alloys in the cold-rolled condition were more severely embrittled than the alloys in the heat-treated condition. For most alloys, the cold-rolled condition did indeed have a higher strength level than did the heat-treated condition. In addition, L-605/HT was the lowest strength alloy tested and it was not embrittled by hydrogen.

However, a general correlation between strength level and severity of embrittlement among all the alloys was not valid. For example, two of the lower strength alloys, TD-NiCr/HT and Hastelloy X/HT were more severely embrittled than several higher strength alloys. In addition, all three cold-rolled conditions of Inconel 718 had equivalent strength levels after exposure in air at 650° C for 1000 hours but were embrittled to varying degrees by an identical exposure in hydrogen.

One additional observation regarding alloy strength level is significant. For many alloy/conditions, differences in strength level after exposures in air and hydrogen exceeded 10 percent. Specifically, after 1000 hours of exposure at 650° C, Inconel 718/20-percent CR, Inconel 718/35-percent CR, Inconel 718/CR plus HT, A-286/CR, A-286/HT, and Udimet 700/CR had tensile strengths at least 10 percent greater after exposure in hydrogen than in air (table III). This is presumably due to interactions between dislocations and hydrogen which produced an increased rate of work hardening but not necessarily an increased susceptibility to hydrogen embrittlement. Furthermore, after 1000 hours of exposure at 650° C, L-605/HT and Hastelloy X/HT had tensile strengths at least 10 percent less after exposure in hydrogen than in air (table III). There is no apparent explanation of this effect, nor is it related to the degree of embrittlement.

Fracture mode. - The most significant result from an extensive scanning electron microscopy investigation of fractured alloy specimens is that hydrogen exposure resulted in only slight changes in fracture mode. A few alloys embrittled by hydrogen exhibited slightly smaller dimple sizes and slightly larger amounts of intergranular fracture than did the same alloys exposed in air.

Some representative scanning electron micrographs of specimens fractured after exposure at 650° C for 1000 hours in air and in hydrogen are presented in figures 15 to 20. Inconel 718/35-percent CR (fig. 15) and TD-NiCr/HT (fig. 17) exhibited slightly smaller dimple sizes and a few indications of intergranular fracture on hydrogen-exposed specimens as compared with air-exposed specimens. More distinct transitions from dimple formation to varying degrees of intergranular fracture were observed for Inconel 718/HT (fig. 16), Rene 41/HT-3 (fig. 18), and A-286/CR (fig. 19). Alloy/conditions with large grain sizes after cold rolling or heat treatment - Udimet 700/HT-1, Udimet 700/HT-2, Udimet 700/HT-3, L-605/CR, and L-605/HT (fig. 20) - fractured in a distinct intergranular mode after both air and hydrogen exposures.

CONCLUDING REMARKS

The results of this investigation suggest that some superalloys may provide reliable service in hydrogen environments not only for aerospace power generating units and propulsion systems operating at elevated temperatures, but also for ground power generation and transmission systems. Although all the alloys tested in this investigation absorbed large quantities of hydrogen during exposure in hydrogen and were embrittled to varying degrees, some of the alloys exhibited sufficient residual ductility to provide reliable service.

It should be pointed out that these alloys were tested in sheet form. Hence, the triaxial stress conditions associated with bulk or notched specimens were minimal. Normally, such triaxial stress conditions accentuate the embrittling effect of interstitially dissolved hydrogen.

In addition, it should be noted that although L-605/HT did not exhibit embrittlement due to hydrogen, the alloy was severely embrittled by the high-temperature exposure in air. L-605, particularly with high silicon contents, is well known to be metallurgically unstable during long-term exposures at elevated temperatures. Both carbides and Laves phase have been reported as being responsible for the embrittlement observed at room temperature (ref. 15). Hence, although L-605/HT was not embrittled by hydrogen, the use of this alloy with its high silicon content at elevated temperatures should be considered with caution.

Mechanism of Embrittlement

This investigation has demonstrated that a wide variety of superalloys in various conditions absorbed large quantities of hydrogen when exposed in hydrogen at elevated temperatures. Most of the alloys were embrittled when subsequently tested at room temperature. Embrittlement of Inconel 718 was more severe at low strain rates than at high strain rates. Ductility of Inconel 718 could be recovered when the hydrogen was outgassed from the alloy at elevated temperatures in either vacuum or air. Although strain-rate sensitivity and recovery of ductility have been shown only for Inconel 718, the author suggests that such behavior may be common to many other superalloys of the types studied in this investigation. The observed analytical and mechanical results are consistent with a mechanism of Internal Reversible Hydrogen Embrittlement due to interstitially dissolved and freely diffusible hydrogen.

Correlation of Results with Disk-Pressure-Test Results

Three of the alloys tested in this investigation were also tested in a parallel investigation (ref. 7). The alloys were from the same heat and lot of sheet stock and were tested in seven cold-rolled and heat-treated conditions that were identical for both investigations. In reference 7 disk-shaped specimens were ruptured in helium and in hydrogen to determine the degree of embrittlement.

Good correlation between the results of the two test methods was obtained. For example, the alloys with high strength and low ductility (Inconel 718/all three amounts of CR, and L-605/CR) were severely embrittled by both test methods. Inconel 718/35-percent CR plus HT, an alloy with high strength and high ductility, was more severely embrittled during disk pressure testing than in the current investigation. A-286/CR and A-286/HT, alloy/conditions of low strength and high ductility, were less severely embrittled during disk pressure testing than in the current investigation.

Scanning electron fractographic examinations of Inconel 718/35-percent CR after fracture by disk pressure testing revealed an intergranular fracture mode for a hydrogen-embrittled specimen. This is in contrast to a dimple fracture mode for a helium-tested baseline specimen. As discussed previously in this report and shown in figure 15, only slight changes in the fracture mode of Inconel 718/35-percent CR were observed for hydrogen-embrittled specimens as compared with air-exposed specimens.

The obvious differences between the sheet tensile and disk pressure testing methods with respect to the initial location, and homogeneity of the hydrogen, as well as the geometrical deformation characteristics of the test specimens, undoubtedly are responsible for the observed differences in fracture modes and relative susceptibilities of the alloys to hydrogen embrittlement. However, the results of these two parallel investigations suggest that the mechanisms of Internal Reversible Hydrogen Embrittlement and Hydrogen Environment Embrittlement are similar. Additional research is required, however, to determine whether in fact this is the case.

SUMMARY OF RESULTS

The susceptibility of seven superalloys to embrittlement by exposure in hydrogen at elevated temperatures was determined. Five nickel-base alloys (Inconel 718, Udimet 700, Rene 41, Hastelloy X, and TD-NiCr), one cobalt-base alloy (L-605), and one iron-base alloy (A-286) were tested in various cold-rolled (CR) and heat-treated (HT) conditions. Specimens were exposed in 0.1-MN/m^2 (15-psi) hydrogen at several temperatures in the range 430° to 980° C for as long as 1000 hours. Embrittlement was determined by mechanically testing the specimens at room temperature at a low strain rate after the hydrogen exposure. For one of the alloys, Inconel 718, the effects of both continuous and cyclic exposures in hydrogen were determined. The effect of stress level applied to the specimens, cyclic frequency, postexposure mechanical strain rate, and postexposure outgassing treatments were determined. The following major results were obtained:

1. The ductility of the alloy/conditions after exposure in hydrogen at 650° C for 1000 hours was chosen as the criterion for ranking susceptibility to hydrogen embrittlement. The results are as follows:

Rene 41/HT-1 Inconel 718/HT A-286/HT	Moderate embrittlement
Hastelloy X/HT Rene 41/HT-2 TD-NiCr/HT Udimet 700/HT-3	Substantial embrittlement
A-286/CR Rene 41/CR Rene 41/HT-3 Inconel 718/0,20,35CR L-605/CR	Severe embrittlement

2. The following alloy/conditions were apparently not embrittled by hydrogen: L-605/HT, Udimet 700/HT-2, Udimet 700/HT-1, and Udimet 700/CR. However, these alloy/conditions had low values of ductility after exposure in air, so their use as structural alloys must be considered with caution.

3. In general, alloys were more severely embrittled when exposed in hydrogen for 1000 hours than for 100 hours. Alloys were usually more severely embrittled in the cold-rolled condition than in the heat-treated condition. However, there was no consistent correlation between the degree of embrittlement and the strength level of the alloys.

4. Substantial concentrations of hydrogen were absorbed by all alloys during exposure in hydrogen. However, no significant changes in microstructural features were observed, and only minor changes in fractographic features were observed in embrittled alloys.

5. For Inconel 718, embrittlement was more severe as the exposure temperature increased from 430° to 760° C. Cyclic exposures appeared to result in slightly more embrittlement than did continuous exposures for the same total exposure time. There was no significant influence of applied stress level during exposure on subsequent embrittlement.

6. For Inconel 718, embrittlement was more severe for specimens tested at low strain rates than at high strain rates. Post-hydrogen-exposure outgassing treatments in air or vacuum at elevated temperatures resulted in hydrogen outgassing and recovery of ductility.

7. For Rene 41, embrittlement was more severe in heat-treated conditions with a large gamma prime precipitate size than in those conditions with a small gamma prime size.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, August 26, 1974,
501-21.

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TABLE I. - COMPOSITION OF ALLOYS

Alloy	Ni	Fe	Co	Cr	W	Mo	Al	Ti	Mn	C	Si	Cb + Ta	P	S	B	V	ThO_2
	Composition, wt. %																
Inconel 718	Bal.	16.74	-----	18.54	-----	3.2	0.68	0.76	0.33	0.05	0.31	5.28	-----	0.007	-----	----	----
Udimet 700 (sheet)	↓	.18	16.1	15.7	-----	5.53	4.18	3.47	.08	.14	<.1	---- ----	-----	-----	0.014	----	----
Udimet 700 (bar)	↓	.31	15.55	14.47	-----	4.32	4.28	3.18	.02	.10	.04	----	0.004	.002	.012	----	----
Rene 41[a]	↓	-----	11.0	19.0	-----	10.0	1.5	3.1	----	.09	----	----	-----	-----	.005	----	----
Hastelloy X	↓	18.87	2.16	21.31	0.62	9.02	----	----	.71	.07	.33	----	.018	.003	-----	----	----
TD-NiCr	↓	-----	-----	19.93	-----	-----	----	----	----	.0154	----	----	-----	.0015	-----	----	2.16
L-605	9.60	1.98	Bal.	20.37	14.49	-----	----	----	1.34	.09	.65	----	.009	.010	-----	----	----
A-286	24.86	Bal.	-----	15.22	-----	1.24	.23	2.12	1.10	.057	.60	----	.028	.007	.0015	0.33	----

[a]Nominal - all others are vendor certified.

TABLE II. - ALLOY CONDITIONS AND HEAT TREATMENTS

Alloy	Vendor[a]	Heat	Thickness		As-received condition[b]	NASA heat treatment in vacuum or argon[b]
			mm	in.		
Inconel 718	Inco	5995E	0.635	0.025	0, 20, and 35 percent cold rolled	HT: 940° C (1725° F)/1 hr, FC; 720° C (1325° F)/8 hr; furnace cool to 620° C (1150° F)/8 hr, AC
Udimet 700 (sheet)	Stellite	67-671	0.457	0.018	Cold rolled	----------------------------------
Udimet 700 (bar)	Allvac	3812	2.5	0.10	Hot-rolled bar, 3 cm by 2 cm	HT-1: 1175° C (2150° F)/4 hr, AC; 1080° C (1975° F)/4 hr, AC; 845° C (1550° F)/24 hr, AC; 760° C (1400° F)/16 hr, AC HT-2: 1175° C (2150° F)/4 hr, AC; 1105° C (2025° F)/4 hr, OQ; 870° C (1600° F)/8 hr, AC; 980° C (1800° F)/4 hr, AC; 650° C (1200° F)/24 hr, AC; 760° C (1400° F)/8 hr, AC HT-3: 1175° C (2150° F)/4 hr, AC; 760° C (1400° F)/16 hr, AC
Rene 41	Haynes-Wallingford	TI-8315	0.546	0.0215	Cold rolled	HT-1: 1065° C (1950° F)/1/2 hr, WQ HT-2: 1065° C (1950° F)/1/2 hr, WQ; 760° C (1400° F)/16 hr, WQ HT-3: 1065° C (1950° F)/1/2 hr, FC; 760° C (1400° F)/16 hr, AC
Hastelloy X	Haynes	4749	0.381	0.015	1175° C (2150° F)/1 hr, RAC	----------------------------------
TD-NiCr	Fansteel	TC 3508	0.685	0.027	1175° C (2150° F)/2 hr, AC	----------------------------------
L-605	Haynes-Wallingford	L-1842	0.635	0.025	25-percent cold rolled	HT: 1230° C (2250° F)/1 hr, AC
A-286	Haynes Wallingford	21467	0.635	0.025	50-percent cold rolled	HT: 980° C (1800° F)/1 hr, AC; 720° C (1325° F)/16 hr, AC

[a]Many alloys obtained from vendors in mid-1960's.
[b]FC = furnace cool; AC = air cool; OQ = oil quench; WQ = water quench; RAC = rapid air cool.

TABLE III. - TENSILE DATA AND HYDROGEN ANALYSES

Exposure conditions				Tensile properties at 23° C			Hydrogen content, ppm by weight
Gas	Temperature, °C	Number of cycles	Time per cycle, hr	Ultimate strength		Elongation, percent	
				MN/m^2	ksi		
Inconel 718/0-percent cold rolled							
Air	---	--	----	1120	163	34	----
Air	---	--	----	1020	148	[a]32	----
Air	430	1	100	1050	153	20	----
Hydrogen	430	1	100	1030	150	19	----
Hydrogen	430	33	3	1020	148	18	----
Air	540	1	100	1160	169	18	----
Hydrogen	540	1	100	1100	160	14	----
Hydrogen	540	33	3	1040	151	13	----
Air	650	1	100	1480	215	11	----
Air			1000	1450	211	9	----
Hydrogen			100	1450	211	6	----
			1000	1400	203	2	----
		33	3	1320	191	6	----
		33	3	1380	200	[b]9	----
		33	3	1360	197	[c]9	----
Air	760	1	100	1340	194	12	----
Hydrogen	760	1	100	1410	205	8	----
Hydrogen	760	33	3	1450	211	6	----
Inconel 718/0-percent cold rolled and heat treated							
Air	---	--	----	1590	231	20	----
Air	430	1	100	1560	227	20	----
Hydrogen	430	1	100	(d)	---	--	----
Hydrogen	430	33	3	1560	227	17	----
Air	540	1	100	1610	233	18	----
Hydrogen	540	1	100	1600	232	16	----
Hydrogen	540	33	3	1580	229	14	----
Air	650	1	100	1590	230	18	----
Air			1000	1430	207	15	----
Hydrogen			100	1500	218	14	----
			1000	1610	233	14	----
		33	3	1570	228	15	----
		33	3	1560	226	[b]19	----
		33	3	1580	229	[c]18	----
Air	760	1	100	1340	194	16	----
Hydrogen	760	1	100	1390	201	14	----
Hydrogen	760	33	3	1440	209	10	----

[a]Data from ref. 7.
[b]Outgassed in air, 650° C/3 hr.
[c]Outgassed in vacuum, 650° C/3 hr.
[d]Broke in grip.

TABLE III. - Continued. TENSILE DATA AND HYDROGEN ANALYSES

Exposure conditions				Tensile properties at 23° C			Hydrogen content, ppm by weight
Gas	Temperature, $^\circ$C	Number of cycles	Time per cycle, hr	Ultimate strength		Elongation, percent	
				MN/m^2	ksi		
Inconel 718/20-percent cold rolled							
Air	---	--	----	1240	180	15	----
Air	---	--	----	1180	171	[a]10	----
Air	430	1	100	1280	186	18	----
Hydrogen	430	1	100	1260	183	15	----
Hydrogen	430	33	3	1270	184	16	----
Air	540	1	100	1380	200	14	----
Hydrogen	540	1	100	1310	190	13	----
Hydrogen	540	33	3	1320	192	12	----
Air	650	1	100	1648	239	10	----
			100	1650	240	9	----
			100	1660	241	[e]8	----
			1000	1450	210	7	----
Hydrogen			100	1650	239	6	----
			100	1630	237	6	----
			1000	1680	244	4	----
		33	3	1590	230	4	----
				1620	235	4	----
				1610	233	5	----
				1610	233	[e]6	----
				1610	234	[e]6	----
				1630	237	[c]6	----
				1610	233	[c]8	----
				1610	233	[b]7	----
				1610	234	[f]5	----
				1620	235	[f]5	----
Air	760	1	100	1180	171	14	----
Air		1	100	1360	197	14	----
Hydrogen		1	100	1410	205	6	----
Hydrogen		33	3	1450	211	6	----
Hydrogen		33	3	1510	219	5	----

[a]Data from ref. 7.
[b]Outgassed in air, 650°/3 hr.
[c]Outgassed in vacuum 650° C/3 hr.
[e]Tensile tested at 1 cm/min.
[f]Stored in air, 23° C/30 days.

TABLE III. - Continued. TENSILE DATA AND HYDROGEN ANALYSES

Exposure conditions				Tensile properties at 23° C			Hydrogen content, ppm by weight
Gas	Temperature, °C	Number of cycles	Time per cycle, hr	Ultimate strength		Elongation, percent	
				MN/m²	ksi		
Inconel 718/20-percent cold rolled and heat treated							
Air	---	--	----	1520	220	21	--------
Air	430	1	100	1490	216	20	--------
Hydrogen	430	1	100	1450	211	15	--------
Hydrogen	430	33	3	1470	213	17	--------
Air	540	1	100	1520	220	19	--------
Hydrogen		1	100	1500	217	18	--------
Hydrogen		1	100	1500	217	18	--------
Hydrogen		33	3	1470	214	16	--------
Air	650	1	100	1500	218	20	--------
Air			100	1510	219	19	--------
Air			1000	1350	196	15	--------
Hydrogen			100	1480	215	16	--------
			100	1500	217	16	--------
			1000	1510	219	14	--------
		33	3	1480	215	11	--------
Air	760	1	100	1290	187	18	--------
Hydrogen	760	1	100	1320	191	16	--------
Hydrogen	760	33	3	1380	200	12	--------
Inconel 718/35-percent cold rolled							
Air	---	--	----	1450	210	5	--------
Air	---	--	----	1390	202	[a]3	--------
Air	430	1	100	1450	211	9	--------
Hydrogen	430	1	100	1470	213	9	--------
Hydrogen	430	33	3	1450	211	9	4, 4
Air	540	1	100	1560	227	7	--------
Hydrogen	540	1	100	1600	232	6	--------
Hydrogen	540	33	3	1490	216	6	--------
Air	650	1	100	1800	261	4	2, 2
Air			100	----	---	--	[g]1, 2
Air			1000	1440	209	8	--------
Hydrogen			100	1810	262	1	--------
			1000	1810	263	1	--------
		10	10	----	---	--	5, 5
		10	10	----	---	--	[b]1, 1
		10	10	----	---	--	[c]1, 2
		33	3	1790	259	2	4, 5, 5, 5
				----	---	--	5, 5, 5, 6
				----	---	--	[b]2, 2
				----	---	--	[c]1, 1, 2
				----	---	--	[g]4, 4, 5, 5
				----	---	--	[h]5, 5
Air	760	1	100	1400	203	11	--------
Hydrogen		1	100	1390	201	6	--------
Hydrogen		33	3	1460	212	6	6, 6
Hydrogen		33	3	1450	210	6	--------

[a]Data from ref. 7.
[b]Outgassed in air, 650°/3 hr.
[c]Outgassed in vacuum, 650° C/3 hr.
[g]Stored in liquid nitrogen until analysis.
[h]Stored in air, 23° C/1 year.

TABLE III. - Continued. TENSILE DATA AND HYDROGEN ANALYSES

Exposure conditions				Tensile properties at 23° C			Hydrogen content, ppm by weight
Gas	Temperature, °C	Number of cycles	Time per cycle, hr	Ultimate strength		Elongation, percent	
				MN/m²	ksi		
Inconel 718/35-percent cold rolled and heat treated							
Air	---	--	----	1520	221	20	-----
Air	---	--	----	1430	207	[a]16	-----
Air	430	1	100	1520	220	21	-----
Hydrogen	430	1	100	1470	213	15	-----
Hydrogen	430	33	3	1510	219	18	-----
Air	540	1	100	1520	221	20	-----
Hydrogen	540	1	100	1520	221	19	-----
Hydrogen	540	33	3	1520	220	16	-----
Air	650	1	100	1530	222	21	-----
Air		1	1000	1360	197	16	-----
Hydrogen		1	100	1500	218	17	-----
Hydrogen		1	1000	1540	223	14	-----
Hydrogen		33	3	1590	230	14	-----
Air	760	1	100	1280	185	19	-----
Hydrogen	760	1	100	1340	194	16	-----
Hydrogen	760	33	3	1390	201	12	-----
Udimet 700/cold rolled							
Air	650	1	100	1320	191	5	4,5
Hydrogen		1	100	1470	213	4	17,17
Hydrogen		33	3	----	---	--	14,14
Air		1	1000	1660	241	3	-----
Hydrogen		1	1000	1650	239	3	20,20
Udimet 700/heat treated (HT-1)							
Air	650	1	100	1190	172	5	-----
Hydrogen	650	1	100	1160	169	5	-----
Udimet 700/heat treated (HT-2)							
Air	650	1	100	1150	167	7	-----
Hydrogen	650	1	100	1160	169	7	-----
Udimet 700/heat treated (HT-3)							
Air	650	1	100	1310	190	11	-----
Hydrogen	650	1	100	1190	172	6	-----

[a]Data from ref. 7.

TABLE III. - Continued. TENSILE DATA AND HYDROGEN ANALYSES

Exposure conditions				Tensile properties at 23° C			Hydrogen content, ppm by weight
Gas	Temperature, °C	Number of cycles	Time per cycle, hr	Ultimate strength		Elongation, percent	
				MN/m^2	ksi		
Rene 41/cold rolled							
Air	---	--	----	1050	152	34	----
Air	650	1	20	1160	168	12	----
Air			1000	1200	174	7	3,3
Hydrogen			20	1030	150	7	----
			20	1050	152	7	----
			1000	1180	171	3	----
		33	3	----	---	--	8,9
	980	28	3	----	---	--	7,7
Rene 41/heat treated (HT-1)							
Air	---	--	----	900	131	50	----
Air	650	1	20	1150	167	25	----
Air			1000	1210	176	24	----
Hydrogen			20	1080	156	24	----
Hydrogen			20	1080	156	26	----
Hydrogen			1000	1190	173	18	----
Rene 41/heat treated (HT-2)							
Air	---	--	----	1290	187	21	----
Air	650	1	20	1330	193	18	----
Air			1000	1280	186	16	----
Hydrogen			20	1280	185	12	----
			20	1280	185	13	----
			75	1320	191	13	----
			1000	1290	187	9	----
Rene 41/heat treated (HT-3)							
Air	---	--	----	1250	181	20	----
	650	1	20	1270	184	18	----
			100	1210	175	14	----
			1000	1200	174	13	----
Hydrogen			20	1220	177	14	----
			20	1230	178	14	----
			75	1240	180	14	----
			1000	1170	170	2	----
Hastelloy X/heat treated							
Air	---	--	----	880	127	[i]38	----
Air	650	1	100	850	123	18	2,2
Hydrogen		1	100	780	113	16	4,4
Hydrogen		33	3	---	---	--	5,6
Air		1	1000	900	130	21	----
Hydrogen		1	1000	700	101	10	5,5
Hydrogen	980	28	3	---	---	--	6,9

[i]Vendor-certified data.

TABLE III. - Concluded. TENSILE DATA AND HYDROGEN ANALYSES

Exposure conditions				Tensile properties at 23° C			Hydrogen content, ppm by weight
Gas	Temperature, °C	Number of cycles	Time per cycle, hr	Ultimate strength		Elongation, percent	
				MN/m²	ksi		
TD-NiCr/heat treated							
Air	---	--	----	830	120	[i]22	----
Air	650	1	100	870	126	14	1, 2
Hydrogen	↓	1	100	850	124	11	5, 5
Hydrogen	↓	33	3	---	---	--	8, 9
Hydrogen	↓	33	3	---	---	--	[h]5, 6
Air	↓	1	1000	880	127	16	----
Hydrogen	↓	1	1000	810	118	8	6, 6
Hydrogen	980	28	3	---	---	--	5, 6
Hydrogen	980	28	3	---	---	--	[h]6, 7
L-605/cold rolled							
Air	---	--	----	1820	264	[a]2	----
Air	650	1	100	1440	209	2	3, 3
Hydrogen	↓	1	100	1300	188	(d)	3, 3
Hydrogen	↓	33	3	----	---	--	5, 5
Air	↓	1	1000	1420	206	3	----
Hydrogen	↓	1	1000	----	---	(d)	3, 4
Hydrogen	980	28	3	----	---	--	5, 6
L-605/heat treated							
Air	650	1	100	590	86	[j]12	----
Hydrogen	↓	↓	100	610	88	[j]12	2, 3
Air	↓	↓	1000	680	99	3	----
Hydrogen	↓	↓	1000	580	84	[j]3	3, 3
A-286/cold rolled							
Air	---	--	----	1010	147	[a]33	----
Air	650	1	100	1410	205	7	2, 3
Hydrogen	↓	1	100	1440	209	5	7, 8
Hydrogen	↓	33	3	----	---	--	5, 6
Air	↓	1	1000	990	143	15	----
Hydrogen	↓	1	1000	1290	187	3	7, 7
A-286/heat treated							
Air	---	--	----	1070	155	[a]20	----
Air	650	1	100	1210	175	19	----
Hydrogen	↓	↓	100	1210	176	12	7, 7
Air	↓	↓	1000	1050	152	18	----
Hydrogen	↓	↓	1000	1220	177	11	6, 7

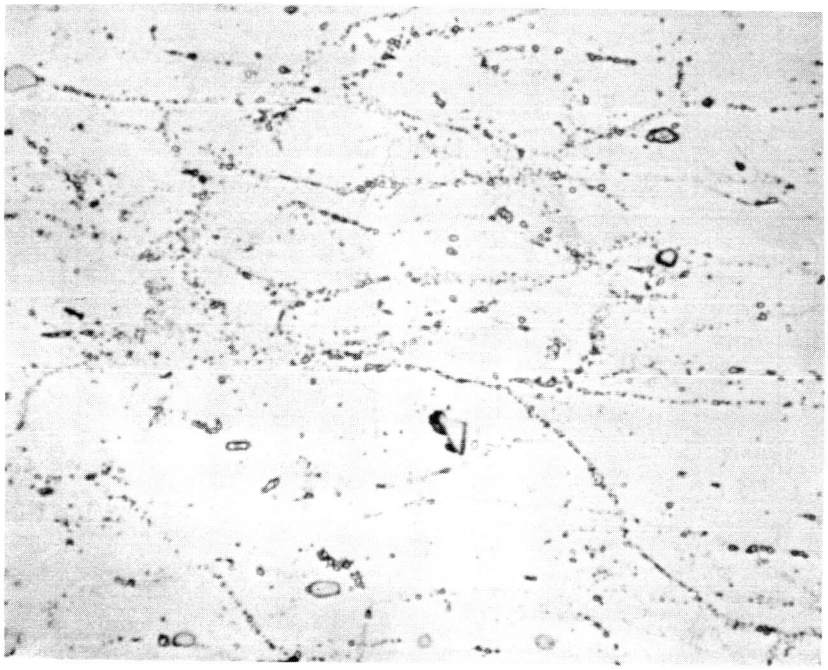
[a]Data from ref. 7.
[d]Broke in grip.
[h]Stored in air, 23° C/1 year.
[i]Vendor-certified data.
[j]Numerous edge cracks during tensile testing.

TABLE IV. - RESIDUAL DUCTILITY OF ALLOYS AFTER

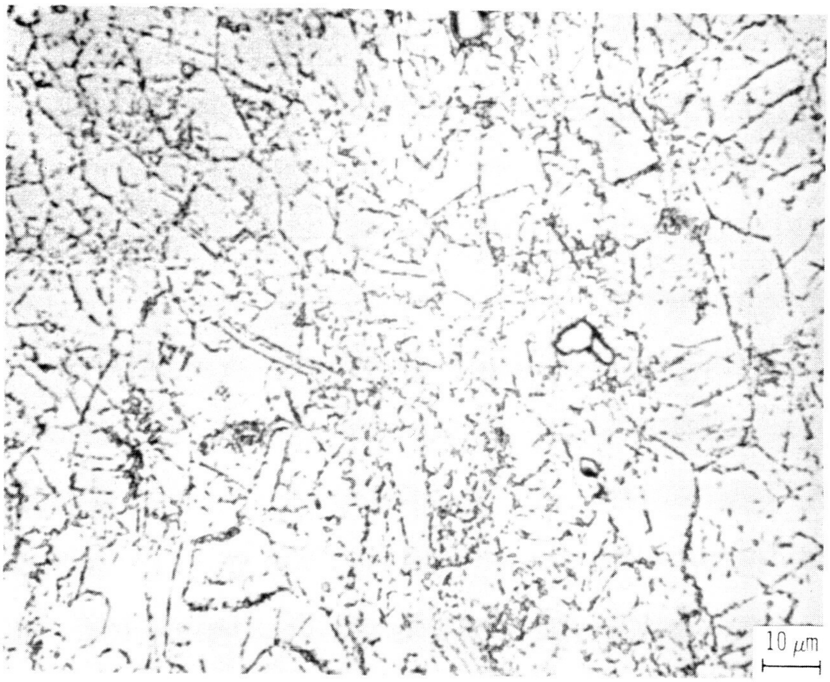
EXPOSURE AT 650° C FOR 1000 HOURS

Alloy/condition[a]	Air exposure	Hydrogen exposure	
	Elongation at 23° C, percent		
Rene 41/HT-1	24	18	Moderate embrittlement
Inconel 718/HT	16	[b]14	
A-286/HT	18	11	
Hastelloy X/HT	21	10	Substantial embrittlement
Rene 41/HT-2	16	9	
TD-NiCr/HT	16	8	
Udimet 700/HT-3	11	[b]6	
A-286/CR	15	3	Severe embrittlement
Rene 41/CR	7	3	
Rene 41/HT-3	13	2	
Inconel 718/0,20,35CR	7-9	1-4	
L-605/CR	3	1	
L-605/HT	3	3	Apparently not embrittled
Udimet 700/HT-2	7	[c]7	
Udimet 700/HT-1	5	[c]5	
Udimet 700/CR	3	[d]3	

[a]HT-1, HT-2, and HT-3 denote various heat treatments and CR denotes cold rolled (table II).
[b]As low as 10 to 12 percent (figs. 5(b), 6(b), and 7(b)).
[c]100-hr exposures with 2.5-mm-thick specimens.
[d]Slight embrittlement observed after 100-hr exposure (fig. 9).

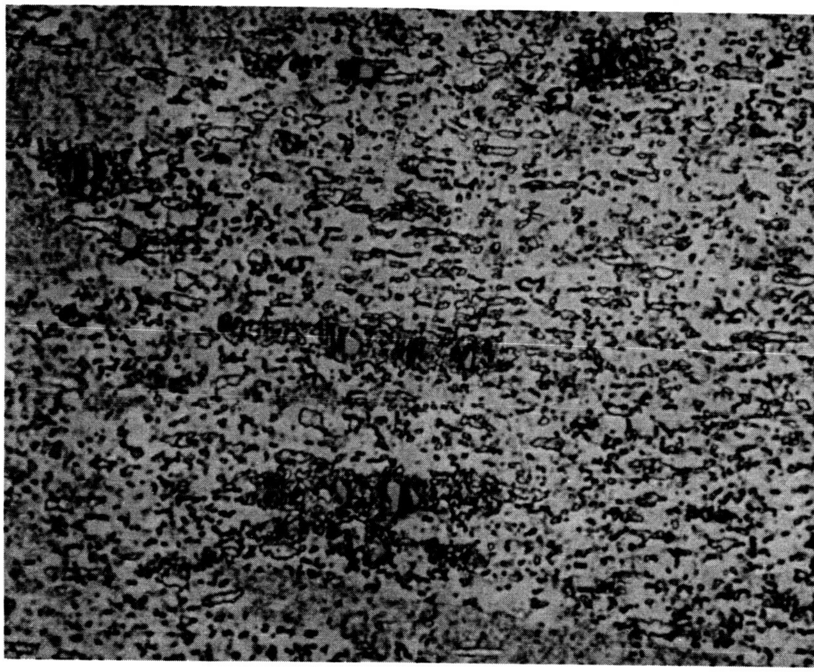


(a) Inconel 718/35-percent cold rolled.

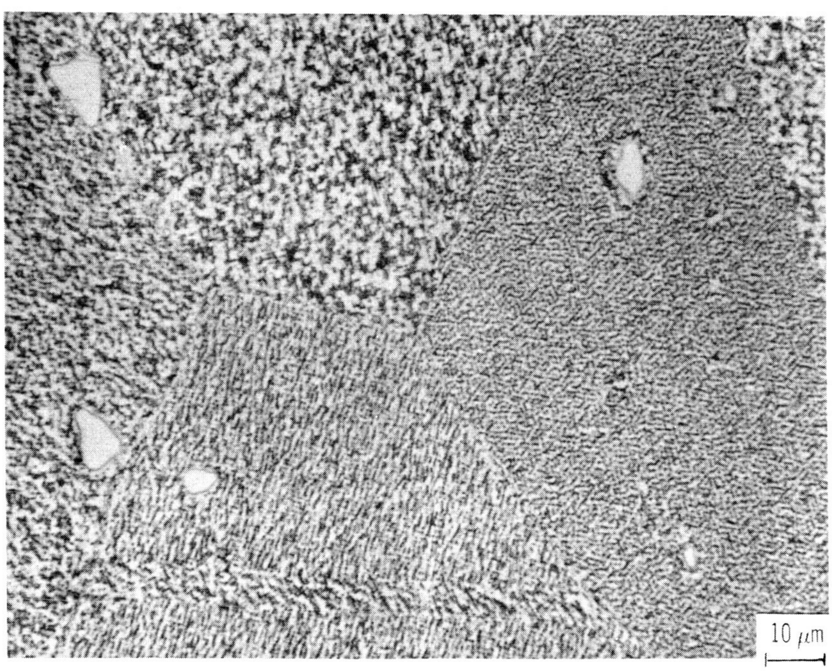


(b) Inconel 718/35-percent cold rolled and heat treated.

Figure 1. - Microstructure of alloys tested in this investigation (see table II for alloy processing conditions).

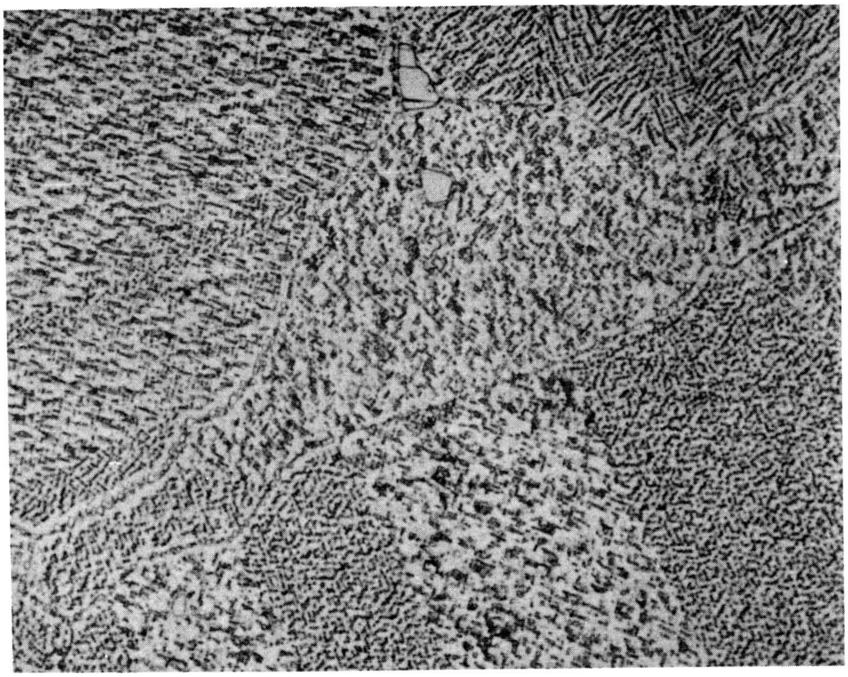


(c) Udimet 700/cold rolled.



(d) Udimet 700/heat treated (HT-1).

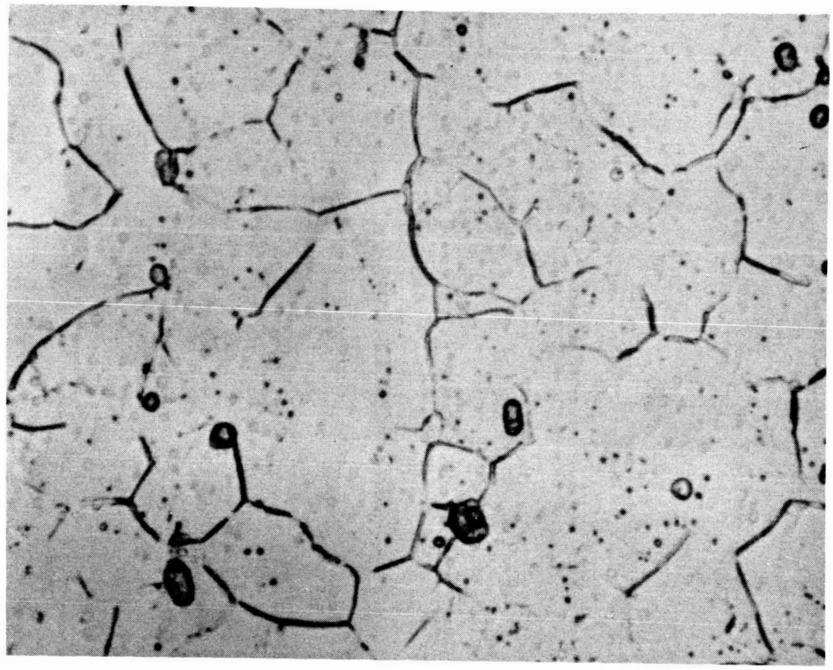
Figure 1. - Continued.



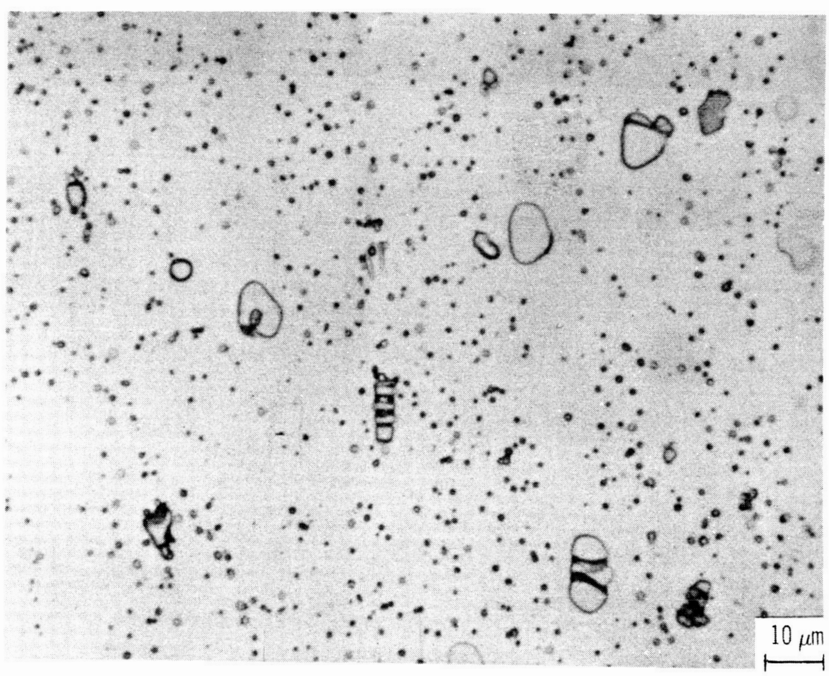
(e) Udimet 700/heat treated (HT-2).

(f) Udimet 700/heat treated (HT-3).

Figure 1. - Continued.

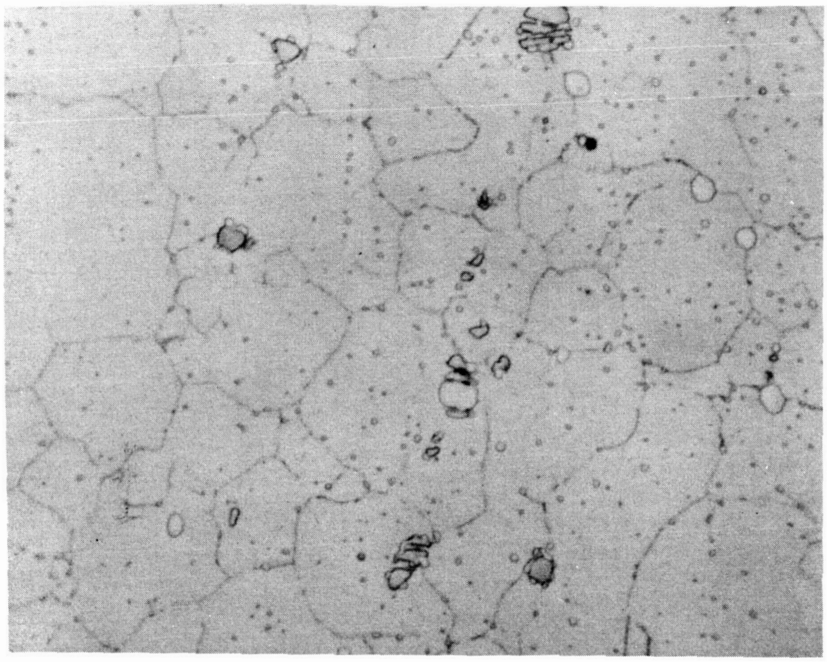


(g) Rene 41/cold rolled.

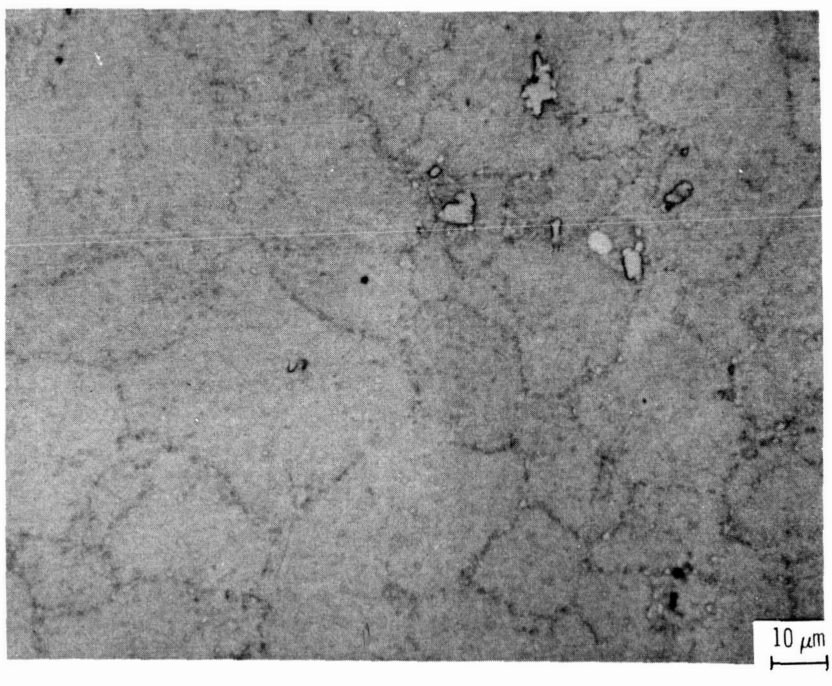


(h) Rene 41/cold rolled and heat treated (HT-1).

Figure 1. - Continued

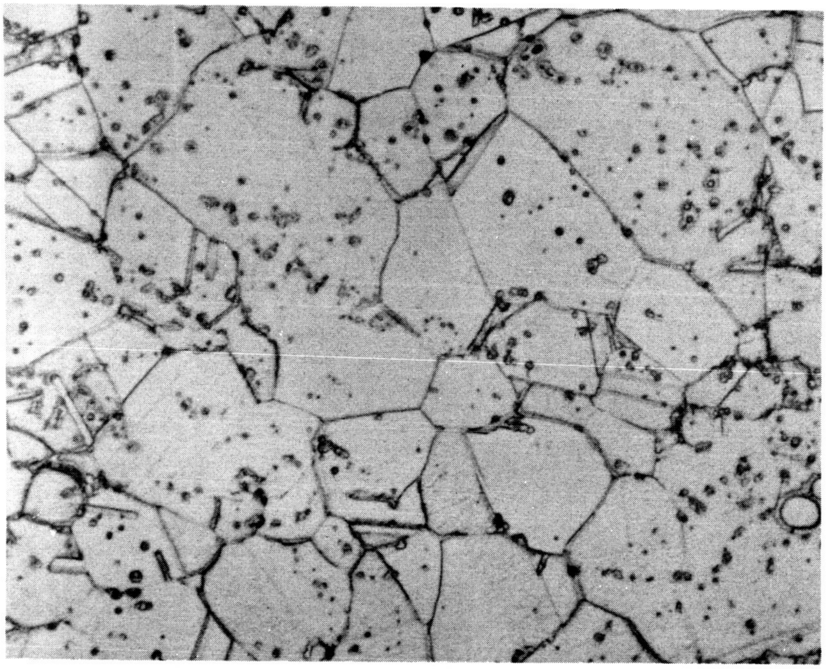


(i) Rene 41/cold rolled and heat treated (HT-2).

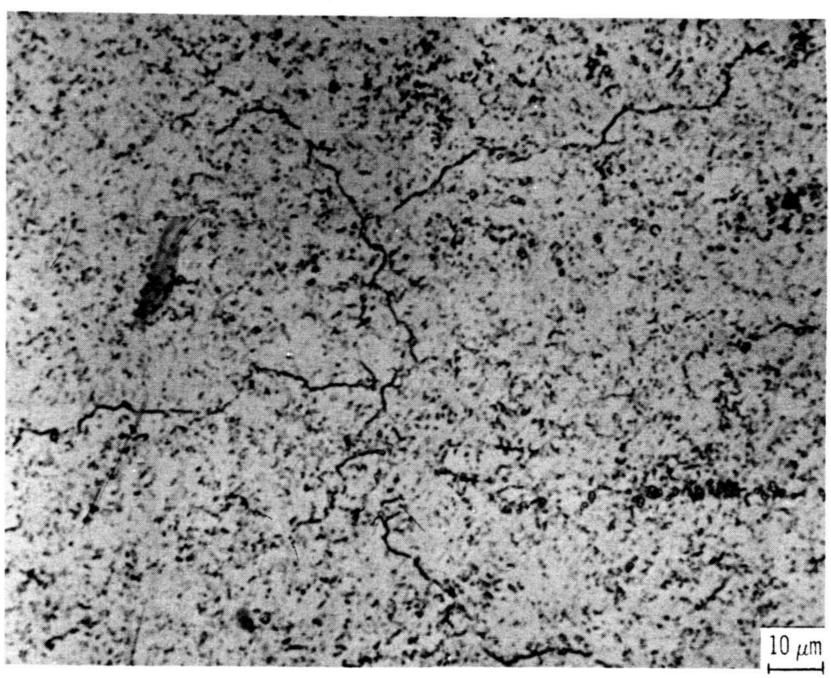


(j) Rene 41/cold rolled and heat treated (HT-3).

Figure 1. - Continued.

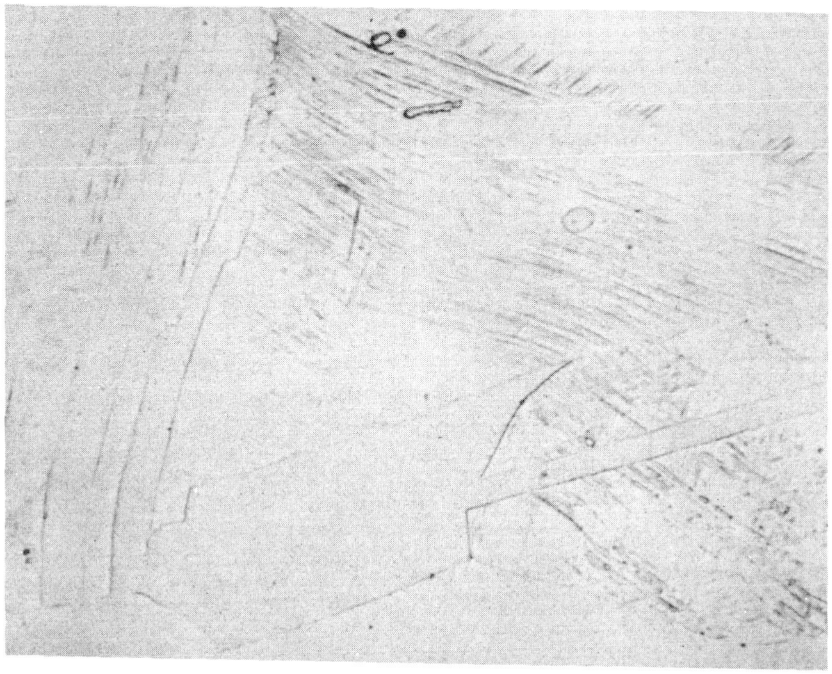


(k) Hastelloy X/heat treated.

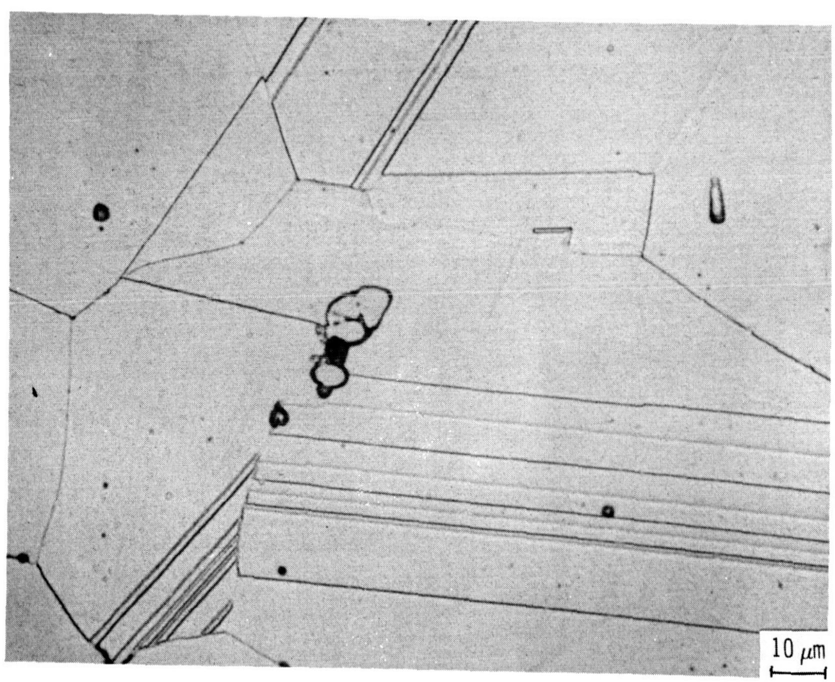


(l) TD-NiCr/heat treated.

Figure 1. - Continued.

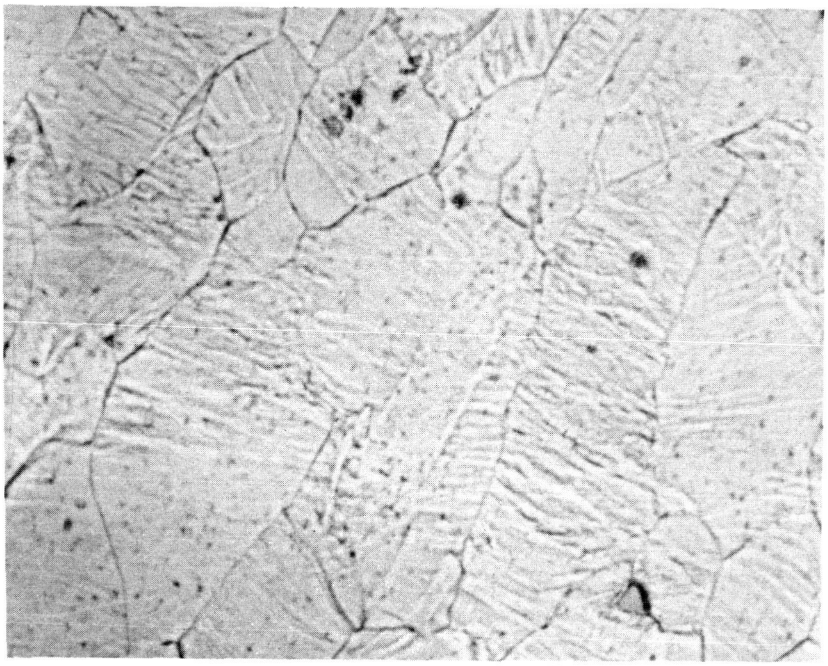


(m) L-605/cold rolled.

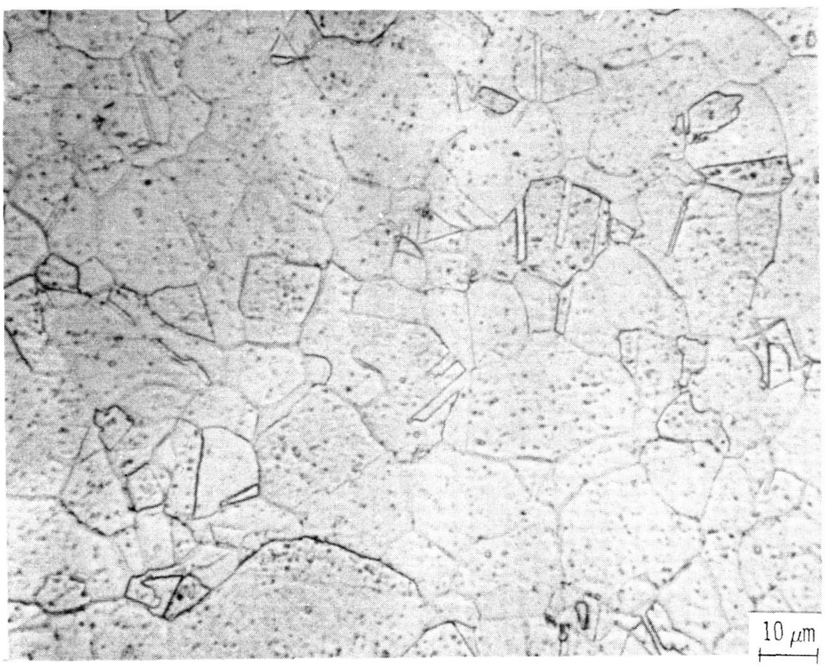


(n) L-605/cold rolled and heat treated.

Figure 1. - Continued.



(o) A-286/cold rolled.



(p) A-286/cold rolled and heat treated.

Figure 1. - Concluded.

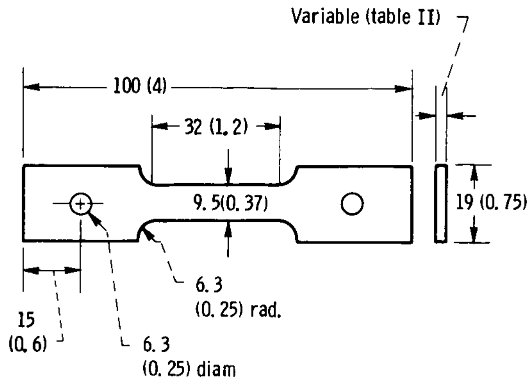


Figure 2. - Sheet specimen used in this investigation. (Dimensions are in mm (in.).)

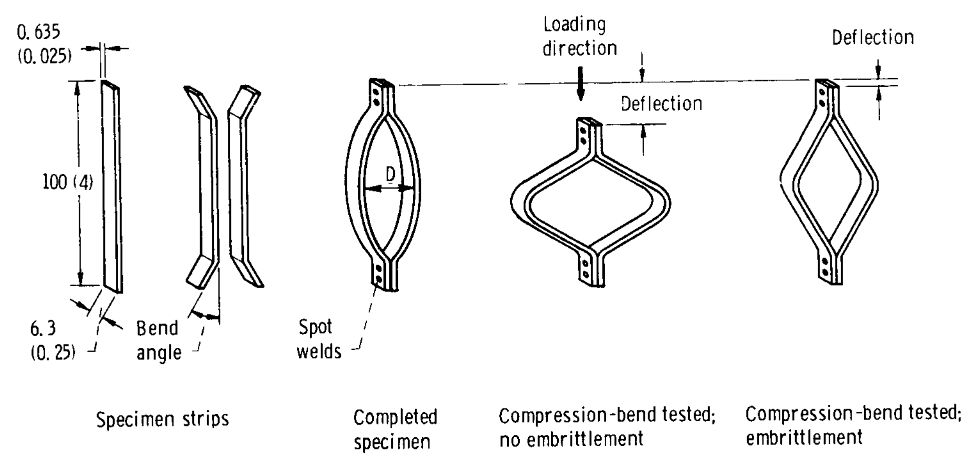


Figure 3. - Self-stressed specimen fabrication and testing procedure. (Dimensions are in mm (in.).)

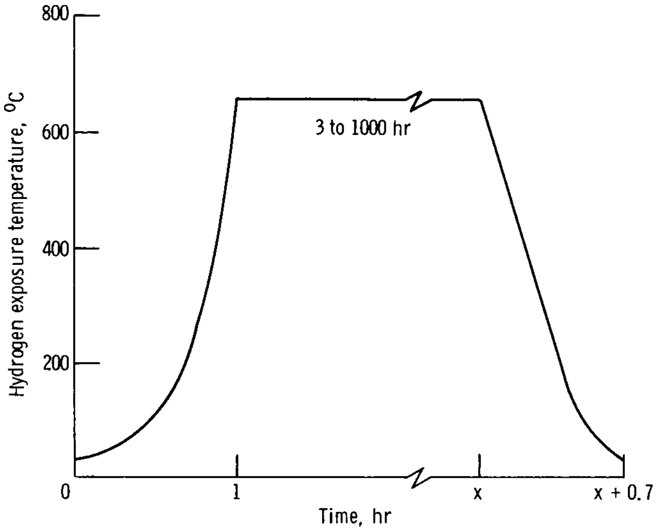


Figure 4. - Temperature-time profile for hydrogen exposures at 650^0 C.

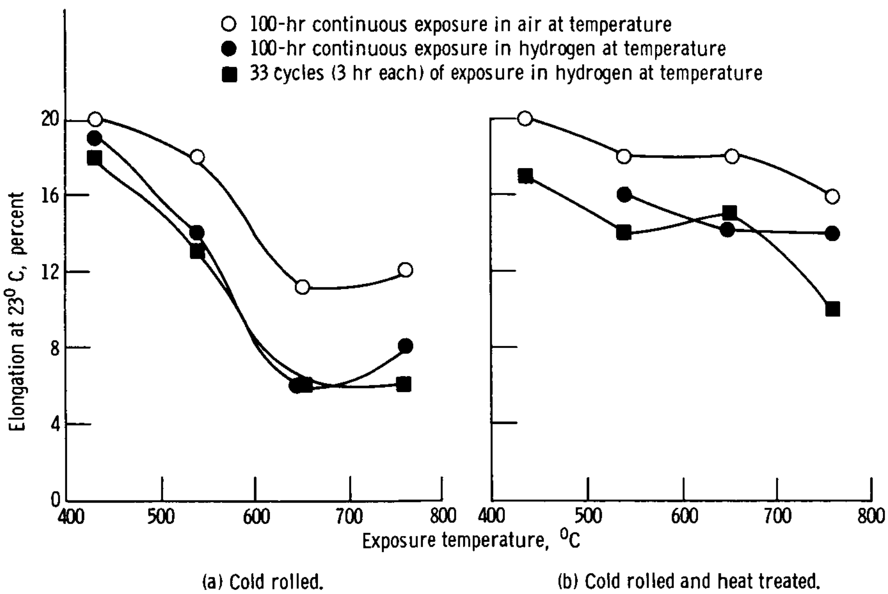


Figure 5. - Effect of exposure temperature on elongation of Inconel 718/0-percent cold rolled. (See table II for alloy processing condition.)

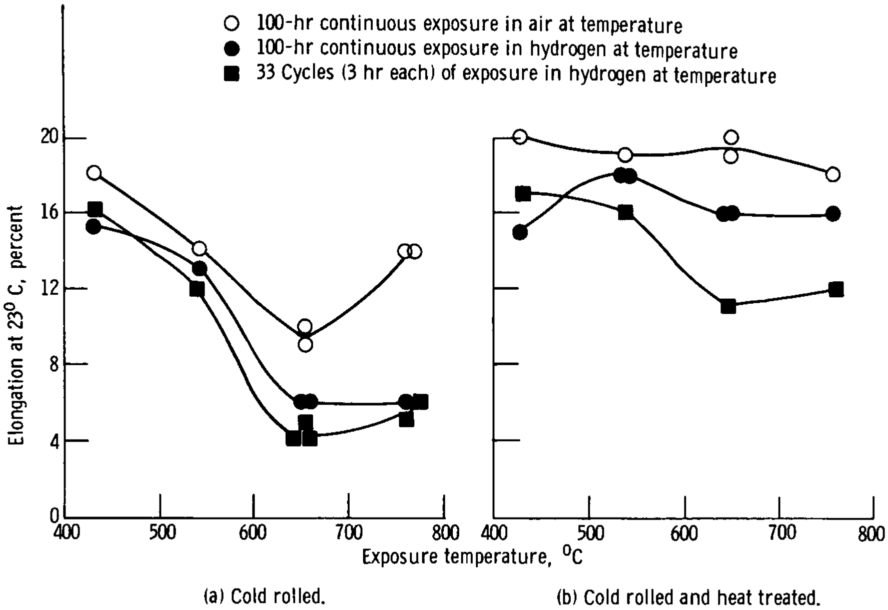


Figure 6. - Effect of exposure temperature on elongation of Inconel 718/20-percent cold rolled (table II).

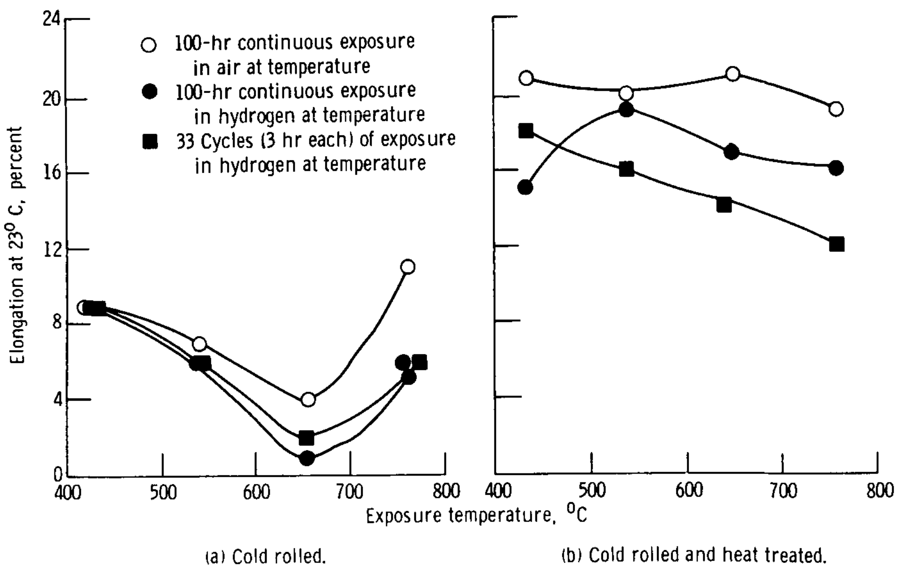


Figure 7. - Effect of exposure temperature on elongation of Inconel 718/35-percent cold rolled (table II).

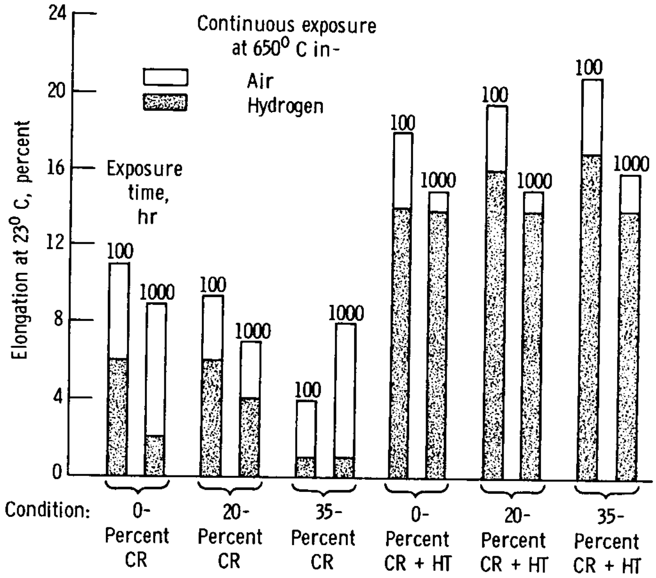


Figure 8. - Elongation of Inconel 718 at room temperature after exposure in air and hydrogen at 650° C for 100 (figs. 5 to 7) and 1000 hours. (CR denotes cold rolled; HT denotes heat treated, see table II.)

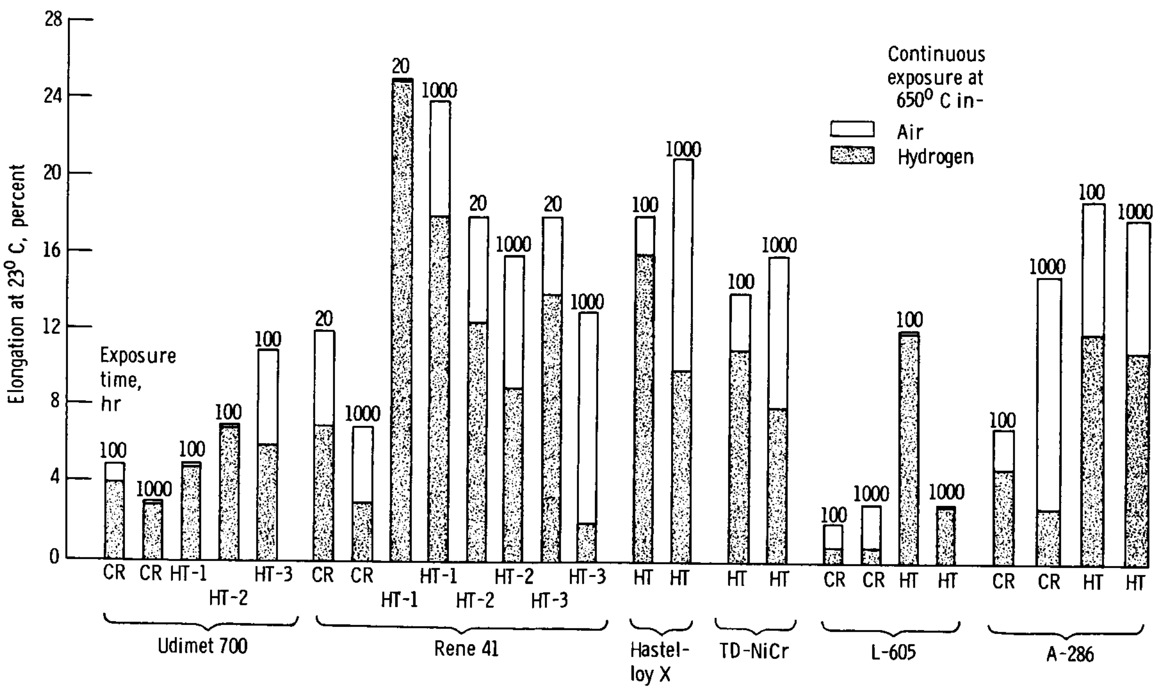


Figure 9. - Elongation of alloys at room temperature after exposure in hydrogen or in air at 650° C for 20, 100, and 1000 hours. (CR denotes cold rolled; HT-1, HT-2, etc., denote various forms of heat treatment, see table II.)

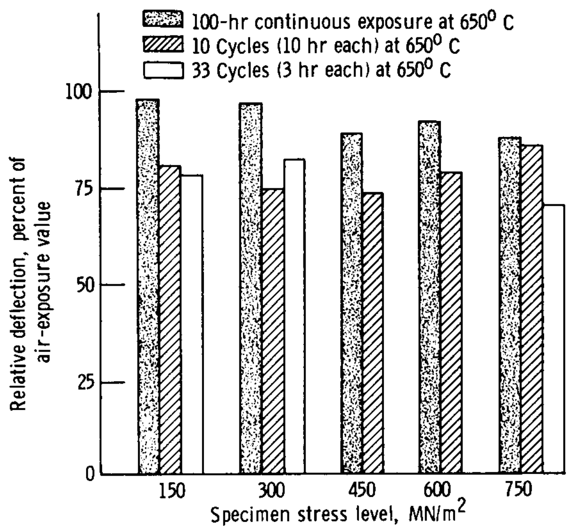


Figure 10. - Effect of applied stress level on degree of embrittlement of self-stressed Inconel 718/35-percent cold rolled, after exposure to hydrogen at 650° C.

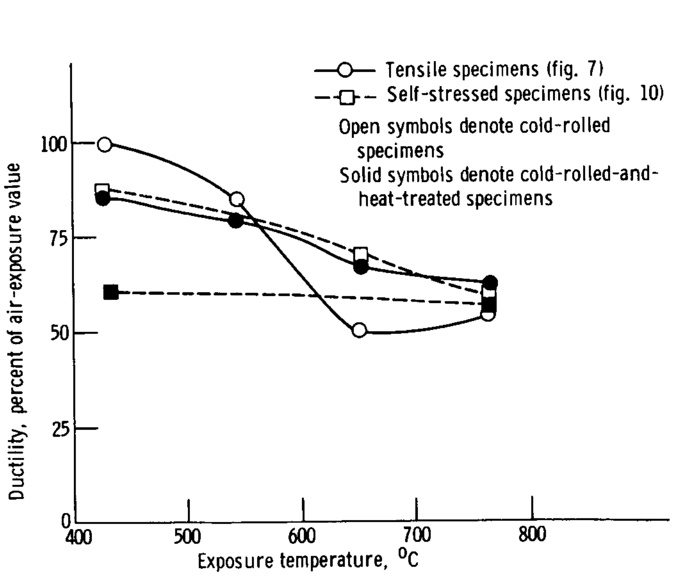


Figure 11. - Comparison of test results obtained with Inconel 718/35-percent cold-rolled tensile and self-stressed (750 MN/m²) specimens - 33 cycles (3 hr each) of exposure at temperature.

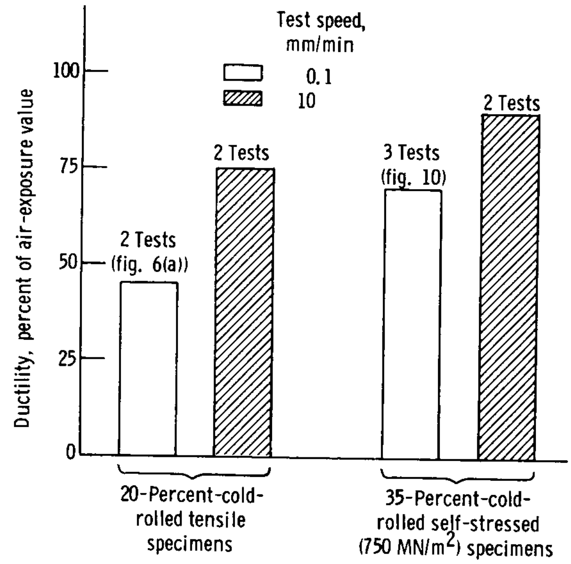


Figure 12. - Effect of test speed (strain rate) on room-temperature ductility of Inconel 718 after exposure to hydrogen - 33 cycles (3 hr each) of exposure at 650° C.

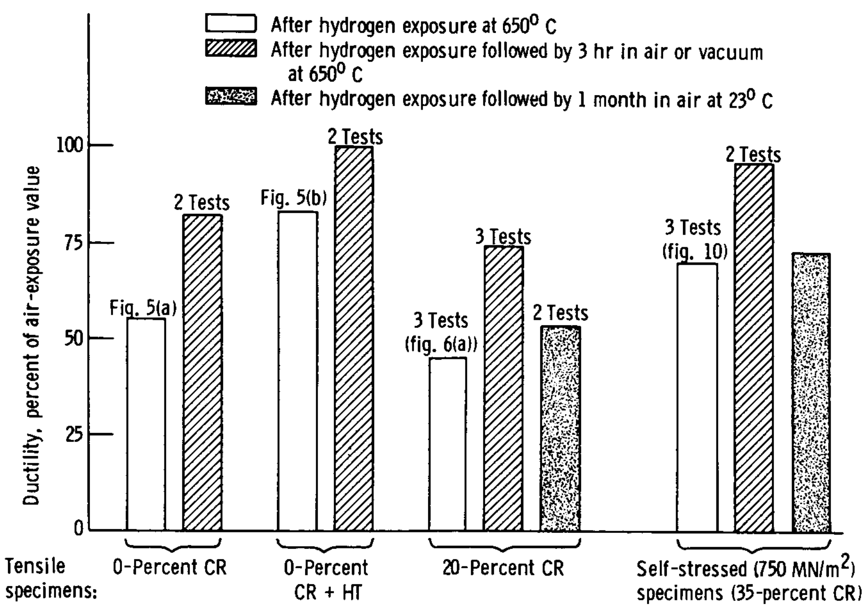
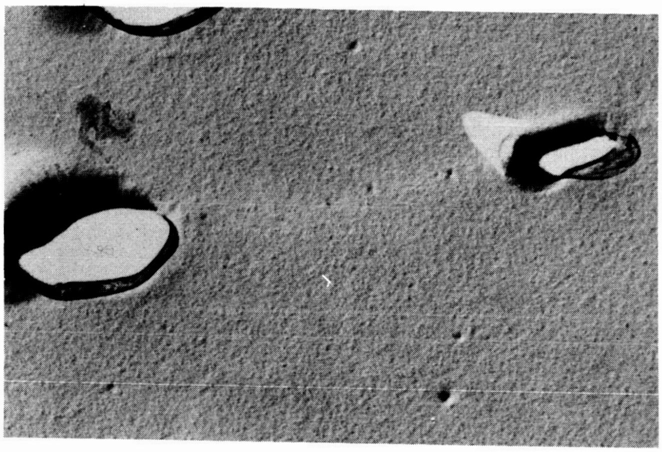
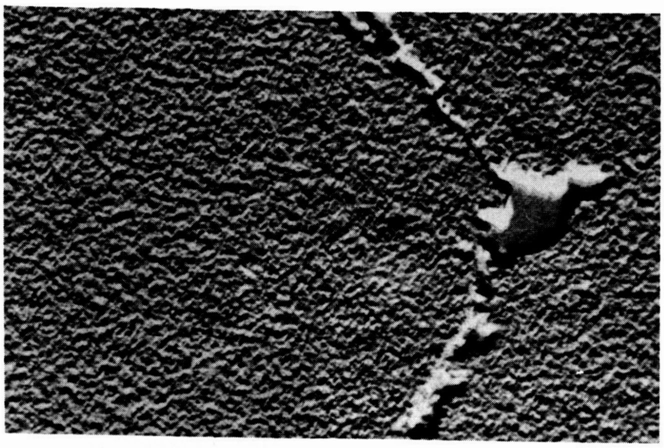


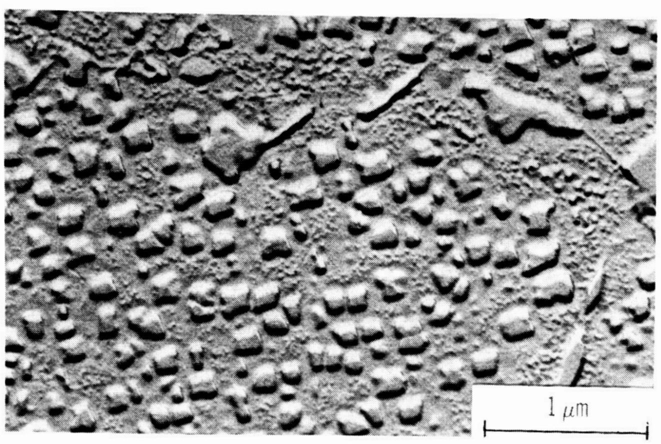
Figure 13. - Effect of postexposure outgassing treatments on room-temperature ductility of Inconel 718 exposed to hydrogen - 33 cycles (3 hr each) of exposure at 650° C. Test speed, 0.1 mm/min.



(a) Rene 41/heat treated (HT-1).

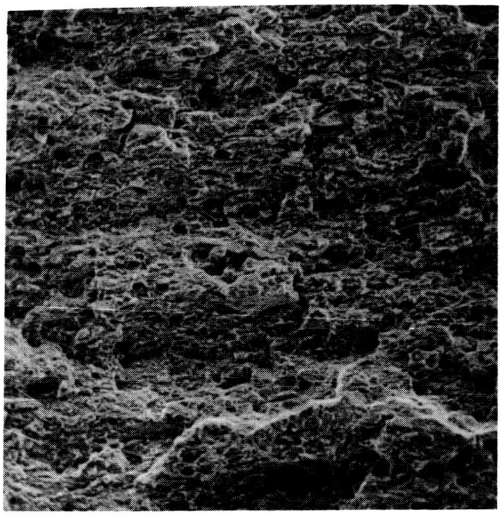


(b) Rene 41/heat treated (HT-2).

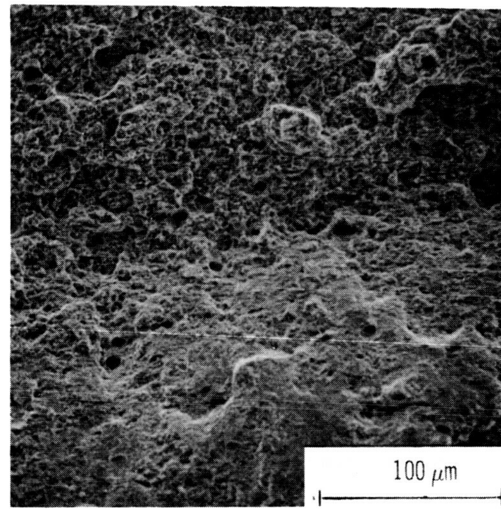


(c) Rene 41/heat treated (HT-3).

Figure 14. - Replica electron micrographs of Rene 41.

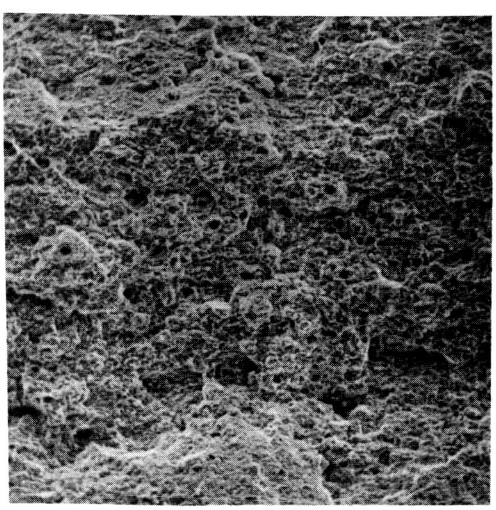


(a) Air exposure.

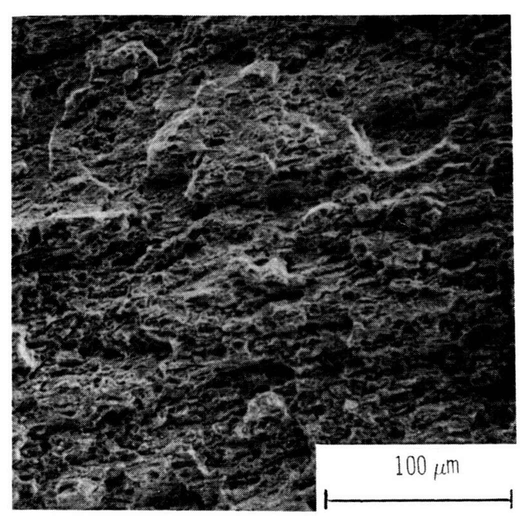


(b) Hydrogen exposure.

Figure 15. - Scanning electron micrographs of fracture surfaces of Inconel 718/35-percent cold rolled, after exposure at 650° C for 1000 hours.

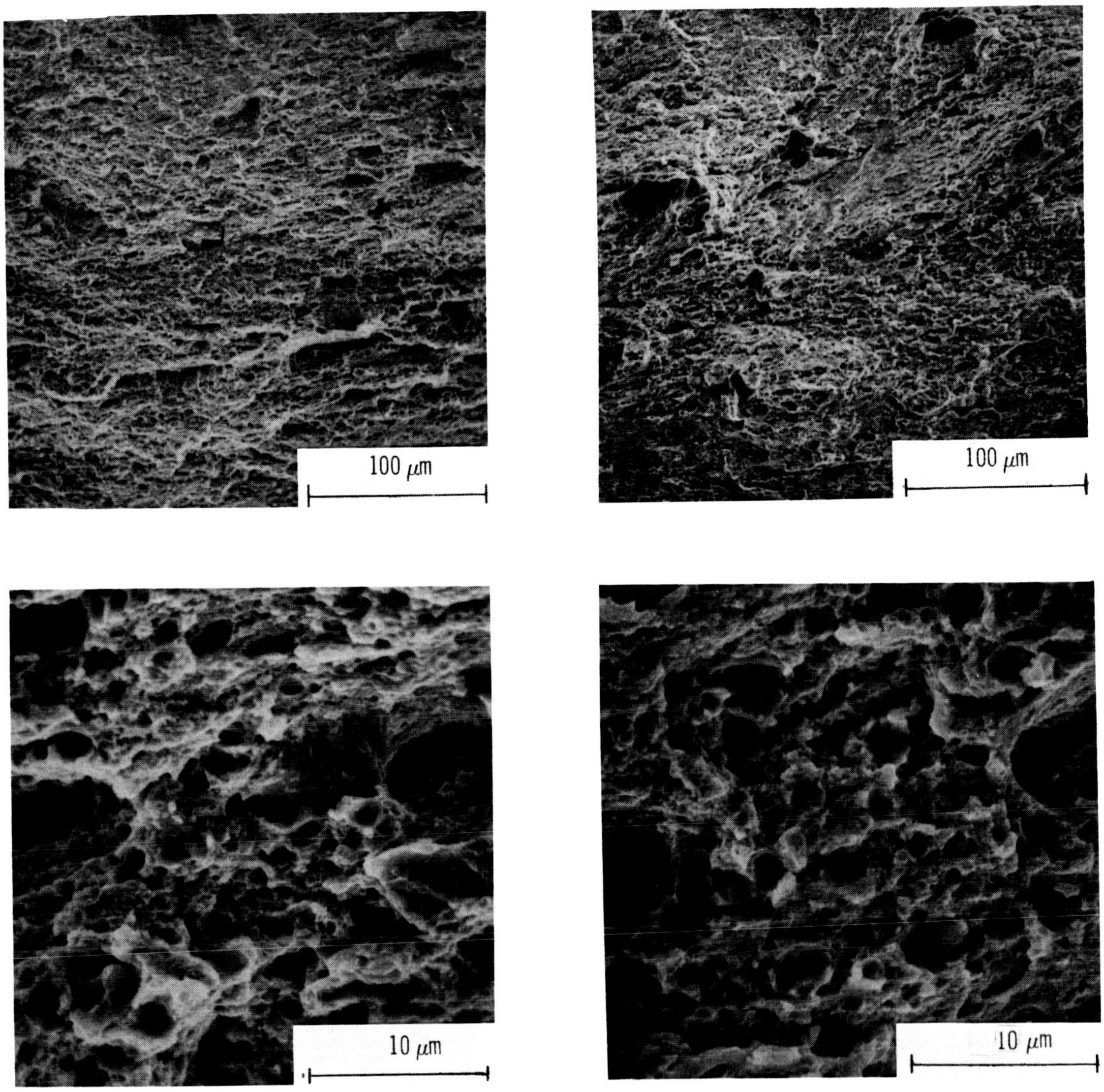


(a) Air exposure.



(b) Hydrogen exposure.

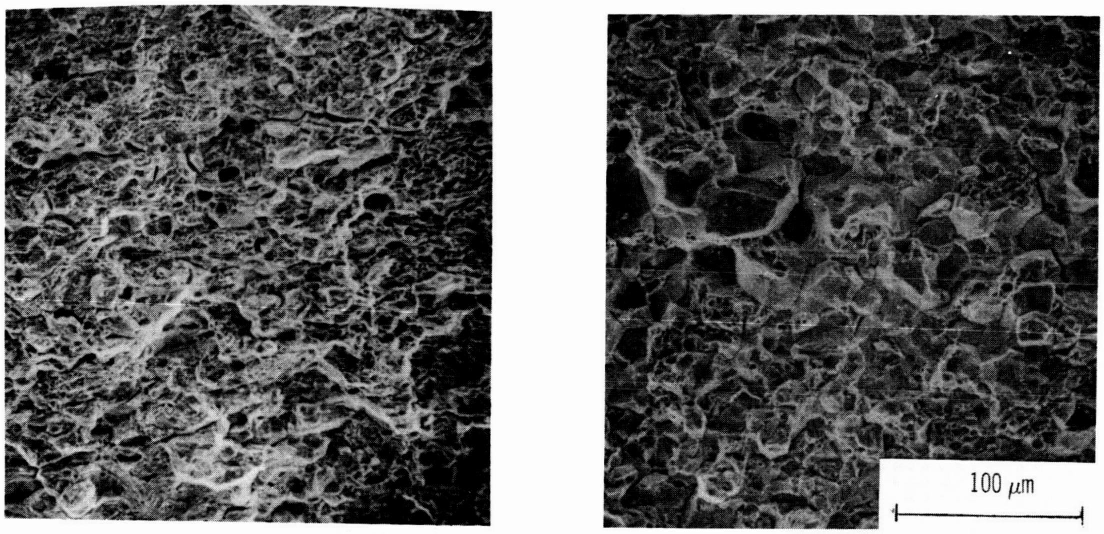
Figure 16. - Scanning electron micrographs of fracture surfaces of Inconel 718/35-percent cold rolled and heat treated, after exposure at 650° C for 1000 hours.



(a) Air exposure.

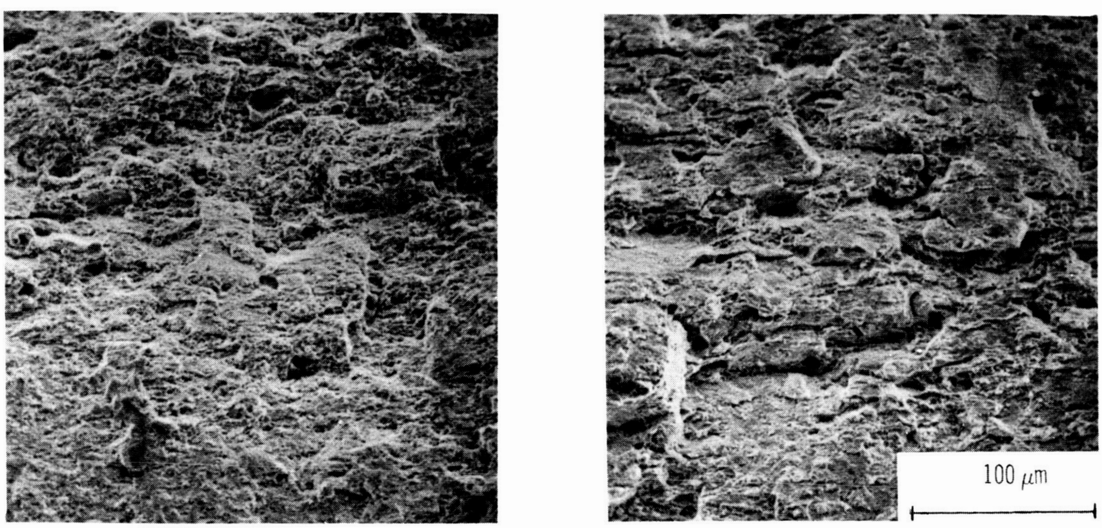
(b) Hydrogen exposure.

Figure 17. - Scanning electron micrographs of fracture surfaces of TD-NiCr/heat treated, after exposure at 650° C for 1000 hours.



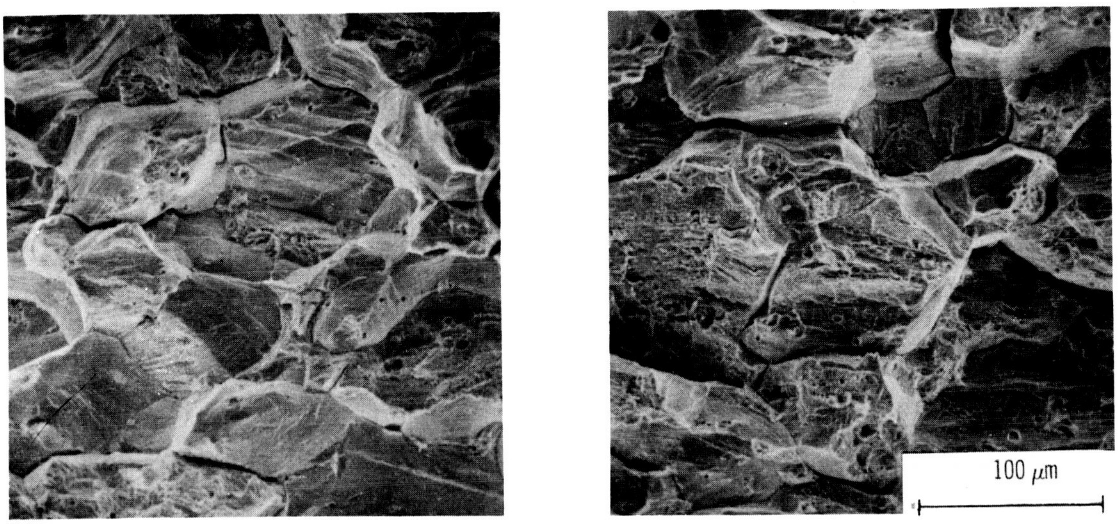
(a) Air exposure. (b) Hydrogen exposure.

Figure 18. - Scanning electron micrographs of fracture surfaces of Rene 41/heat treated (HT-3), after exposure at 650° C for 1000 hours.



(a) Air exposure. (b) Hydrogen exposure.

Figure 19. - Scanning electron micrographs of fracture surfaces of A-286/cold rolled, after exposure at 650° C for 1000 hours.



(a) Air exposure.

(b) Hydrogen exposure.

Figure 20. - Scanning electron micrographs of fracture surfaces of L-605/heat treated, after exposure at 650° C for 1000 hours.

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