

Hydrogen Compatibility of Dispersion-Strengthened Alloys

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Hydrogen compatibility of Ni-2 ThO₂, Ni-20 Cr, Ni-20 Cr-2 ThO₂, and Inconel MA 753 was investigated with both hydrogen charging and tests in high-pressure hydrogen gas. The former were conducted at 194 K as well as at room temperature. Ductility and fractography results showed that recrystallized Ni-20 Cr-2 ThO₂ and MA 753 were not embrittled by hydrogen, while Ni-2 ThO₂ showed small ductility losses with no change in fracture morphology. Annealed Ni-20 Cr-2 ThO₂ and as-received Ni-20 Cr-2 ThO₂ showed no ductility losses in charged hydrogen, but showed large ductility losses in high-pressure hydrogen; no changes in fracture morphology were observed. Ni-20 Cr was markedly embrittled by both sources of hydrogen. These results support the hypothesis of Thompson and Wilcox, that the presence of a dispersoid can confer improved performance in hydrogen.

MANY of the energy alternatives for the future involve handling substantial quantities of hydrogen. These alternatives include not only the much-discussed "hydrogen economy",¹⁻³ with its need to transport and store large quantities of hydrogen, but also such possibilities as coal gasification to produce power gas, containing about 15 pct hydrogen.⁵ A critical aspect of hydrogen-handling systems is the need for high-strength materials in pumps, valves and other parts, because the high-strength steels normally specified for such applications are prone to gross embrittlement by hydrogen. Although some austenitic stainless steels⁴⁻⁶ and aluminum alloys⁵ appear to be compatible with hydrogen, such materials have yield strengths below 500 MN/m² (about 75 ksi). Modest increases above this level have been reported in forged stainless^{4,5} and in marginally-compatible precipitation-strengthened stainless alloys,⁴ but these do not exceed about 700 MN/m² yield strength. Thus the identification of high-strength alloys which are compatible with hydrogen is an important task for development of future energy systems.

At least two approaches to this problem can be identified. One approach is to attempt a modification of the microstructure of precipitation-strengthened stainless alloys to improve compatibility and strength.⁷ The second, which forms the subject of this paper, is to exploit the compatibility conferred by a dispersoid, an effect discovered by Thompson and Wilcox.⁸ Their experiments were on Ni-2 ThO₂, an alloy no stronger than an austenitic stainless steel. However, alloys in the 850 to 1000 MN/m² (up to about 150 ksi) yield strength range have been developed, such as dispersion-strengthened Ni-20 Cr. It would be expected from previous work⁸ that the dispersoid, which apparently confers hydrogen compatibility by providing hydrogen sinks during deformation, would be as effective in Ni-20 Cr-2 ThO₂ as in Ni-2 ThO₂.

Development of dispersion-strengthened alloys has been pursued for high-temperature use, but the present

interest is ambient temperature service in hydrogen. Thus the effort to improve low-temperature strength of dispersion-strengthened alloys by the addition of γ' precipitate strengthening^{9,10} is of particular interest here. The alloy Inconel MA 753* (previously called

*Trade name of International Nickel Co.

IN 853) was selected for investigation. It is produced by a process called mechanical alloying,⁹ thus the designation "MA".

In addition to the aim of evaluating hydrogen compatibility of stronger alloys, it seemed appropriate to investigate the microstructural reasons for behavior in hydrogen. For this purpose, tests on Ni-2 ThO₂ were conducted to evaluate an alternative rationale for fracture morphology in hydrogen.

EXPERIMENTAL PROCEDURE

The Ni-2 vol pct ThO₂ and Ni-20 Cr-2 vol pct ThO₂ alloys were provided by Fansteel, Inc. Commercial names of these alloys are, respectively, TD-Ni and TD-NiCr.* These were received as 7.5 mm plate: the

*Trade names of Fansteel, Inc.; now property of Stellite Division of Cabot Corp.

Ni-2 ThO₂ had been warm-rolled, while the Ni-20 Cr-2 ThO₂ was in the fine-grained, unrecrystallized condition called "specially processed."¹¹ The grain structure of these two materials has been described before.^{8,12} Briefly, the Ni-2 ThO₂ had broad lath-shaped grains about 50 μ m thick (in the plate thickness direction), 100 μ m wide, and 100 to 400 μ m long. The unrecrystallized Ni-20 Cr-2 ThO₂ had an approximately equiaxed grain size of about 4 μ m.

For purposes of comparison to the Ni-20 Cr-2 ThO₂, a Ni-20 Cr alloy was provided by B. A. Wilcox which had been prepared at Fansteel Inc. by powder metallurgy processes (to include amounts of Cr₂O₃ comparable to those found in TD-NiCr). It was received as cold-finished 10 mm bar. The Ni-20 Cr-2.2 Ti-1.1 Al-2 vol pct Y₂O₃ alloy,⁹ Inconel MA 753, was provided by the International Nickel Co. as warm-finished 18 mm round bar. The grains were elongated parallel to the

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Table I. Mechanical Properties of Experimental Materials

| Material | Condition* | Temp, K | Yield Strength, [†] MN/m ² | Ultimate Strength, MN/m ² | Uniform True Strain | RA, pct |
|---------------------------|----------------------|---------|---|---|------------------------|---------|
| Ni-2ThO ₂ | Long. | 295 | 234 | 428 | 0.150 | 74 |
| | | 194 | 251 | 487 | 0.127 | 73 |
| Ni-20Cr | 45 deg Recryst. | 194 | 245 | 509 | 0.230 | 52 |
| | | 295 | 248 | 675 | 0.098 | 57 |
| Ni-20Cr-2ThO ₂ | As-received | 194 | 281 | 763 | 0.273 | 53 |
| | | 295 | 899 | 1037 | 0.111 | 16 |
| | Annealed | 295 | 851 | 1008 | 0.144 | 22 |
| | | 194 | 886 | 1064 | 0.113 | 19 |
| Recryst. | 295 | 866 | 1032 | 0.092 | 13 | |
| | 194 | 914 | 1083 | 0.087 | 16 | |
| MA 753 | Aged and Annealed | 295 | 872 | 1090 | 0.275 | 14 |
| | | 194 | 943 | 1166 | 0.135 | 9 |

*Please see text for details.

[†]Flow stress at 0.002 plastic strain.

bar axis and were roughly rod-shaped. The rod diameters averaged 520 μm , the lengths 1830 μm . The average number of grains across the tensile specimen diameter (see below) was 5.8, enough to ensure polycrystalline behavior.¹³ Prior to heat treatment, all materials were machined into tensile specimens with a gage length of 13 mm and diam of 3 mm. These were longitudinally oriented, except some specimens of Ni-2 ThO₂ which were oriented at 45 deg to the rolling direction.

Materials were tested after heat treatment for 2 h at 1075 K in helium, and, in some cases, in the as-received condition. The heat treatment, referred to below as an "anneal," caused neither recrystallization nor grain growth in the dispersion-strengthened materials. The Ni-20 Cr, however, recrystallized to an equiaxed size of 15 μm . All grain sizes quoted are true volume grain diameters.¹⁴ The Ni-20 Cr-2 ThO₂ was also tested after heat treatment of 1 h at 1575 K in helium to recrystallize it; grain size remained at about 4 μm . Such material is referred to below as "recrystallized." MA 753 was solution treated at 1350 K for 8 h, air cooled, and aged at 980 K for 16 h and air cooled, prior to the annealing heat treatment. Results of tests in air on each of the alloys are shown in Table I.

Two types of hydrogen tests were performed. One of these was testing in air after thermal charging in 1 atm (~ 0.1 MPa) H₂; this produces a uniform hydrogen distribution and is believed not to produce nickel hydride.¹⁵ All charging was conducted at 1075 K, and was terminated by rapid cooling to room temperature (within 3 min) in hydrogen. Based on data for diffusivity and solubility of hydrogen in nickel¹⁶ and Ni-20 Cr,¹⁷ calculations indicated that the charged specimens contained a concentration of 5.4 wt ppm H (Ni-2 ThO₂) or 5.6 wt ppm (Ni-20 Cr, Ni-20 Cr-2 ThO₂, MA 753), and had no hydrogen gradient. Accuracy of the calculated values was verified in spot checks by vacuum extraction analysis.⁸ These concentrations are supersaturations relative to room temperature, since the room temperature solubilities are, respectively, 0.12 wt ppm and 0.5 wt ppm.^{16,17} Tensile tests on charged specimens were always conducted within 30 min of the conclusion of charging.

The second test technique was to strain an uncharged specimen in an environment of 69 MPa (10,000 psi) hydrogen gas at room temperature. This technique, in

some cases, gives results which differ from the results obtained on charged specimens.^{4-6,18}

In addition to room temperature tests, charged and uncharged specimens were tested at 194 K, which is near the temperature of maximum embrittlement of pure nickel.¹⁵ Tests at 194 K were made in a bath of methanol and solid CO₂. Most tests were conducted at an initial strain rate of 1.67×10^{-4} s⁻¹ to facilitate comparison with earlier work.^{8,15}

A scanning electron microscope was used to examine fracture surfaces. All fractography was conducted with the viewing direction normal to the fracture surface. Thin foils were prepared from Ni-20 Cr-2 ThO₂ using an electropolishing solution developed for titanium alloys,¹⁹ while MA 753 foils were thinned in a solution originally used for irradiated stainless steel.²⁰ Foils were examined in a Philips EM 300 electron microscope.

RESULTS

The microstructures of TD-Ni and, to some extent, TD-NiCr are familiar from the work of a number of authors.²¹⁻²⁵ The three conditions of Ni-20 Cr-2 ThO₂ tested in this work are shown in Figs. 1(a) to 1(c). There is no detectable difference between the as-received (Fig. 1(a)) and annealed (Fig. 1(b)) microstructures, while the only changes upon recrystallization (Fig. 1(c)) are the presence of fine annealing twins (not shown in Fig. 1(c)) and a modest reduction in dislocation density. The aged and annealed MA 753 in Fig. 1(d) displays γ' particles (strain contrast) together with the yttria dispersoid (particles with sharp image). Despite the 1075 K anneal after aging, the γ' precipitates in Fig. 1(d) appear coherent with the matrix, which suggests that little overaging has taken place. That conclusion is supported by the agreement between yield strength data in the literature⁹ and in Table I. The dispersoid particle sizes are not as uniform in Fig. 1 as has been reported in other cases.²³⁻²⁵

The material used as a standard of comparison in this study was Ni-20 Cr, just as pure Ni has been used as a standard for tests on Ni-2 ThO₂.⁸ The Ni-20 Cr was found to be embrittled by hydrogen, as Fig. 2 demonstrates. Three of the distinctive indicators of hydrogen behavior, shown in Fig. 2(b), are those which have been described for stainless steel:²⁶ prominent surface

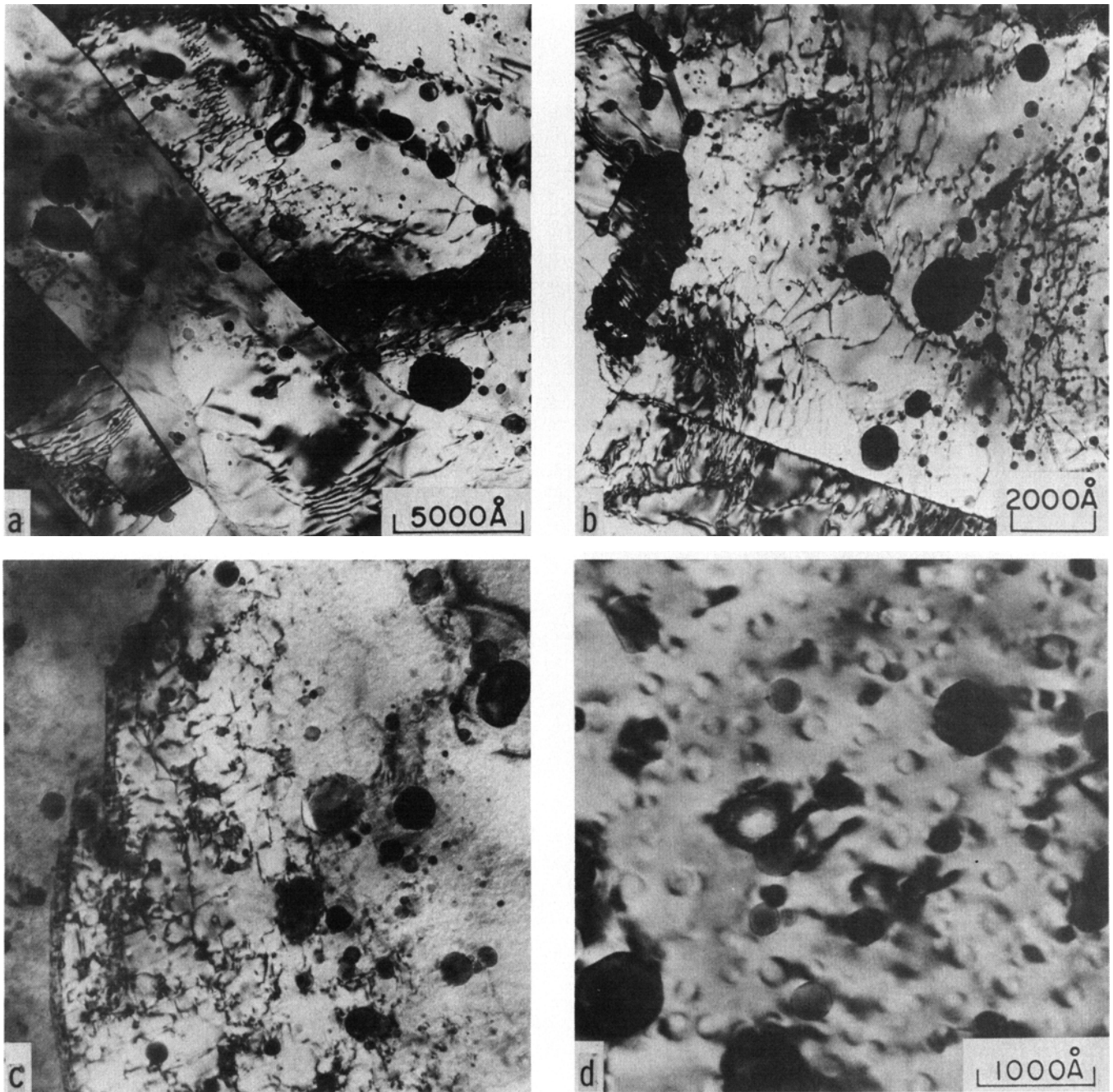


Fig. 1—Transmission electron micrographs of experimental materials. (a) As-received Ni-20Cr-2ThO₂. (b) Material of (a) after 1057K anneal. (c) Material of (a) after recrystallization at 1575 K; same magnification as (b). (d) Aged and annealed MA 753; foil tilted near matrix $\langle 110 \rangle$ to reveal strain contrast at γ' precipitates.

cracking; marked decrease of reduction in area (RA), from 57 pct to 18 pct; and an approximately flat fracture instead of cup-and-cone fracture. There was also secondary cracking on the fracture surface, both on a macroscale (Fig. 2(c)) and on a microscale (Fig. 3(b)). A fifth feature was intergranular fracture, also shown in Fig. 3(b). Fig. 3(a) shows the ductile rupture of Ni-20Cr in air. These features together indicate considerable embrittlement by hydrogen, consistent with a previous brief report.²⁷ As will be shown below, Ni-20Cr was also embrittled at 194 K. The question of interest, then, was whether the dispersion-strengthened

alloys, Ni-20Cr-2ThO₂ and MA 753, would also be embrittled.

The microstructurally-appropriate comparison to Ni-20Cr is the recrystallized Ni-20Cr-2ThO₂; ductility results are compared in Fig. 4. No loss in ductility of charged specimens of Ni-20Cr-2ThO₂ occurred at 194 K or 295 K, within the precision of the results. The high-pressure hydrogen result also appears to be within the scatter of the triplicate tests on uncharged specimens. The Ni-20Cr, by contrast, showed large losses in RA, together with the indications in Fig. 2. Fig. 5 shows results on the as-received and the annealed mi-

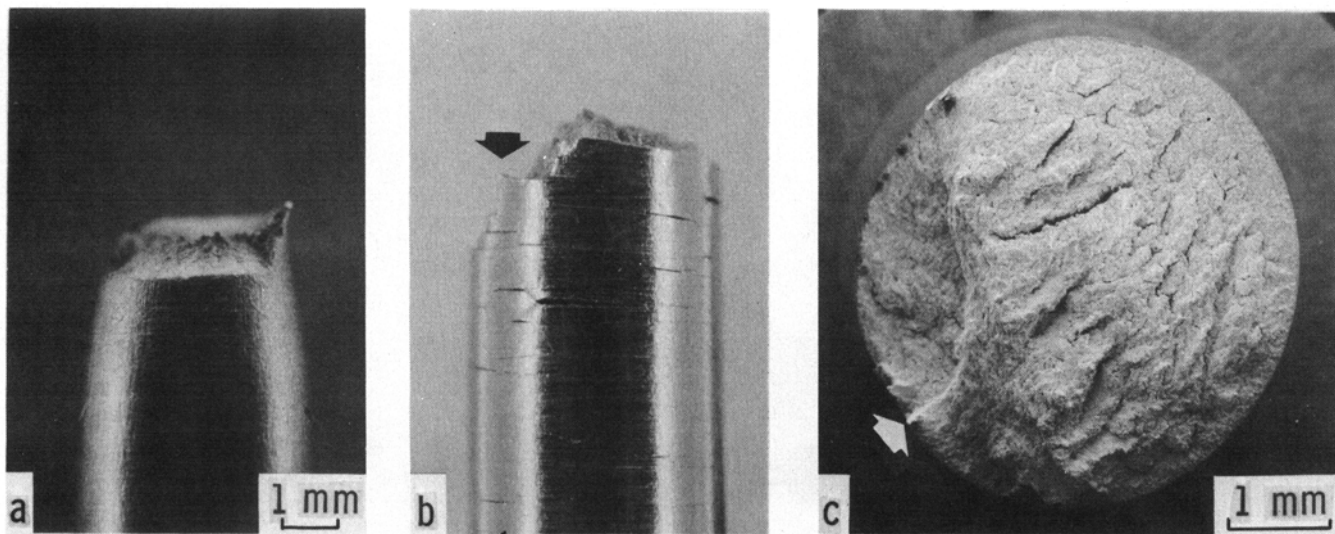
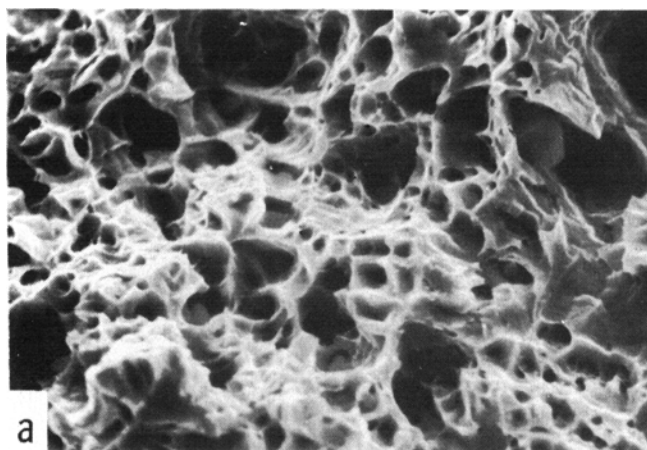
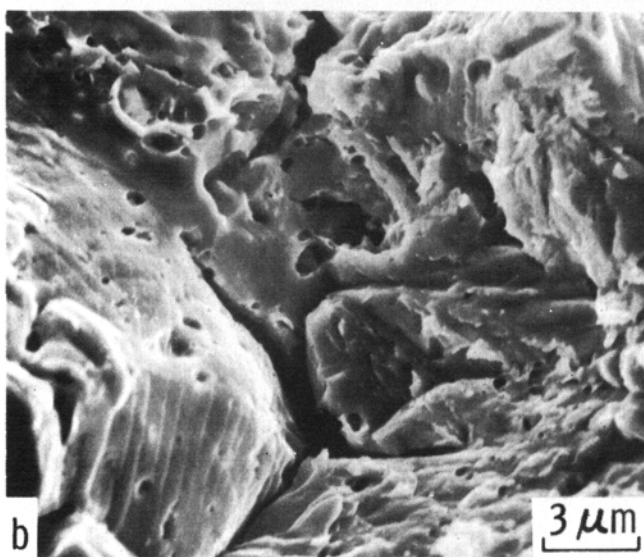


Fig. 2—Effect of hydrogen on fracture of recrystallized Ni-20Cr. (a) Light micrograph of specimen tested in air. (b) Light micrograph, same magnification, of specimen tested in 69 MPa hydrogen. (c) Scanning electron micrograph of specimen shown in (b); arrows indicate the same point on the fracture surface circumference in each photo.



a



b

Fig. 3—Fracture surface topography of recrystallized Ni-20Cr; scanning electron micrographs at same magnification. (a) Specimen tested in air. (b) Specimen tested in 69 MPa hydrogen.

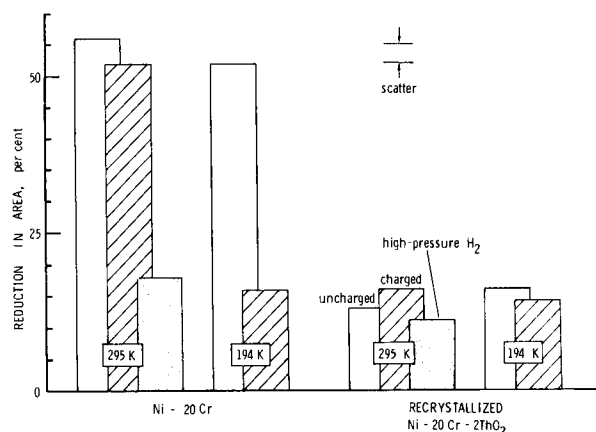


Fig. 4—Ductility of Ni-20Cr and Ni-20Cr-2ThO₂, both recrystallized, tested at 194 K and 295 K. Results are shown for uncharged and hydrogen-charged specimens, and for specimens tested in 69 MPa hydrogen.

microstructures of Ni-20Cr-2ThO₂; the former could not be hydrogen-charged without annealing it. Both these materials showed large ductility losses in high-pressure hydrogen, but fracture surface appearance was in all cases quite similar to the recrystallized Ni-20Cr-2ThO₂. Fig. 6 shows the fracture topography of the annealed material. The large particles, Fig. 6(b), were determined by an energy-dispersive X-ray technique to be chromium oxide, while the very small ones were found to be thoria. Fig. 6 is typical of what was observed after tests of all the Ni-20Cr-2ThO₂ microstructures; fracture surface appearance, which was unaltered by hydrogen, is similar to that reported earlier.^{12,28}

The MA 753 also showed behavior indicating compatibility with hydrogen. Fig. 7 shows the ductility results obtained in duplicate tests. Comparison to the Ni-20Cr results in Fig. 4 is instructive, but no data from an entirely analogous "undispersed" alloy (which would be Nimonic 80A) were obtained. Results on a variety of nickel-base superalloys, however, indicate^{5,18,29,30} that

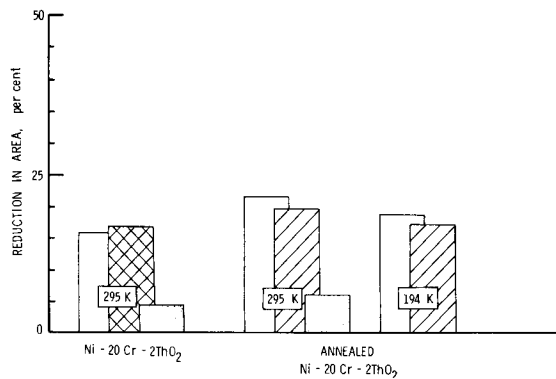


Fig. 5—Ductility of Ni-20Cr-2ThO₂ in the as-received and the annealed conditions. Cross-hatched result is for a test in 69 MPa helium; other results keyed as in Fig. 4.

compatibility of Nimonic 80A would be expected to be poor. If so, that would make the behavior of MA 753 consistent with that of Ni-2ThO₂⁸ and Ni-20Cr-2ThO₂ (Figs. 4 through 6): the presence of the dispersion confers compatibility with hydrogen. Fracture surfaces of MA 753 were blocky on a microscale, with very fine dimples, as shown in Fig. 8(a). This topography was not altered by hydrogen, as Fig. 8(b) shows.

The behavior of two grain sizes (obtained by different processing) of Ni-2ThO₂ was studied in earlier work;⁸ both were found to be compatible with thermally-charged hydrogen. Repetition of some of those tests to increase confidence in the results, and duplicate testing in 69 MPa hydrogen, are combined in Fig. 9 with earlier data to summarize the behavior of Ni-2ThO₂. Comparison data on pure Ni⁸ are also included. Even the coarse-grained material at 194 K, which showed some ductility loss, showed no change in fracture mode.⁸

In the earlier work,⁸ it was suggested that the presence of the dispersoid conferred the compatibility. It was suggested, however, that the different grain shape, or grain aspect ratio,²⁵ might tend to prevent the intergranular failure seen in Ni,⁸ because an intergranular fracture path would be so long around the "ends" of the elongated grains.³¹ That concept was tested with Ni-2ThO₂ specimens cut at 45 deg to the rolling direction, so that fracture in the grain-face boundaries would not require an unduly long fracture path. After hydrogen charging, these specimens were tested at 194 K. Ductility results, expressed as the pct loss [= 100(1 - R), where R = (charged RA)/(uncharged RA)] to account for ductility differences from the longitudinal direction, were very consistent with data in Fig. 9. For coarse-grained material at 194 K in each case, Fig. 9 shows a longitudinal loss of 25 pct; the 45 deg specimens exhibited 29 pct loss. In pure Ni, at 194 K by comparison, the loss was 77 pct.⁸ Thus the enhanced compatibility of Ni-2ThO₂ compared to pure Ni is not due to a grain orientation effect. That conclusion is supported by results (Fig. 4) for Ni-20Cr-2ThO₂, in which grains were essentially equiaxed, but intergranular fracture did not occur.

DISCUSSION

The prediction of Thompson and Wilcox,⁸ that materials much higher in strength than Ni-2ThO₂ could be compatible with hydrogen when containing a dispersoid,

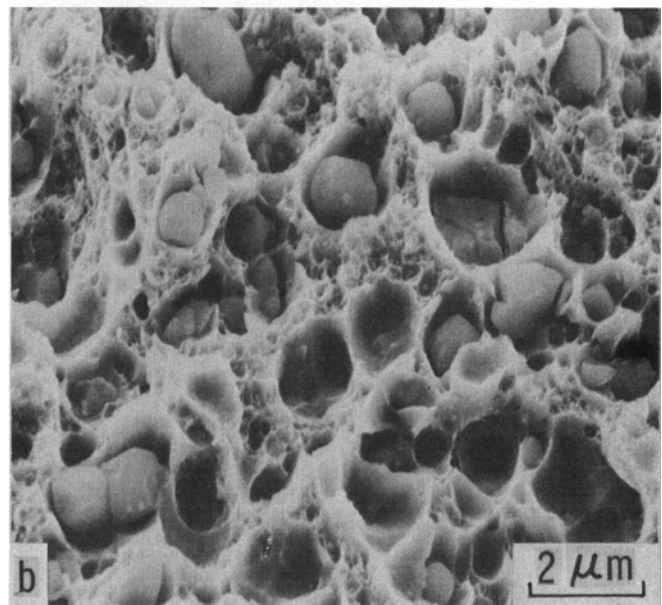
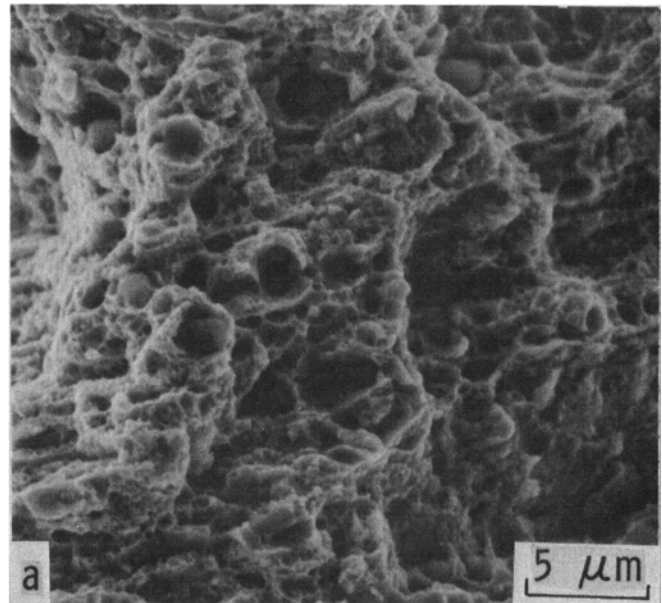


Fig. 6—Fracture surface topography of annealed Ni-20Cr-2ThO₂. (a) Typical fracture surface, independent of presence of hydrogen (in this case tested at 194 K after hydrogen charging). (b) Detail of fracture surface in an area with high density of chromium oxide particles.

appears to be correct. A static or limited hydrogen supply does not embrittle the dispersion-strengthened materials, possibly because the oxide particles provide voids during deformation into which hydrogen is swept by dislocations.^{4-6,8,12,26,32} The process is expected to be similar in Ni-2ThO₂ and Ni-20Cr-2ThO₂, although the alloys differ in two offsetting ways. First, the higher particle-matrix interface strength³³ in Ni-20Cr-2ThO₂ means that opening of voids is postponed until a higher strain, which should reduce the efficiency of the hydrogen sink behavior. But on the other hand, the stacking fault energy (SFE) of Ni-20Cr is considerably below that for Ni.³⁴⁻³⁶ This reduces ease of cross-slip and implies a tendency for dislocations to pile up at particles rather than cross-slip around them. Pile-ups,



Fig. 7—Ductility of aged and annealed MA 753 at 194 K and 295 K. Key to tests as in Fig. 4.

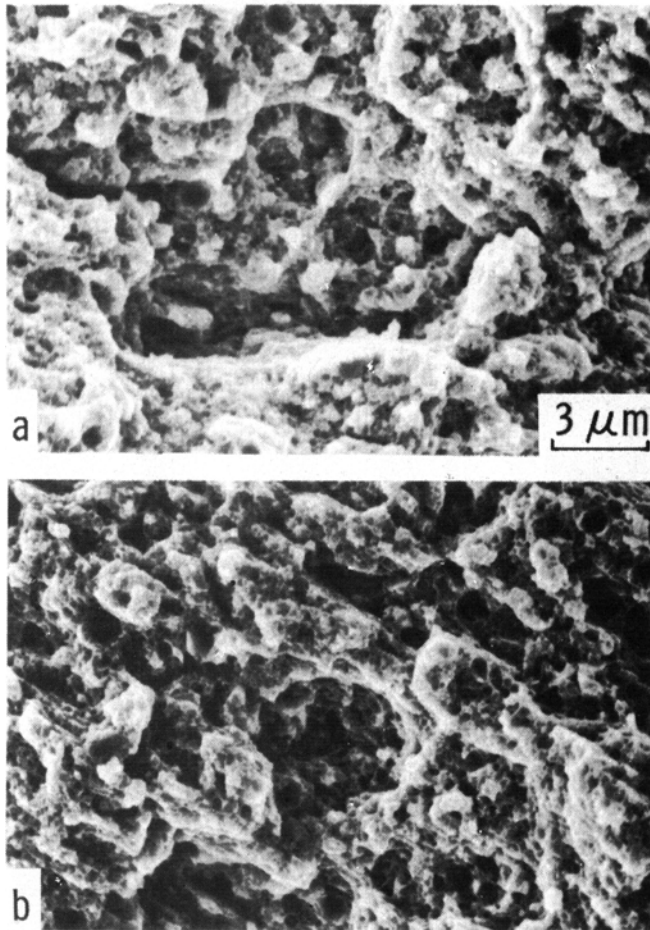


Fig. 8—Fracture surface topography of MA 753; scanning electron micrographs at same magnification. (a) Uncharged specimen tested at 194 K. (b) Specimen hydrogen-charged, tested at 194 K.

in turn, mean easier access to the particle from hydrogen atmospheres around dislocations in the pile-ups. Once hydrogen reaches the particle, it can be expected to be trapped there.³⁷ In the same way as has been described⁸ for Ni-2 ThO₂, this trapping greatly reduces access of hydrogen to the grain boundaries, thereby preventing intergranular failure (cf. Fig. 3) and ductility loss.

The hydrogen build-up at dispersoid particles may itself reduce ductility somewhat by accelerating ductile rupture, as happens in stainless steels.^{4,26,32} But because the build-up essentially prevents the large ductility loss (Fig. 4) associated with a change from ductile rupture to intergranular fracture, changes in the ductile rupture process are unimportant by comparison. It is possible, however, that the ductility losses in high-

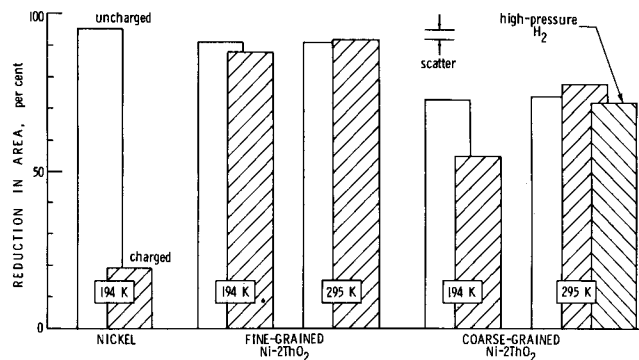


Fig. 9—Ductility of Ni and Ni-2 ThO₂ for tests at 194 K and 295 K; some data from Ref. 8.

pressure hydrogen shown in Fig. 5, which were ductile in all cases, were due to ductile rupture acceleration. The important point is that sink efficiency, as influenced by interface strength and SFE, is essential to hydrogen compatibility.

The relative importance of these two effects, particle interface strength and SFE, is difficult to determine. A comparison of Fig. 4 (fine-grained, recrystallized Ni-20 Cr-2 ThO₂) and the fine-grained results⁸ in Fig. 9 (annealed Ni-2 ThO₂) suggests the two may be about balanced, because the two sets of results at 194 K and 295 K on hydrogen-charged specimens are similarly related to the respective uncharged results. This comparison, however, may be overshadowed by the beneficial effects of the fine grain size.⁸ A more interesting comparison can be made with the coarse-grained data of Fig. 7. These data suggest a predominant role for reduced SFE when compared to the coarse-grained data in Fig. 9, since precipitation of the Ni₃(Ti, Al)γ' phase removes Ni from the matrix and further reduces SFE.³⁴ A similar conclusion about the importance of SFE and ease of cross-slip in affecting hydrogen behavior has been reached in studies on stainless steel.^{4,26,32}

It should be emphasized, as stated above, that these results apply to a relatively limited hydrogen supply, even though supersaturations were as high as a factor of 45 in Ni-2 ThO₂. Frandsen, Paton and Marcus³⁸ showed that fatigue crack growth in TD-Ni and TD-NiCr was somewhat more rapid in hydrogen than in vacuum; the increase in rate was similar to that seen in Ni. An unlimited supply of hydrogen can evidently saturate the sinks at oxide particles, and then cause fracture changes in the same way as in the pure material. Thus the dispersion may effectively prevent hydrogen damage only when the deformation rate exceeds the rate of hydrogen supply to the deforming region or crack tip. This conclusion, however, may contain a complication. Frandsen, *et al.*³⁸ used tests in vacuum rather than in air as their standard of comparison, and they found that oxygen caused the same cracking rate acceleration as did hydrogen. Other studies on nickel-base alloys have also shown oxygen to be damaging, either about the same as³⁹ or less than⁴⁰ hydrogen. Thus it is possible that the fatigue properties of dispersion-strengthened alloys may not be much worse in hydrogen than in air.

For structural applications, the possibility of rate-limited compatibility would suggest the following. Fatigue life-limited applications in hydrogen environ-

ments would require further careful study of environmental effects on failure.³⁸⁻⁴⁰ But for cases where infrequent high stresses or accidental overloads determine the design limits in hydrogen, dispersion-strengthened materials would be an appropriate choice. The case of sustained loads, as in a pressurized structure, has not been studied, but lack of fracture mode changes in hydrogen (Figs. 6 and 8) suggest no great susceptibility. It does seem clear that dispersion-strengthened alloys will be suitable for some applications in hydrogen, and work on forming, joining,^{11,12} and corrosion resistance now seems appropriate.

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