Hydrogen Assisted Cracking of Type 304 Stainless Steel

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This paper reports a study of hydrogen assisted cracking in type 304 stainless steel. It shows that the most detrimental effect in increasing the susceptibility of the material to hydrogen cracking is the formation of martensite upon deformation. This is particularly damaging if the martensite is localized at the grain boundaries. With martensite present intergranular impurities such as phosphorus play a secondary role. As martensite becomes more difficult to form, the importance of impurities increases.

T YPE 304 stainless steel is one of the most commonly used austenitic steels. Its uses would be even wider were it not for the fact that it becomes quite susceptible to intergranular corrosion after heat treatment in the temperature range of 600 to 800°C, for times even as short as 15 min. This phenomenon, often referred to as sensitization, is usually attributed to the formation of chromium carbides at the grain boundaries and the resulting depletion of chromium around the precipitates.[1] Since hydrogen is a by-product of most corrosion reactions, it is important to know if sensitization to corrosion also makes the material more susceptible to hydrogen cracking. More generally, it is important to know if 304 stainless steel in either the sensitized or unsensitized condition is susceptible to hydrogen cracking, as this could limit its use in aqueous or other hydrogen bearing environments.

Many investigators have studied hydrogen assisted cracking in austenitic stainless steels.[2-21] Early work suggested that the formation of deformation induced martensite was essential for the embrittlement to be observed.[2,8] This view arose because steels which partially transform to martensite upon straining, such as 304L, were found to be susceptible to hydrogen cracking whereas those with stable austenite phases, such as 310, were difficult to embrittle. Also the fracture mode in 304L often appeared to be quasi-cleavage along martensite laths. However, hydrogen assisted cracking has been reported in 310 stainless steel,[5,6] and Thompson could not correlate hydrogen embrittlement with martensite formation in 309 stainless.[14] Recently, it has been suggested that low stacking fault energy and coplanar dislocation motion greatly enhance hydrogen cracking in stainless steels.[10,17,20,21] Odegard, Brooks, and West[10] found that the susceptibility to hydrogen cracking in steels which did not transform to martensite upon deformation correlated very well with these parameters, and that the fracture mode in hydrogen changed from ductile rupture to intergranular fracture as the embrittlement became more severe. They also found that martensite formation increased the susceptibility to hydrogen fracture over that expected from stacking fault energy measurements. All of these results indicate that if martensite is present it increases the suscepti-

bility to hydrogen assisted cracking, but that embrittlement can occur without it.

There have been several studies of the effect of sensitization on the hydrogen cracking susceptibility of austenitic stainless steels. Thompson[14] examined sensitization in 309 stainless and found that it did not change the mechanical properties of this material in hydrogen, although highly sensitized material did show a decreased dimple size. Fidelle and coworkers[12,13] found that sensitized 304 stainless steel failed intergranularly when the grain boundary carbides were continuous. I also reported that sensitized 304 stainless steel failed intergranularly in hydrogen even though the carbides were not continuous along the grain boundaries.[15] The main reason for this was that chromium and carbon depletion at the boundaries allowed this region to transform to martensite upon deformation, thus providing an easy fracture path along the grain boundaries.[16]

In this paper I report a further study of hydrogen cracking in type 304 stainless steel. In particular, this work examines the effect of intergranular impurities. These elements greatly reduce the resistance of low alloy, ferritic steels to hydrogen cracking,[22-24] and it is important to know if they play a similar role here. As I will show, the overriding effect is that of martensite formation. When it forms in a continuous path along the grain boundaries, as in a sensitized stainless steel, the presence or absence of grain boundary impurities is of little matter. Only when martensite is difficult to form do impurities become important.

EXPERIMENTAL

All alloys used in this study were melted and fabricated by the General Electric Company. The final product was 3.25 mm rod. The chemical compositions of these materials are given in Table I. Two heats are basic 304 steels. One heat has a very low impurity content and the other heat is doped with 0.06 wt pct phosphorus. Phosphorus was added to this heat to study the effects of intergranular impurities. It was chosen because it has been found on the grain boundaries of commercial heats of 304 stainless steel.[25] The other two heats have lower carbon contents. Again one is a high purity alloy and the other is doped with 0.06 wt pct P.

All of the samples used in these experiments were first solution annealed for one hour at 1100°C (1373 K) and then water quenched. Some samples were given a

Table I. Chemical Compositions of Alloys Studied

Heat Description	Ni	Cr	P	C	S	N
High Carbon, High Purity	9.4	18.6	0.003	0.069	0.009	0.002
High Carbon + 0.06 Wt Pct P	9.4	17.6	0.060	0.063	0.007	0.002
Low Carbon, High Purity	9.2	18.6	0.004	0.022	0.006	0.01
Low Carbon + 0.06 Wt Pct P	9.2	18.3	0.060	0.024	0.006	0.01

further heat treatment of 650°C for 24 h. This latter heat treatment should allow for additional phosphorus segregation. It also caused the precipitation of chromium carbides along the grain boundaries. In the high carbon heats this precipitation occurred along almost every boundary and made the samples quite susceptible to intergranular corrosion. In the low carbon heats, precipitation occurred on less than 5 pct of the boundaries, and the material was still relatively immune to intergranular corrosion. Throughout this study I have compared solution annealed samples (1100°C for 1 h only) with samples given the additional sensitizing treatment of 650°C for 24 h.

Two types of tests were employed to study hydrogen cracking. One was a time to failure test. In these tests smooth bar tensile samples (gage diam of 1.52 mm) were mounted in a charging cell and an initial stress was applied. The samples were then cathodically charged with hydrogen throughout the test and the time to failure was measured. The charging solution was 0.1 N H_2SO_4, containing 500 mg/l of sodium arsenite. The current density was 40 mA/cm^2. Initially tests were run for one week, and if a sample had not failed within that time the test was terminated. However, after many such tests it became clear that samples usually failed within the first 12 h of the test and always within the first 48 h of the test, or not at all. Figure 1 shows the experimental evidence for this. The data shown in this figure are for the commercial heat of 304 stainless steel studied in Ref. 15. The heat treatment for these samples was 1100°C for 1 h plus 650°C for 24 h. As can be seen on this figure, the time to failure suddenly shifts from less than 10 h to longer than 48 h as the initial applied stress is decreased. Based on this the termination time for this test was shortened to 48 h. Therefore, the measure of susceptibility to hydrogen cracking, as determined by this test, is the minimum stress required to cause failure within 48 h.

The second test was a constant displacement rate test in hydrogen gas. Notched tensile bars were
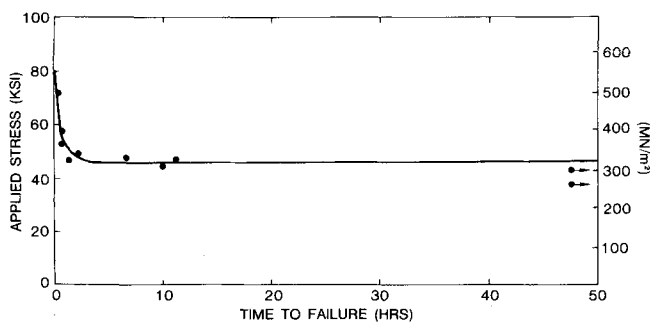

Fig. 1—A typical time to failure curve for a commercial heat of 304 stainless steel sensitized at 650°C for 24 h. The time to failure is plotted as a function of the initial applied stress.

mounted in a stainless steel chamber.[15] The chamber was evacuated to a pressure of 0.13 Pa. It was then backfilled with dried hydrogen gas to a pressure of 0.17 MPa (1.7 atm). The samples were slowly pulled to failure at a constant crosshead speed of 0.05 mm/min. After this test, the fracture modes of the samples were carefully examined.

The results of these tests gave the following information. First, the cathodic charging tests provided a ranking of the susceptibility of the different alloys to hydrogen cracking. Secondly, by comparing the fracture modes of samples which failed during cathodic charging and those pulled to failure in the gaseous hydrogen experiments, I was essentially comparing the effect of two different hydrogen pressures. In all of these tests duplicate samples were run.

RESULTS

The results of the time to failure tests are given in Table II. In these tests I have measured the lowest stress which will cause failure within 48 h. Several things should be noted. The most susceptible materials were the high carbon model 304 heats given the sensitizing heat treatment of 650°C for 24 h. The presence or absence of phosphorus made no difference. The second most susceptible materials were the lower carbon alloys. Again the presence or absence of phosphorus was unimportant, and there was no observable difference between the solution annealed samples and those given the additional heat treatment of 650°C for 24 h. By far the most resistant materials to hydrogen cracking were the high carbon alloys which had only received the high temperature solution anneal. No failures were observed for the pure heat in this condition at stresses as high as 414 MN/m^2. However, a sample of the phosphorus doped heat did fail in 47 h at an applied stress of 393 MN/m^2.

Typical fracture surfaces from these samples are shown in Figs. 2 to 4. Figure 2 shows fractures of the sensitized, high carbon materials. The initial hydrogen fracture was always predominantly intergranular and the rapid fracture at the center of the sample was microvoid coalescence. In addition, a striated transgranular fracture was observed for both heats, especially near the onset of fast fracture, Fig. 2(e).

Table II. Minimum Stress Required to Cause Failure During 48 H of Cathodic Charging

| Heat	Heat Treatment	MN/m^2	KSI
High Carbon, High Purity	1100°C for 1 h	>414	>60
High Carbon + 0.06P	1100°C for 1 h	379-414	55-60
Low Carbon, High Purity	1100°C for 1 h	275-310	40-45
Low Carbon + 0.06P	1100°C for 1 h	275-310	40-45
Low Carbon, High Purity	1100°C for 1 h; 650°C for 24 h	275-310	40-45
Low Carbon + 0.06P	1100°C for 1 h; 650°C for 24 h	275-310	40-45
High Carbon, High Purity	1100°C for 1 h; 650°C for 24 h	206-241	30-35
High Carbon + 0.06P	1100°C for 1 h; 650°C for 24 h	206-241	30-35
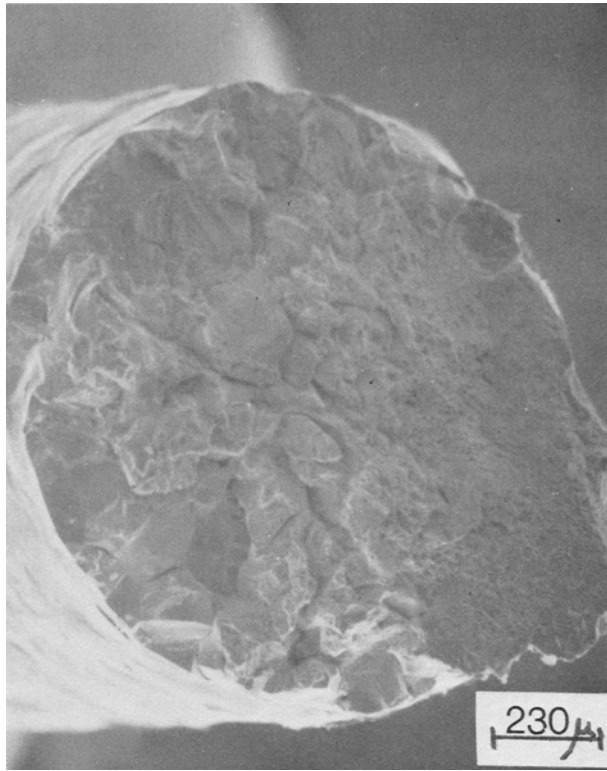
(a)
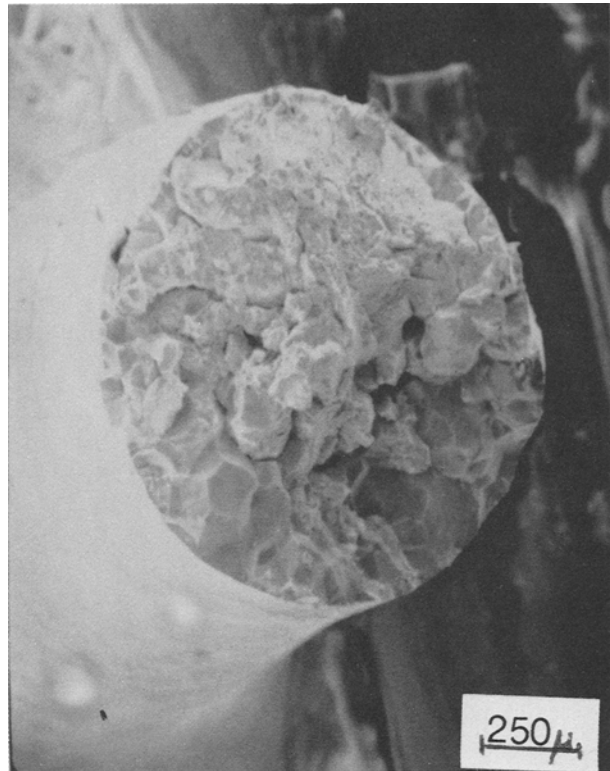
(c)

(b)
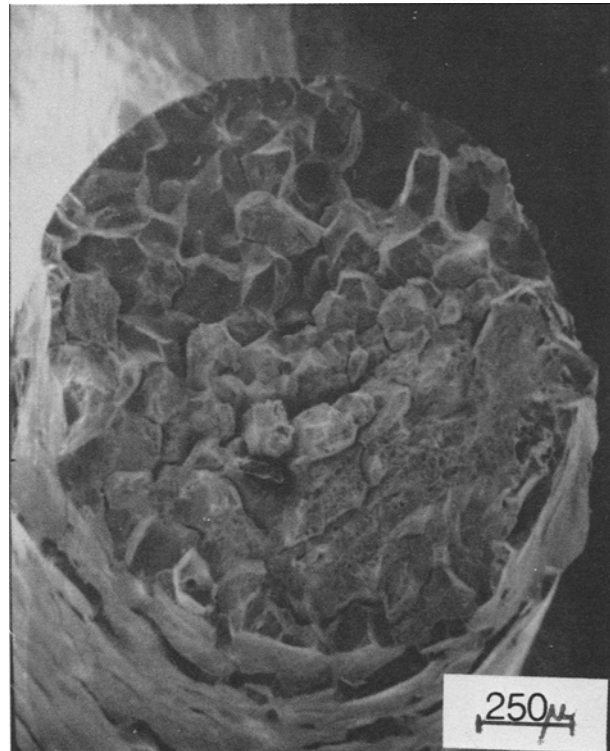
(d)

Fig. 2—The fracture surfaces of the high carbon 304 samples, sensitized at 650°C for 24 h, after cathodic charging failures. (a) The high purity heat with an initial applied stress of 379 MN/m², (b) The phosphorus doped heat held at an initial applied stress of 379 MN/m², (c) The high purity heat held at an initial stress of 250 MN/m², (d) The phosphorus doped heat held at an initial stress of 250 MN/m², (e) An example of the striated transgranular fracture observed in these samples.
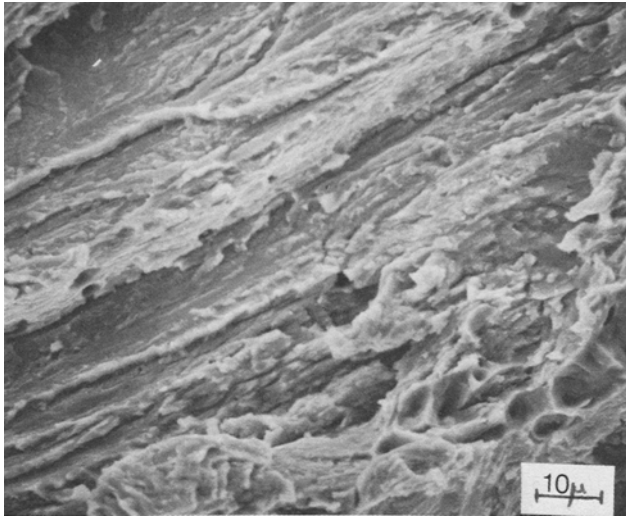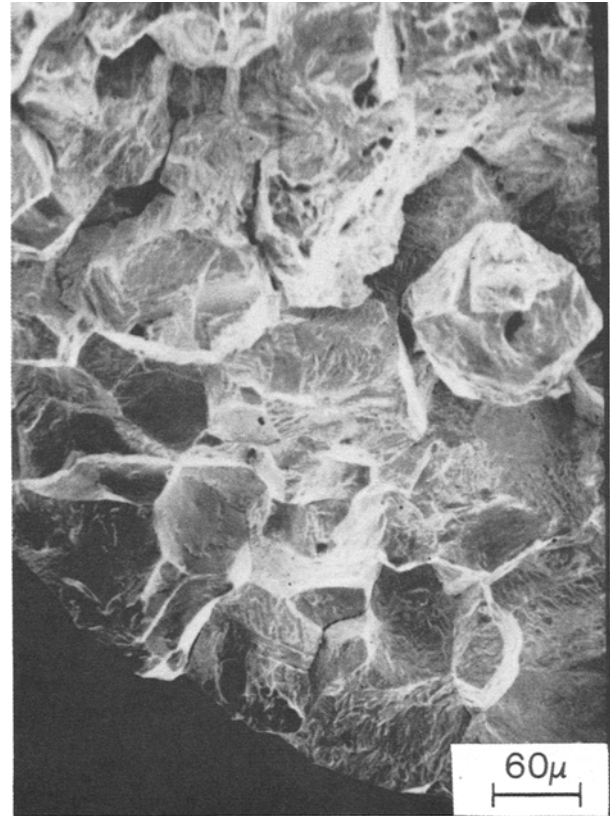
Fig. 2—Continued. *(e)*

The extent of intergranular fracture depended on two factors. The primary one was the applied stress; the lower the applied stress and the longer the time to failure, the more intergranular fracture observed. Also, at any given stress which caused failure, more intergranular fracture was observed in the phosphorus doped steel, Fig. 2(a) to (d).

Figure 3 shows typical fracture surfaces of the low carbon alloys. The surfaces of the solution annealed samples and samples given the additional heat treatment of 650°C for 24 h were quite similar. Over 60 pct of the grain boundaries in the high purity heat had ductile tearing on them whereas those in the phosphorus doped samples were nearly always smooth. Figure 4 shows the fracture surface of a solution annealed sample of the high carbon heat doped with phosphorus. Again it is clear that the initial hydrogen fracture is predominantly intergranular.
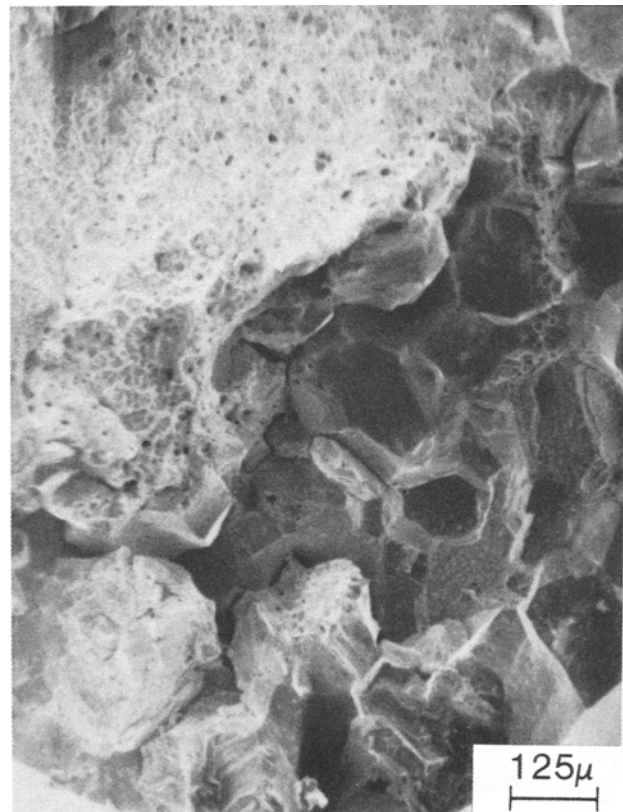
In the slow extension rate tests notched tensile bars were pulled to failure in dry hydrogen gas, and the fracture surfaces of the samples were examined. A summary of these results is given in Table III and typical fractographs are shown in Figs. 5 to 7. In Table III I have listed three types of fracture mode:

Table III. Fracture Mode in Hydrogen Gas

Heat	Heat Treatment	Fracture Mode (Pct of Surface)		
		Inter-granular	Striated Transgranular	Microvoid Coalescence
High Carbon, High Purity	1100°C for 1 h	—	20	80
High Carbon + 0.06P	1100°C for 1 h	20	40	40
Low Carbon, High Purity	1100°C for 1 h	30	20	50
Low Carbon + 0.06P	1100°C for 1 h;	60	20	20
Low Carbon + 0.06P	1100°C for 1 h; 650°C for 24 h	60	20	20
High Carbon, High Purity	1100°C for 1 h; 650°C for 24 h	90	10	—
High Carbon + 0.06P	1100°C for 1 h; 650°C for 24 h	95	5	—

(a)

(b)

Fig. 3—The fracture surfaces of the low carbon alloys after cathodic charging failures. (a) is the high purity heat, (b) is the phosphorus doped heat.
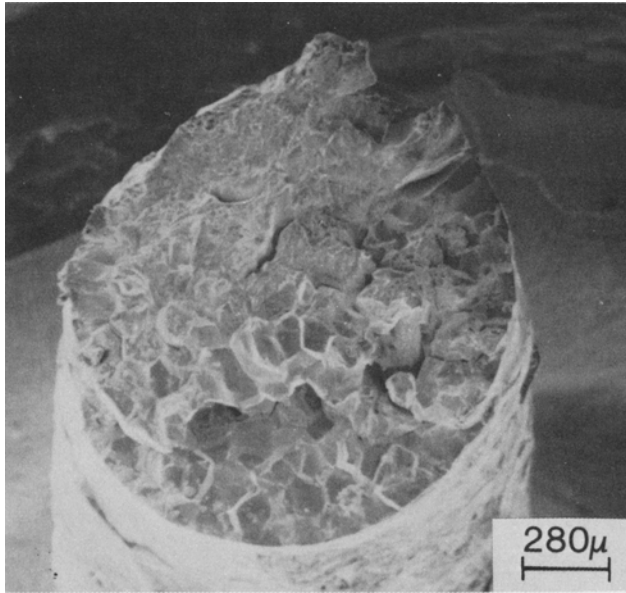
Fig. 4—The fracture surface of the unsensitized, phosphorus doped high carbon sample that failed during cathodic charging.

intergranular fracture, striated transgranular fracture,[18] and microvoid coalescence. The term striated transgranular refers to the type of transgranular fracture shown in Fig. 2(e). Intergranular fracture and striated transgranular fracture are completely due to hydrogen assisted cracking. Microvoid coalescence is the rapid fracture mode in both air and hydrogen.

The results of these tests rank in the same general order as do those from the cathodic charging experiments. In the high carbon heats sensitized at 650°C for 24 h intergranular hydrogen assisted fracture was produced across the entire surface, Fig. 5. In the low carbon alloys some microvoid coalescence was observed in the center of the fracture surface, Fig. 6. In the solution annealed, high carbon materials microvoid coalescence was the primary fracture mode. Also the effect of phosphorus was much more obvious in these experiments, especially as the susceptibility of the material to hydrogen cracking decreased. In sensitized samples of the high purity, high carbon alloy the intergranular surfaces were often ductile; in the phosphorus doped heat they were quite smooth, Fig. 5. More intergranular fracture was observed in the phosphorus doped heats of the low carbon alloys and in solution annealed samples of the high carbon steels than in the corresponding high purity materials, Figs. 6 and 7.

In summary, both experiments show that the high carbon heats of 304 stainless steel sensitized at 650°C for 24 h are the most susceptible to hydrogen assisted cracking. If these alloys are not sensitized, they are very resistant to hydrogen cracking. The low carbon steels have an intermediate susceptibility.

DISCUSSION

Intergranular hydrogen assisted fracture has been reported many times in many different materials.[26] This should obviously be the predominant fracture mode if the boundaries are inherently weak. A very

good example of this is seen in low alloy, ferritic steels. When impurities segregate to the grain boundaries of these steels and weaken them, the material's susceptibility to hydrogen cracking is greatly enhanced and the hydrogen fracture becomes increasingly intergranular.[22,23] Clearly, a similar impurity mechanism does not explain the results reported here. If it did, a great difference would be observed in the susceptibility of the high purity heats and the phosphorus doped heats. This is not the case. The major difference is between the solution annealed and sensitized high carbon materials. The susceptibility of the low carbon alloys falls between these two extremes and is not changed by the additional heat treatment of 650°C for 24 h.

It has been reported that martensitic and ferritic materials are more susceptible to hydrogen cracking than are austenitic materials.[26] Also, earlier work has shown that austenitic materials which transform to martensite upon deformation are considerably more susceptible to hydrogen cracking than those that do not.[2,3,6,8,10,17,21] This transformation to martensite is not only dependent on the amount of deformation given to the material, but is also determined by the alloy content.[27] Almost all elements tend to retard this transformation, but chromium, carbon, and nitrogen are particularly strong inhibitors. Therefore, the low carbon alloys would be more susceptible to martensite formation than the solution annealed high carbon materials. The most susceptible of all would be the grain boundary regions of the sensitized high carbon materials. During sensitization chromium carbides $(Cr_{23}C_6)$ form along the boundaries and deplete the adjacent regions of both chromium and carbon.[14,28] Martensite can then easily form along the grain boundaries of the sensitized high carbon materials. It will only form at points of highest deformation in the unsensitized samples. Martensite should form easily throughout the low carbon materials, but would not be particularly localized at the grain boundaries. Because of the lower carbon level sensitization at 650°C for 24 h causes very little precipitation of chromium carbides, and thus little Cr depletion. Therefore, it does not change the pattern of martensite formation.

One way to check these ideas qualitatively is by the ferrofluid technique.[29] Ferrofluid is a colloidal suspension of iron filings. In this experiment a specimen is mounted and polished but not etched. A drop of fluid is placed on the sample and spread out with a cover glass. When a magnetic field is applied, the iron filings are attracted to the ferromagnetic regions of the material. Here, this would be the martensite, not the paramagnetic carbides or austenite.[29]

Figure 8 shows the results of these experiments. In the sensitized high carbon materials the continuous intergranular martensite is clearly delineated, Fig. 8(a). In the unsensitized, high carbon samples only regions of maximum deformation such as the slip lines are transformed to martensite, Fig. 8(b). Also, in these unsensitized alloys the amount of martensite formation varies greatly from grain to grain. In the low carbon alloys, Fig. 8(c), martensite formation occurs in every grain. The grain boundaries, then, are not completely martensitic, but most all are partially martensitic.
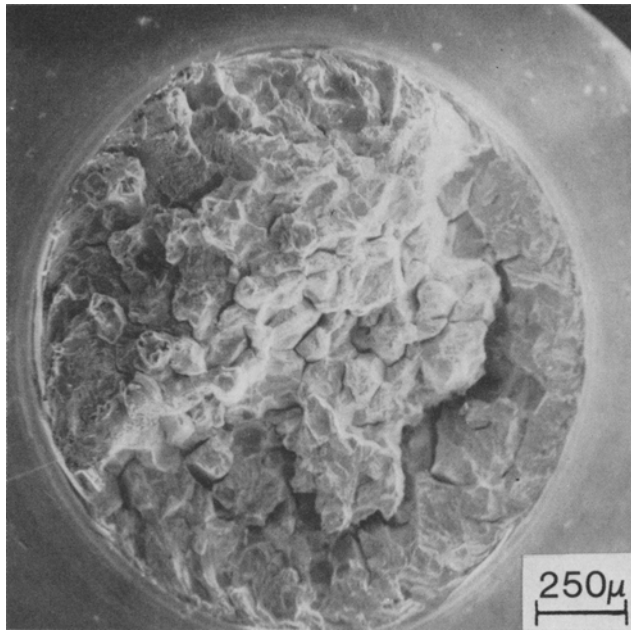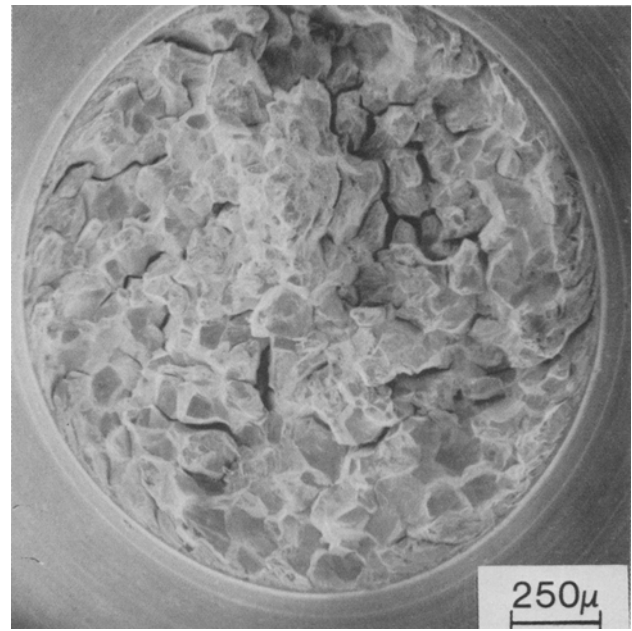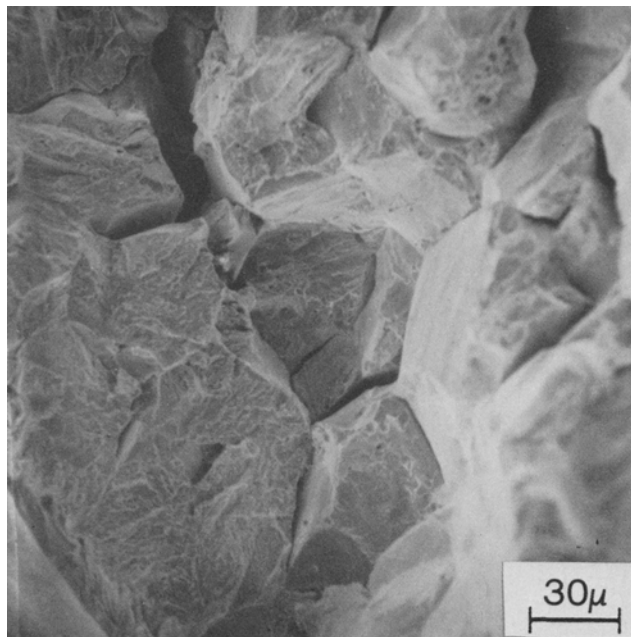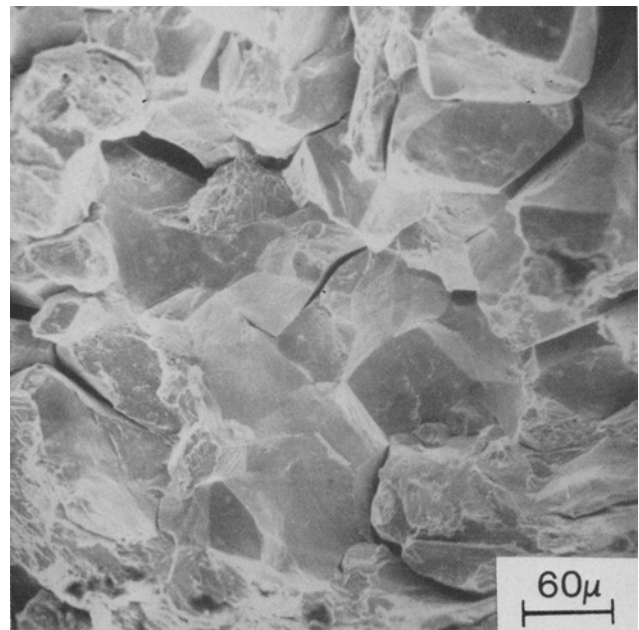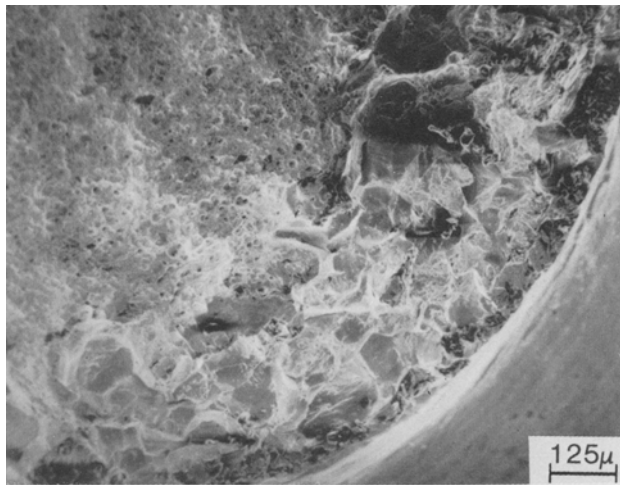
Fig. 5—Fracture surfaces of the sensitized high carbon samples pulled to failure in hydrogen gas. (*a*) and (*b*) are the high purity heat, samples (*c*) and (*d*) are from the phosphorus doped heat.

The fracture mode of these materials in hydrogen and the susceptibility to hydrogen cracking, as measured by the cathodic charging test, correlate well with these observations of martensite formation. When the martensite is continuous along the grain boundaries, as in the sensitized high carbon samples, the susceptibility is the greatest. As the martensite becomes less continuous at the boundaries, higher stresses are required to produce hydrogen failures. The fracture becomes less intergranular and often proceeds along the transgranular martensite bands leading to the striated fractures shown in Fig. 2(*e*), 6, and 7 and reported in Table III. The main effect of phosphorus is to increase the extent of intergranular fracture, Figs. 2

to 7. This is particularly obvious when martensite is not continuous along the grain boundaries. Only when martensite is very difficult to form, as in the solution annealed high carbon samples, does phosphorus measurably affect the susceptibility to hydrogen cracking.

These results, which show that the presence of martensite greatly lowers the resistance of austenitic stainless steels to hydrogen cracking, are clearly in agreement with prior work,[2,6,8,10,17,18,21] and the low carbon alloys used in this study have carbon concentrations equivalent to the commercial 304L heats used in previous studies.[3,6,8,10,17,18,21] However, I have observed more intergranular fracture than has been reported in the past. There are two probable causes
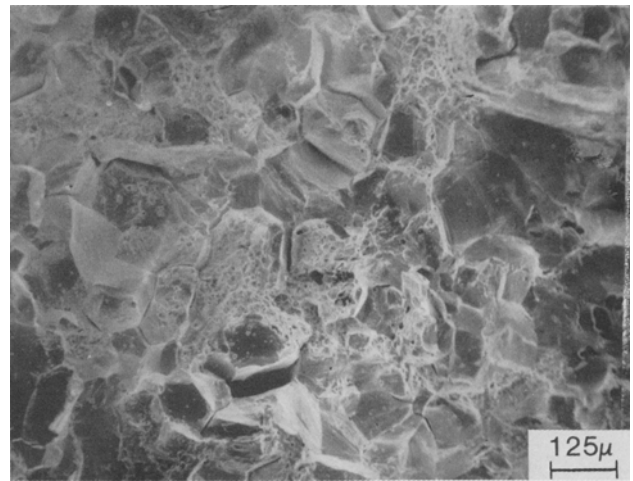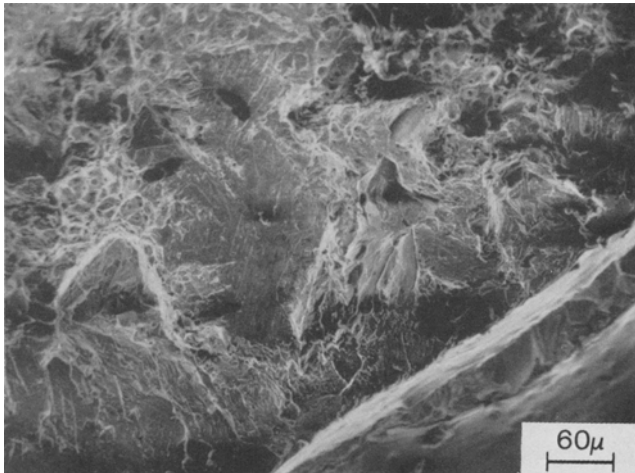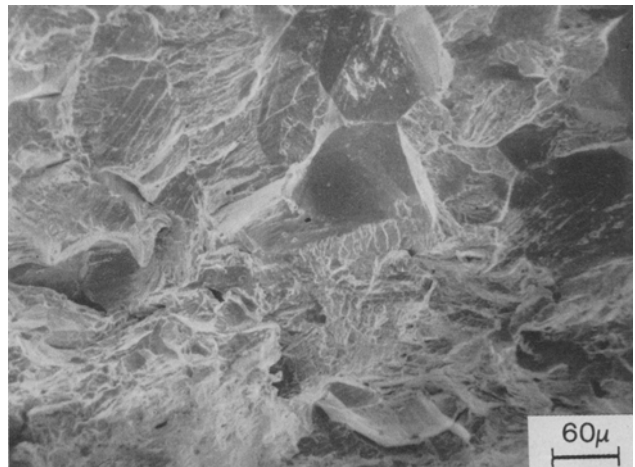
(a) · *(b)*

Fig. 6—Fracture surfaces of the low carbon alloy samples pulled to failure in hydrogen gas. (*a*) is the high purity heat, (*b*) is the phosphorus doped heat.

(a) · *(b)*

Fig. 7—Fracture surfaces of solution annealed high carbon samples pulled to failure in hydrogen gas. (*a*) is the high purity material, (*b*) is the phosphorus doped material.

for this. First, in many of the previous experiments the samples were charged before pulling to failure. Hydrogen charging produces martensite and can cause cracks along the martensite bands, even in the absence of stress.[3,19] Therefore, the observed fracture could be simply continuations of these transgranular cracks. Secondly, these commercial heats usually contain Si, Mn, N, and other additional elements which inhibit the transformation from austenite to martensite upon deformation. As was discussed above, the reason why intergranular fracture is observed in the lower carbon alloys is that many martensite bands intersect the boundary, giving a semicontinuous region of martensite. With these other elements present the regions of untransformed austenite would become even greater.

The reason why martensite is so much more susceptible to hydrogen cracking remains unclear. Changes in slip behavior, hydrogen permeability, hydrogen solubility, dislocation, and tensile properties within this phase have all been suggested as possibilities.

The results given here generally agree with other studies of the effect of sensitization on hydrogen cracking in austenitic stainless steels. Thompson[14] found that sensitization only affected the fracture mode in hydrogen when the carbides were essentially continuous along the grain boundaries. He observed a decreased dimple size and intergranular rupture along the grain boundary carbides. However, the sensitization heat treatment used in the study possibly did not provide enough Cr depletion to allow extensive martensite formation. Fidelle *et al*[11,12] observed intergranular fracture in sensitized 304 stainless when the carbides were continuous along the grain boundaries. Again, this is probably due to martensite formation.

Although the martensite problem has been investigated in the past, practically no research has been done on the effect of grain boundary impurities on hydrogen cracking. Nearly all previous studies have used commercial heats with varying levels of elements such as S, Si, P, and N. The work reported here shows that when martensite does not readily form, impurities may affect both the fracture mode and the cracking susceptibility of austenitic stainless steels in hydro-

gen. Even when martensite forms along the grain boundaries, impurities can increase the extent of intergranular hydrogen fracture. The only other study which provides similar results is that of Odegard,
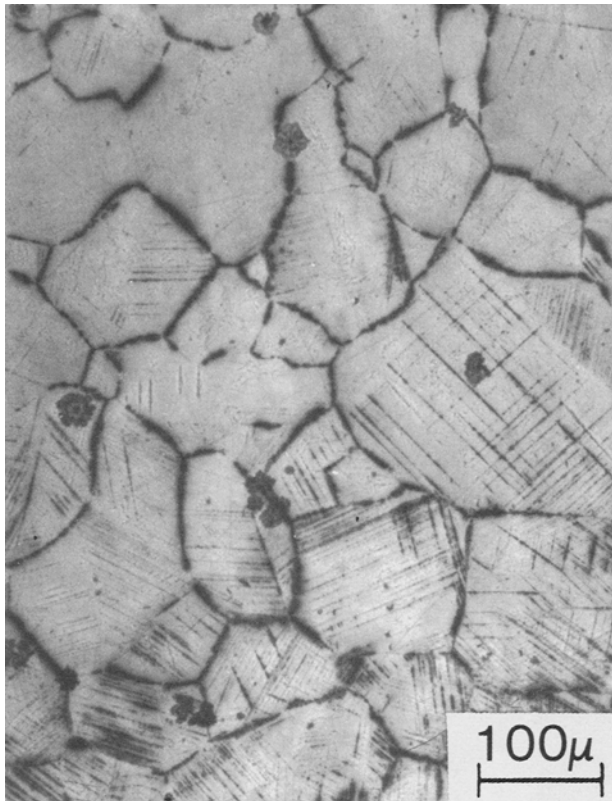
(a)

(b)

(c)

Fig. 8—Micrographs of samples covered with ferrofluid and placed in a magnetic field. (a) is a sensitized high carbon sample stressed to a level of 276 MN/m^2. (b) is an unsensitized high carbon sample stressed to a level of 393 MN/m^2. (c) is a low carbon alloy stressed to a level of 290 MN/m^2. Sample C was charged with hydrogen while the stress was applied. Note that a crack had formed along one of the highly martensitic grain boundaries.

Brooks, and West.[10] They found that increasing the nitrogen concentration in a 21-6-9 stainless increased the tendency toward intergranular fracture and decreased the resistance to hydrogen assisted cracking. Although N is a grain boundary embrittler,[24] this increase in N concentration also raised the yield strength of the alloy and changed the dislocation motion from cross slip to coplanar. Therefore, it is difficult to isolate the effect nitrogen had on the grain boundary strength in these steels. Phosphorus and nitrogen are also both relatively weak grain boundary embrittlers in ferritic steels;[24] it is possible that elements such as Sn and Sb will be even more detrimental to the hydrogen cracking susceptibility of stainless steel.

CONCLUSIONS

In this paper I have reported a study of hydrogen assisted cracking in type 304 stainless steel. The results show the following:

1) The material is most susceptible if a continuous path of martensite exists along the grain boundaries, as in the sensitized high carbon alloys.

2) If this martensite exists, hydrogen is a sufficient grain boundary embrittler. The presence or absence of other impurities cause only secondary effects.

3) If a continuous path of grain boundary martensite is not formed, but large quantities of matrix martensite are formed as in the low carbon alloys, the material is still rather susceptible to hydrogen cracking. However, the extent of intergranular fracture becomes more dependent on hydrogen pressure and the presence of additional grain boundary impurities.

4) As it becomes increasingly difficult to form deformation induced martensite, it also becomes more difficult to produce hydrogen cracks. In this case grain boundary impurities can lower the susceptibility to hydrogen cracking and make the hydrogen fracture path at least partially intergranular.

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