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Hydrogen embrittlement of maraging steel

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Specimens of 18 Ni 1800 MPa (M250) grade maraging steel were charged with different quantities of hydrogen by an electrochemical method. The tensile properties and fracture characteristics have been correlated with the quantity of hydrogen picked up by the material. A drastic decrease in ultimate tensile strength from 1768 MPa to 750 MPa, elongation from 6% to less than 2%, and reduction in area from 55% to less than 5%, were observed as the hydrogen content of the steel increased from less than 2 p.p.m. to 7 p.p.m. However, hydrogen does not affect the hardness of the steel. The effect of baking at different temperatures on hydrogen embrittlement was also studied. A change in fractographic features from ductile dimples to mixed mode, intergranular separation and transgranular cleavage was observed as the amount of absorbed hydrogen increased.

1. Introduction

The phenomenon, commonly termed hydrogen embrittlement, is of great concern for the use of advanced high-strength materials to a broad spectrum of high technology applications [1–4]. The effects of hydrogen on the mechanical properties of iron and steels have been widely investigated [5–27]. The deleterious effect of hydrogen on the mechanical properties of steels, especially high-strength martensitic steels, is no exception [1, 2, 7].

Owing to the unique mechanical and fracture properties of 18 Ni 1800 MPa grade maraging steel, it is a good candidate as a structural material for solid fuel rocket motors. It is likely that the steel can be contaminated with small quantities of hydrogen during processing into various end-products. Small quantities of hydrogen, of the order of parts per million levels, when present in this steel, can embrittle it.

Although extensive literature is available on gaseous hydrogen embrittlement of 18 Ni 1800 MPa maraging steel [8, 9], little work has been carried out on internal hydrogen embrittlement of this steel. The present work is carried out to complement the earlier studies and to expand the database for understanding the influence of internal hydrogen on tensile properties and fractographic features of 18 Ni 1800 MPa maraging steel.

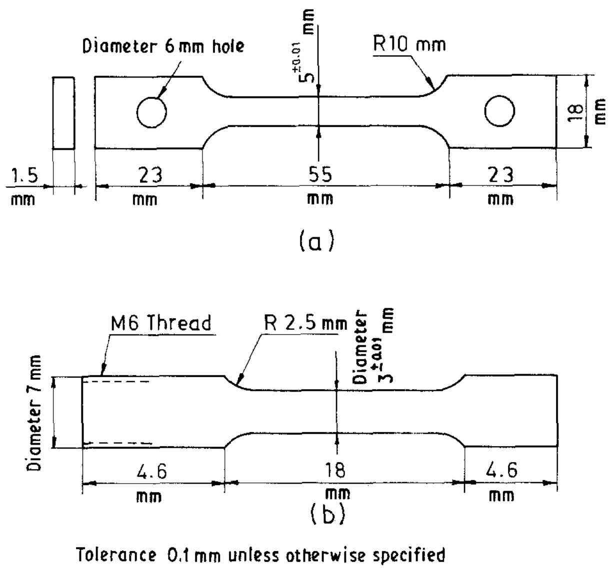

Figure 1 (a) Pin-loaded flat tensile specimen, (b) round-bar tensile specimen.

2. Experimental procedure

Flat and round-bar tensile specimens (Fig. 1) are made from 18 Ni 1800 maraging steel in the solution-treated (1148 K for 1 h) condition. The nominal composition of the steel used is given in Table I. The specimens were aged at 753 K for $3\frac{1}{2}$ h and air cooled to room temperature. Hardness and tensile properties of the steel in the aged condition were evaluated using a Rockwell hardness tester and Instron model 8033 machine, respectively. Tensile tests were carried out at a crosshead speed of $0.003\ \mathrm{m\,min^{-1}}$. The results are given in Table II. Surface preparation of the specimens was done with SiC grit paper followed by thorough rinsing with water and acetone before hydrogen charging.

Hydrogen charging was carried out galvanostatically at a cathodic current density of $10 \pm 0.5\ \mathrm{mA\,cm^{-2}}$ in an aqueous solution of 5% H_2SO_4 containing $1\ \mathrm{g\,l^{-1}}$ thiourea as a hydrogen recombination poison.

TABLE I Nominal composition of 18 Ni 1800 MPa maraging steel

	C	Ni	Co	Mo	Ti	Al	Mn, Si	P, S	H	Fe
Quantity (wt %)	0.03	18	8	4.5	0.42	0.12	0.10	0.01	< 2 p.p.m.	Bal.

TABLE II Tensile properties of 18 Ni 1800 MPa maraging steel (solution-treated and aged condition)

Ultimate tensile strength	1768 MPa
% Elongation, GL = 25 mm	6%
% Reduction in area, diameter 3 mm	52%
Hardness	47 R_c

Hydrogen charging was carried out for time periods ranging from 1 h to a maximum of 5 h. The specimens were then subjected to hardness measurements, tensile testing and hydrogen analysis. Hydrogen analysis was carried out at Bhabha Atomic Research Centre, BARC, Bombay (using a LECO-RH-1E hydrogen analyser). The samples were fused in a graphite crucible in a flowing stream of argon gas, then the evolved hydrogen was estimated by thermal conductivity measurements.

Studies were carried out to determine the effect of baking on hydrogen embrittlement behaviour of the steel in both solution-treated and aged conditions. For this purpose tensile specimens were hydrogen charged at 10 ± 0.5 mA cm^{-2} current density for 2 h. Fractographic studies on hydrogen-charged and parent material were carried out with a Cambridge Stereoscan 250 MK3 scanning electron microscope.

3. Results and discussion

Fig. 2 shows the hydrogen pick-up in the 18 Ni 1800 MPa maraging steel round-bar specimens as the charging time is increased. It is noticed that the pick-up of hydrogen is very rapid, reaching 5 p.p.m. in 3 h and later it almost levels off at 7 p.p.m. Fig. 3 shows the variation of ultimate tensile strength (UTS) with hydrogen charging time for flat (both LT and TL orientation) and round-bar specimens. It is seen that up to 180 min hydrogen charging, the reduction in UTS is about 44%, whereas beyond 180 min the per cent reduction in UTS tapers off. The limiting value of UTS, i.e. 750 MPa, corresponds to the amount of maximum hydrogen that can be absorbed by the steel due to the hydrogen fugacity that prevailed over the surface of the specimen under the charging conditions, i.e. under the applied cathodic current density of 10 mA cm^{-2}. For a flat specimen, the material property behaviour is similar for both LT and TL directions. Ductility as measured by per cent elongation

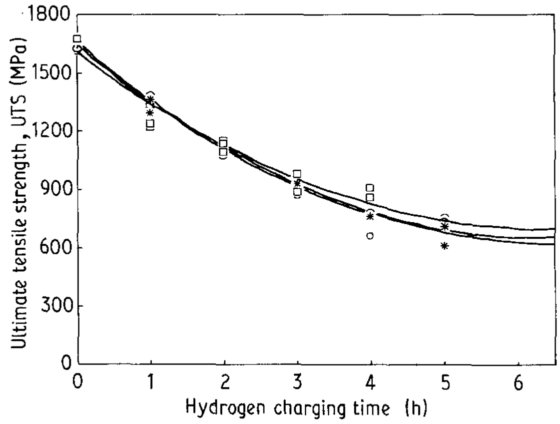

Figure 3 Variation of UTS with charging time in 18 Ni 1800 MPa maraging steel. (○) L direction, (*) TL direction, (□) round-bar.

(%El) changes from 6% (parent metal) to less than 2% at 1 h charging, corresponding to about 3 p.p.m. hydrogen. With further increase in charging time, the ductility decreased to less than 1%. For round-bar specimens, ductility measured by per cent reduction in area (%RA) decreases from 52% (parent metal) to less than 5% beyond 1 h hydrogen charging. This shows that %El and %RA are more sensitive to absorbed hydrogen, because more than 90% loss in these properties is seen after a charging period of 1 h. However, it was found that absorbed hydrogen had no effect on the hardness of the steel. Specimens with hydrogen charged for 5 h exhibited an R_c of 48 whereas the steel without hydrogen charge showed 47 R_c. Fig. 4 shows the decrease in UTS with increased absorbed hydrogen. It can also be observed here that the drop in UTS is about 45% up to 6 p.p.m. hydrogen, beyond which the drop in UTS is very much less, thus exhibiting behaviour similar to that shown in Fig. 3.

For low-alloy low-strength steels it has been observed that the hydrogen-charged specimens show increased strength associated with the decrease in ductility, which is attributed to a hydrogen-induced hardening effect [10, 11]. The present investigations show that 18 Ni 1800 MPa maraging steel specimens charged with hydrogen exhibit a decrease in tensile strength associated with a large decrease in ductility. It is reported [12] that the single structural feature which leads to environmental susceptibility is slip planarity, which is strongly dependent on stacking

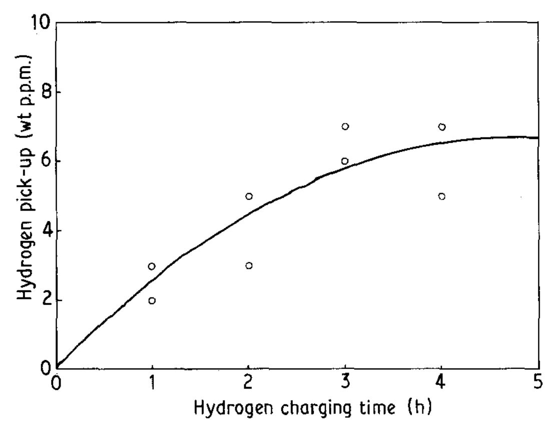

Figure 2 Amount of hydrogen pick-up with charging time in 18 Ni 1800 MPa maraging steel.

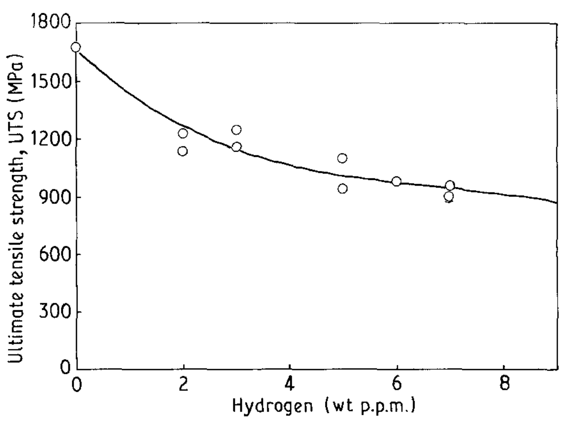

Figure 4 Variation of UTS with hydrogen content in 18 Ni 1800 MPa maraging steel.

fault energy (SFE) of the material. Low SFE makes cross-slip difficult and dislocations tend to move on single slip planes, and to lower the strength. Thus SFE should influence the hydrogen susceptibility of steels. The hydrogen-charged maraging steel shows a decrease in strength which, in turn, means that planar slip is increasing. Therefore, it is believed that SFE of maraging steel is lowered by absorbed hydrogen; this needs to be confirmed further by transmission electron microscopy.

Because, baking is normally done to materials that have picked up hydrogen during various processes such as melting, pickling, electroplating, etc., it was decided to study the effect of baking on hydrogen-charged maraging steel specimens. For baking studies, specimens (solution-treated as well as solution-treated and aged condition) were charged at a current density of $10 \pm 0.5\ \mathrm{mA\,cm^{-2}}$ for 2 h corresponding to a pick-up of about 5 p.p.m. hydrogen. Stress–strain curves for the 18 Ni 1800 MPa maraging steel for various conditions, including that for baking, are shown in Fig. 5.

3.1. Solution-treated material

The hydrogen-charged material shows a 5% increase in UTS which is similar to the hardening behaviour exhibited by low-alloy low-strength steels [10, 11]. Baking at 473 K for $3\frac{1}{2}$ h results in about 4% increase in UTS, thus showing a lesser strengthening effect. However, after baking at 753 K for $3\frac{1}{2}$ h, the strength level increases to the level of fully maraged steel. It is interesting to note here that the total strain to failure is the same for the solution-treated, solution-treated hydrogen-charged and, solution-treated hydrogen-charged and baked at 473 K conditions. However, for the solution-treated hydrogen-charged and baked at 753 K condition, strength and fracture strain are very close to that of solution-treated and aged condition.

3.2. Solution-treated and aged material

Initial charging to a level of 5 p.p.m. hydrogen decreases strength by 30% which is contrary to the (decrease in strength) behaviour of solution-treated material. Separate baking at 373 K for $3\frac{1}{2}$ h, 473 K, $3\frac{1}{2}$ h and 753 K for $3\frac{1}{2}$ h (which is equal to inducing double-aged treatment) results in a gradual recovery of strength up to 96% of the initial value. However, another set of experiments conducted on specimens which were subjected to maraging treatment twice (ageing at 753 K for $3\frac{1}{2}$ h followed by ageing at 753 K for $3\frac{1}{2}$ h) showed a much higher strength level which confirmed that absorbed hydrogen had not been totally baked out even after solution treatment, ageing, hydrogen charging and baking at 753 K treatment. Our distinct observation is that the plastic strain at fracture is almost zero for the solution-treated, aged and hydrogen-charged condition (Fig. 5 curve SAH) whereas for the cases of SA, SAHB1, SAHB2 and SD (Fig. 5), the total strain is almost the same.

3.3. SEM observations

The tensile-tested specimens were used for the fractographic studies. Fig. 6 shows fine dimples arising from microvoid formation and coalescence, typical of ductile failure in 18 Ni 1800 MPa maraging steel. As the charging time increased from 1 h to 3 h resulting in gradual pick-up of hydrogen, the fractographic features changed from coarse dimples (Fig. 7), to a mixed mode, i.e. dimple and intergranular features (Fig. 8), intergranular separation and transgranular cleavage fracture (Fig. 9) corresponding to 7 p.p.m. hydrogen absorbed by the steel. An increase in dimple size and a decrease in the number of microvoids cause accumulation of stress concentrations at these sites, resulting in an increase in average internal stress in the lattice. This decreases the effective stress experienced by dislocations in the intervoid regions and causes a decrease in the mean velocity of dislocations resulting in less plastic flow in these regions.

4. Conclusions

Based on the experiments conducted on 18 Ni 1800 MPa maraging steel the following conclusions are drawn.

1. For the solution-treated material, absorbed hydrogen results in small increase in strength only, whereas for the solution-treated and aged material it results in a large decrease in strength and ductility.

2. For the solution-treated material in the hydrogen-charged condition, baking at 753 K for $3\frac{1}{2}$ h gives

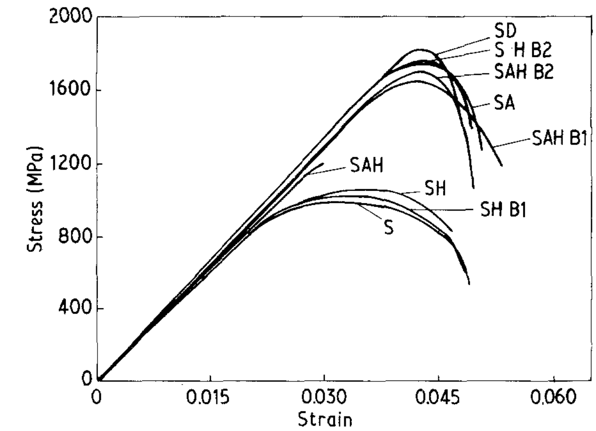

Figure 5 Stress–strain curves of 18 Ni 1800 MPa maraging steel for various conditions. S, solution treated; A, aged; D, double aged; H, hydrogen charged; B1, baked at 473 K; B2, baked at 753 K.

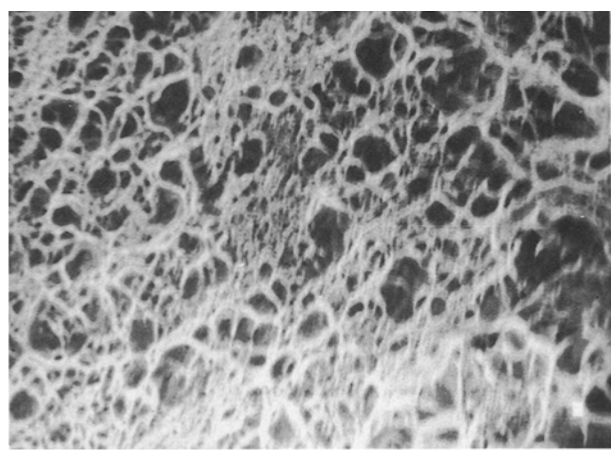

Figure 6 Fractograph of maraging steel (parent metal) without hydrogen charging; fine dimples seen (SEM) × 1300.

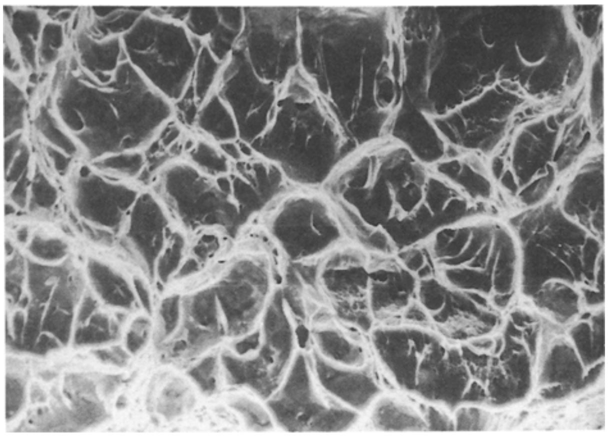

Figure 7 Fractograph of hydrogen-charged maraging steel; coarse dimples (SEM) × 1300.

Figure 8 Fractograph of hydrogen-charged maraging steel; mixed mode, dimples and intergranular fracture (SEM) × 2400.

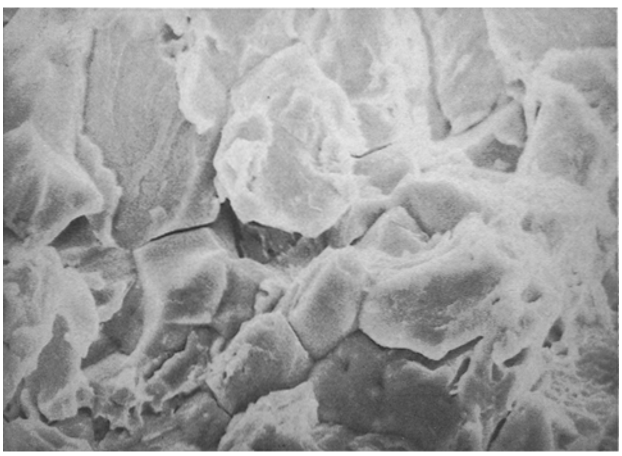

Figure 9 Fractograph of hydrogen-charged maraging steel; intergranular separation and transgranular separation (SEM) × 1700.

strength levels equal to that of the fully maraged condition.

3. For solution-treated and aged material in the hydrogen-charged condition, baking at 373, 473 and 753 K for $3\frac{1}{2}$ h recovers almost the initial strength level.

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