LASER SURFACE ANNEALING FOR IMPROVING HYDROGEN EMBRITTLEMENT RESISTANCE OF AGED INCONEL 718: EVALUATION OF THE EFFECTS OF PRECIPITATES

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ABSTRACT

Application of the aged Inconel 718 in hydrogen environment is seriously restraint by its high hydrogen embrittlement (HE) sensitivity. In previous researches, we have suggested the possibility and applicability of the laser surface annealing (LSA) process in improving the HE resistance of this alloy. Sequentially, a study on the effects of the precipitates in the Inconel 718 on its HE sensitivity was conducted in this research. Firstly, flat bar specimens were heat-treated to obtain various kinds of precipitation microstructures concerning the γ'' phase and the δ phase. Hydrogen was charged into the specimen by a cathodic charging process. The loss in reduction of area (RA) caused by hydrogen charging was used to assess the HE sensitivity. The HE sensitivity of the alloy was lowered with decreasing the volume fraction of γ''. Moreover, it was possible to increase the HE resistance of the aged alloy by dissolving the δ phase, keeping the strength at the same level as that of the common aged alloy. Thus, we concluded that both the δ phase and the γ'' phase affected the HE sensitivity of Inconel 718. Next, two kinds of notch tensile specimens were fabricated, one kind having δ phase and the other having no δ phase. All these specimens were aged via the same aging heat treatment process. The LSA process annealed a thin layer of the notch bottom of each specimen. One specimen of each kind was charged with hydrogen by the cathodic hydrogen charging process. Loss in the notch tensile strength (NTS) caused by hydrogen was used to evaluate the HE sensitivity. It was found that while the HE sensitivity of conventionally aged Inconel 718 was decreased by the LSA process, the HE sensitivity of the δ-free aged Inconel 718 could further be decreased. Therefore, for applications in hydrogen environments, it is possible to fabricate alloys with both good HE resistance and high strength by controlling the precipitation conditions, and to improve HE resistance further via applying the LSA process.

KEYWORDS

Laser annealing, Inconel 718, hydrogen embrittlement, cathodic hydrogen charging

1. Introduction

Inconel 718 is a nickel-iron base superalloy that is strengthened primarily by an ordered, body-centered tetragonal phase, i.e. γ'' [1, 2]. It has been widely used because it has good mechanical properties up to 923K [3]. However, the applications of aged Inconel 718 in hydrogen environment are restraint because of the serious HE sensitivity. Therefore, researchers have been engaging efforts in studying the hydrogen embrittlement of the aged alloy [4-9], and also in developing approaches to decrease this HE sensitivity [10~13].

Hydrogen causes serious ductility loss, and fracture mode change from the ductile micro-void coalescence

(MVC) mode to the brittle cleavage mode. It is widely accepted that there was a correlation between the HE sensitivity and the carbide particles because the carbide/matrix interfaces are irreversible trapping sites to hydrogen. However, there are few reports about the effects of the γ'' phase and the δ phase on the HE sensitivity of this alloy. This research was conducted to study the effects of these two phases.

Moreover, the LSA process that has been suggested to be effective in decreasing the HE sensitivity of aged Inconel 718 [10-13] was employed to anneal the notch locals of notch tensile specimens. The effect of the LSA on the HE sensitivity of notch tensile specimens was studied. Further, the possibility in fabricating parts that have both good HE resistance and high strength by employing LSA on δ-free aged Inconel 718 was suggested.

2. Material and experiments

2.1 Material

The chemical composition of the employed Inconel 718 is given in Table 1. Mitsubishi Materials Corporation supplied the material as forged, 3mm-thick plates. Before delivery, the plates had been annealed at 1253K for $1.2x10^3$s, and water quenched. The hardness of the as-received alloy was measured to be about 225HV (Load: 1.96N). The aged alloy was made via the standard double-aging process, i.e., 993K/8h/furnace cooled to 894K/8h/air cooled. The hardness of the double-aged alloy was measured to be about 450HV (Load: 1.96N).

Table 1 Chemical composition of Inconel 718 (wt %).

C	Mn	Si	P	S	Cr	Ni	Mo	Nb+Ta	Ti	Al	Co	B	Cu	Fe
0.03	0.15	<0.01	<0.001	0.002	18.54	52.07	3.04	5.35	1.01	0.51	0.02	0.004	0.01	REM

2.2 Preparation of specimens with various precipitation status

Several kinds of specimens with various precipitation conditions were fabricated via different heat treatment processes, as given in Table 2. The aged alloy, as stated in section 2.1, was just used in making aged specimens without any further processing. The specimens with other precipitation status of γ'' and δ were obtained by annealing the aged materials with various heat treatments, as given in Table 2. We carried out the annealing processes on the aged specimens to obtain different precipitation status of the γ'' phase instead of obtaining them by aging the totally annealed specimens, principally aimed at simulating the dissolution reaction of the γ'' phase that occurs in the LSA processes [10-13]. The two specimens of each kind were heat treated at the same time to ensure that they had identical precipitation status. After being polished, the hardness of each specimen was measured with a load as 1.96N and a holding time as 15s. For the specimens with partly dissolved γ'', the volume fraction of the γ'' phase was calculated from the measured hardness based on a relationship proposed in references [12, 13].

2.3 Cathodic hydrogen charging

Cathodic hydrogen charging was conducted with a salt consisted of $NaHSO_4$ and $KHSO_4$ (1mol:1mol).

Water vapor was bubbled through the molten salt to stir the molten electrolyte and to provide sufficient hydrogen ions. For the plate bar specimens, we used a charging time as 1.08x105s with an electric current as 0.5mA/mm2. For the notched specimens, a charging time as 9x104s and an electric current as 1mA/mm2 were employed. After being charged with hydrogen, the specimens were cleaned with distilled water and then stored in the liquid nitrogen till the tensile tests or the hydrogen concentration measurements.

Table 2 Precipitation status and the corresponding heat treatment processes

Precipitation status No.	Precipitation status	Heat treatment process	Hardness (HV, 1.96N)
1	γ" was totally dissolved	13K/s, 1303K, 0s, W.Q.*	225
2	γ" was partly dissolved	14K/s, 1219K, 0s, W.Q.*	260
3	γ" was partly dissolved	13K/s, 1204K, 0s, W.Q.*	350
4	Aged	Standard double-aging	450
5	δ-free, γ"-free	Annealing (1313K/3.6ks, W.Q.**)	182
6	δ-free, aged	Annealing (1313K/3.6ks, W.Q.**) → Standard double-aging	476

Note: * In the format of (heating rate, peak temperature, holding time, quenching method). ** In the format of (temperature/holding time, water quenched).

2.4 Tensile tests

Plate bar tensile specimens were made with the smooth part of sizes as 40^l x 7^w x 3^tmm. All tensile tests were conducted in air at the room temperature, with a slow crosshead speed of 1.67×10^{-3}mm/s. The RA and the tensile strength were used to assess the mechanical properties of each kind of alloy. The percent loss in RA caused by precharged hydrogen (as defined by Eq.(3)) was used to assess the HE sensitivity of these specimens.

$$\text{Percent loss in RA} = \frac{RA_0 - RA_H}{RA_0} * 100 \qquad (3)$$

Here, RA_0 and RA_H are RA of the hydrogen-free and hydrogen-precharged specimens, respectively.

Notch tensile specimens with precipitation conditions 1, 4, 6 (as given in Table 2) were fabricated. These specimens had 90° notches with 0.2mm root radiuses on both edges of gauge part that had sizes as 3^wx2^tmm. The percent loss in NTS caused by precharged hydrogen was used to evaluate the HE sensitivity of the notch tensile specimens, as defined by Eq.(4).

$$\text{Percent loss in NTS} = \frac{NTS_0 - NTS_H}{NTS_0} * 100 \qquad (4)$$

Here, NTS_0 and NTS_H are NTS of the hydrogen-free and hydrogen-precharged specimens, respectively.

2.5 Laser surface annealing of notch locals

LSA processes were conducted to anneal the notched regions of notch tensile specimens with a 2.5kW CO_2 laser in continuous wave mode. Coaxial argon flowed at a rate of 1.67×10^{-4} m^3/s. Two sets of laser conditions, i.e. defocus distance as 50mm and traverse speed as 5mm/s for conventional aged alloy (denoted as LSA-1), and defocus distance as 50mm and traverse speed as 8mm/s for δ-free aged specimen (denoted as LSA-2), were employed. The defocus distances were set with the central bottoms of the notches as irradiation surfaces. The traverse direction of the laser beam was along the root of the notch. The notch surfaces were polished with #1500 emery paper and then cleaned with ethanol. In each kind, two specimens were LSA processed: one was charged with hydrogen before the tensile test, the other was tensioned without charging.

3. Results and discussions

3.1. Effects of the δ phase and the γ" phase on hydrogen embrittlement

The partly annealed specimens (2 and 3 in Table 2) had γ" particles that had lower densities and smaller sizes than those in the as-aged alloy. For comparison, the hardness of these specimens was measured, and listed in Table 3. Further, using the relationship between the volume fraction of the γ'' phase, f, and the alloy hardness, H_V (Eq.(5) [12, 13]), f was calculated and also given in Table 3.

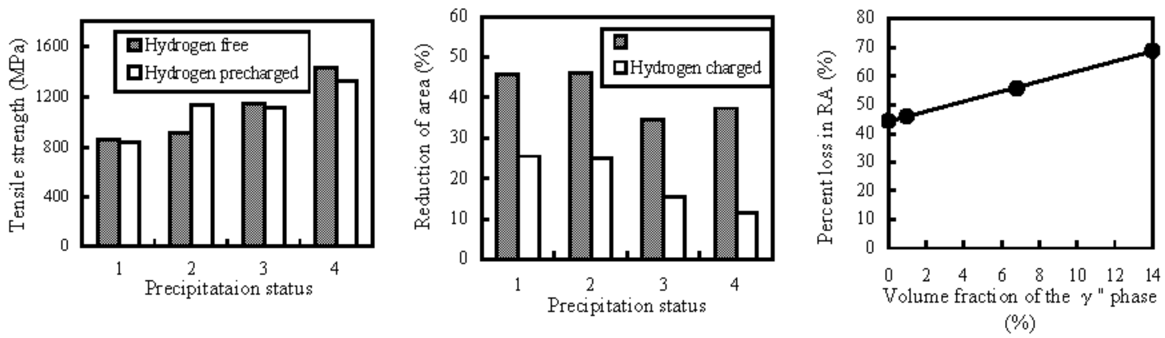
$$f = f_0 * \left(\frac{H_V - H_{V0}}{H_{V\max} - H_{V0}} \right)^{\frac{3}{2}} \quad (5)$$

Here, f_0 is the volume fraction of the γ'' phase. $H_{V\max}$ and H_{V0} are the hardness of the aged alloy and the totally annealed alloy, i.e., 450HV and 225HV, respectively. H_V is the hardness of an alloy that was annealed from the aged alloy via some heat treatment process, as those given in Table 3. f_0 was the volume fraction of the γ'' phase, viz. 14% [12].

Table 3 Hardness and the precipitation status of various specimens.

Precipitation status No.	Hardness (HV, Load: 1.96N)	Volume fraction of γ'' (%)
1	225	0
2	263	0.97
3	364	6.80
4	450	14

The tensile strengths and the RAs of the specimens with various precipitation status of the γ'', when hydrogen was precharged and free, are as shown in Fig.1 (a) and Fig.1 (b). Regardless of the hydrogen's charging condition, the tensile strength enhanced with increasing the volume fraction of the γ'' phase, while the reduction of area decreased contrarily. For all kinds of specimens, the reduction of area was decreased by precharged hydrogen. That is, all kinds of specimens had HE sensitivity. However, the reduction of area decreased from 25.5% of the totally annealed alloy to 11.6% of the aged alloy, i.e., the HE sensitivity varied with the precipitation status of the γ'' phase. To evaluate the correlation between the HE sensitivity and the precipitation status of the γ'' phase, the percent loss in RA that was caused by the precharged hydrogen was plotted in Fig.1 (c), with the volume fraction of the γ''-phase as the abscissa. The percent losses in RA were calculated using Eq.(3); the volume fractions of the γ'' phase were from Table 3. As shown in this figure, the

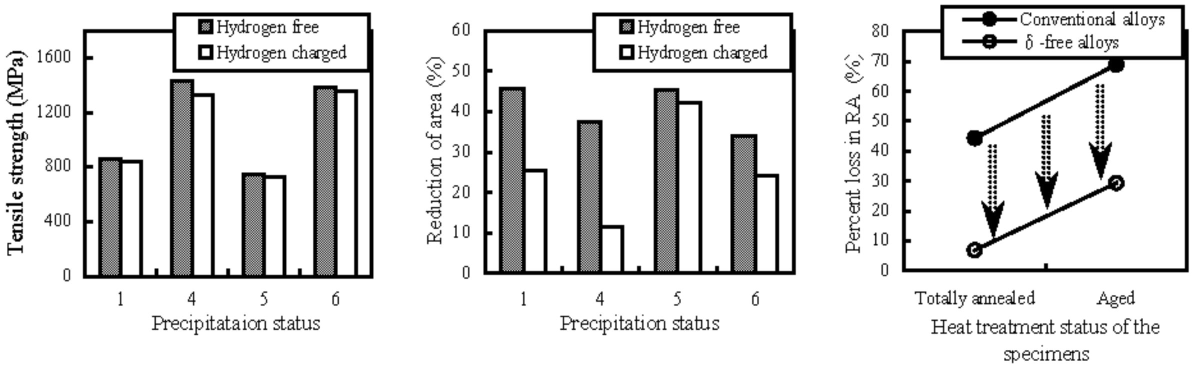


(a)

Fig.1 Effects of the precipitation status of the γ'' phase on the HE sensitivity of Inconel 718. (a) Tensile strength, (b) Reduction of area. (Precipitation status numbers are quoted from Table 2), (c) Percent loss in RA vs. the volume fraction of γ''.

percent loss in RA decreased almost linearly with increasing the volume fraction of the γ". When there was no precharged hydrogen, fractures all occurred in micro voids covalence mode; in contrast to this, when hydrogen was precharged, fractures generally originated from the surfaces and all showed brittle cleavage mode at the fracture origins. This suggested that the γ" particles had significant effects on the HE sensitivity of Inconel 718, and it is possible to decrease the HE sensitivity of aged Inconel 718 by dissolving the γ" phase.

As shown in Fig.2 (a), dissolution of the δ particles caused only small decrease in the tensile strength of both the annealed alloy (No.5 in Table 2) and the aged alloy (No.6 in Table 2), regardless of the charging status of hydrogen. However, as shown in Fig.2 (b), δ-free annealed specimen (No.5 in Table 2) had much higher RA than the conventionally annealed specimen (No.1 in Table 2) and δ-free aged specimen (No.6 in Table 2) had much higher RA than the conventionally aged specimen (No.4 in Table 2) when there was precharged hydrogen. As shown in Fig.2 (c), the loss in RA was suppressed to a large extent by dissolving the δ particles. Such results



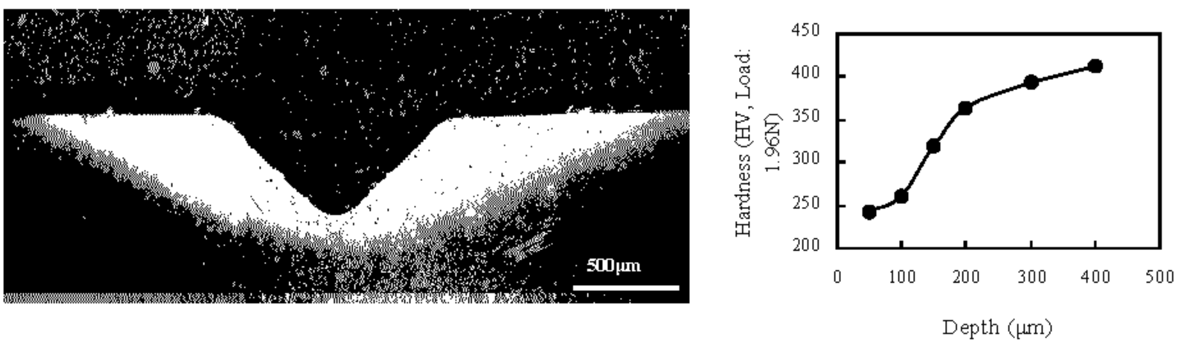
(a) (b) (c)

Fig.2 Effects of the precipitation status of the δ phase on the HE sensitivity of Inconel 718. (a) Tensile strength, (b) Reduction of area. (Precipitation status numbers are quoted from Table 2), (c) Percent loss in RA caused by precharged hydrogen with and without δ phase.

suggested that it was possible to fabricate alloys with both improved HE resistance and high strength by processing δ-free Inconel 718 with conventional double-aging process.

3.2. Decreasing the HE sensitivity by LSA process

LSA processes formed annealed zones, as the white part in Fig.3 (a). The hardness distribution along the depth direction at the notch root was shown in Fig.3 (b). Figure 4 shows the effects of the LSA processes on the



(a) (b)

Fig.3 Laser irradiation annealed the notch locals. (a) Optical image; (b) Hardness distribution.

HE sensitivity of Inconel 718. As shown in Fig.4 (a), there were no large losses in NTSs by the LSA processes (from 4 to LSA-1, and from 6 to LSA-2) in hydrogen-precharged specimens. However, it can be seen from Fig.4 (b) that the hydrogen-caused losses in NTSs were greatly decreased by LSA processes (comparison between LSA-1 and 4, and LSA-2 and 6). Moreover, it could be seen from Fig.4 (b) that LSA-2 had lower loss in NTS than LSA-1, i.e., LSA-2 had better HE resistance. This suggests that the best approach for good HE resistance

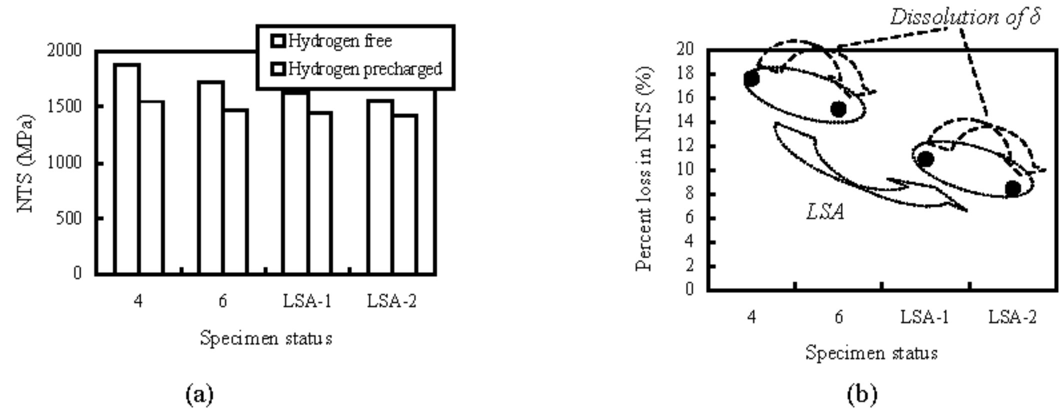


Fig.4 Enhancement in HE resistance of Inconel 718 by LSA processes. (a) NTS; (b) Percent loss in NTS (Precipitation status numbers are quoted from Table 2).

and high strength is applying LSA on δ-free aged specimens.

4. Conclusions

(1). The γ'' phase deteriorated the HE resistance of Inconel 718. The loss in RA caused by precharged hydrogen increased almost linearly with increasing the volume fraction of the γ'' phase.

(2). The HE sensitivity was greatly decreased by dissolving the δ particles, keeping high strength level.

(3). Applying LSA processes on notch tensile specimens decreased the HE sensitivity. It suggested that the best approach for good HE resistance and high strength is applying LSA on δ-free aged specimens.

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