

Influence of Environment on Fatigue Crack Growth in Titanium Aluminides and Hydrogen Evolution Behavior Evaluated by Thermal Desorption Spectroscopy

K. Komai¹, K. Minoshima¹, K. Obara² and N. Minamino¹

¹ Department of Mechanical Engineering, Kyoto University

² Ishikawajima-Harima Heavy Industries, Co., Ltd.

ABSTRACT: The influence of environment on fatigue crack growth behavior was investigated in duplex titanium aluminides, and the hydrogen evolution kinetics was analyzed by thermal desorption spectroscopy. The tensile strength decreases in the order of the extent of the water molecule content in the environment. When cathodic charging is applied, the fatigue crack growth rate becomes higher than in dry air, particularly in the higher stress intensity factor range. The hydrogen evolution rate is increased by cathodic charging, with lower temperature peaks and higher ones. The peaks at lower temperatures are correlated with the decomposition of hydrides and de-trapping of hydrogen from microstructural imperfections such as microvoids. As-received materials also show an evolution peak at a higher temperature, and the evolution rate is almost independent of cathodic charging. In addition, the evolution rate at a high temperature (above 800°C) is increased by cathodic charging. The hydrogen is considered to have an important role on fatigue crack growth acceleration.

INTRODUCTION

The mechanical properties required in many technological fields are high-strength, low-specific density, high wear resistance, and heat-resistance. Of the various structural materials, intermetallic compounds are one of the promising candidate materials for high-temperature applications. However, the disadvantage is that the intermetallic

TABLE 1: Chemical compositions of TiAl intermetallic compounds, mass %.

C	Al	O	N	Ti
Less than 0.01	35.9	0.05	0.003	Bal.

compounds are brittle at room temperature. This brittleness comes from not only the intrinsic nature of the material but also extrinsic or environmental effects [1, 2]. It is believed that the fatigue crack growth enhancement and a decrease in ductility or tensile strength are a result of hydrogen-induced embrittlement (hydrogen-embrittlement) due to the presence of moisture in the test environment [1]. In this investigation, the effects of environment on fatigue crack growth behaviour were investigated in duplex titanium aluminide intermetallic compounds. The effects of a cathodically charged condition in an aqueous solution on fatigue crack growth behaviour were evaluated. The hydrogen evolution kinetics was also studied by means of thermal desorption spectroscopy, and the influence of environment on fatigue crack growth is discussed.

EXPERIMENTAL PROCEDURES

The materials tested was a duplex material of equiaxed γ phase and $(\alpha+\gamma)$ lamellar structures, whose chemical compositions are shown in Table 1. Slow-strain rate tests under displacement control were performed using smooth specimens having 5 mm width, 5 mm thickness and a 10 mm gauge-length. The tests were conducted in vacuum (2.4×10^{-4} Pa), laboratory air, and in de-ionized water. The displacement rates were 1.67×10^{-3} mm/s (0.1 mm/min) in laboratory air and 8.33×10^{-5} mm/s (0.005 mm/min) in vacuum and in water.

Crack propagation tests were conducted using CT specimens. The tests were conducted in laboratory air, dry air, dry nitrogen gas, and under a cathodically charged condition in a $1/3$ mol/L H_3BO_3 + $1/30$ mol/L KCl solution. The dew point of dry air was -70.4 °C. The tests were conducted at a room temperature of about 21-24 °C. The current density of the cathodic charge was 50 A/m², and the solution was kept at 25°C. Fatigue crack growth tests at a stress ratio R of 0.1 were performed at a stress cycle frequency of 3 - 5 Hz. In the cathodically charged condition, the stress cycle frequency

TABLE 2: Results of slow strain rate tests of the duplex material.

	Water	Laboratory air	Vacuum
Tensile strength	510MPa	542MPa	593MPa
Elongation at break	1.09%	1.13%	1.93%
Number of samples tested	6	8	4

was set at 2 Hz or 0.1 Hz.

Hydrogen evolution behaviour was evaluated by means of thermal desorption spectroscopy (TDS). The analyzed samples were the as-received materials preconditioned in laboratory air and a cathodically charged sample under the same condition as that used in fatigue tests.

EXPERIMENTAL RESULTS AND DISCUSSION

Slow Strain Tests of Smooth Samples

Table 2 summarizes the mechanical properties by slow strain rate testing. The tensile strength decreased in the order of vacuum, laboratory air, and water. This indicates that the TiAl was embrittled even in laboratory air. These results are consistent with those reported before [1, 2]. The fracture surfaces were translamellar and interlamellar with γ -phase transgranular cracking characterized by river patterns, irrespective of the test environment. The other interesting point was that the number of small surface cracks was larger in laboratory air or in water than in vacuum. We must note that TiAl is so sensitive to an environment, and that even laboratory air can embrittle the material, resulting in a decrease in tensile strength compared with that in vacuum.

Fatigue Crack Growth Properties

Figure 1 shows the effects of environment on fatigue crack growth rate of the duplex material in the L-R crack plane orientation. In terms of ΔK , the crack growth rate in laboratory air was slightly higher than that in dry air, inclusive of the threshold stress intensity factor. This acceleration was clearly observed in the intermediate ΔK region. When the growth rate was plotted against ΔK_{eff} , the crack growth rate may be higher than that in dry air, in particular at low ΔK_{eff} . This indicates that the growth rate can be

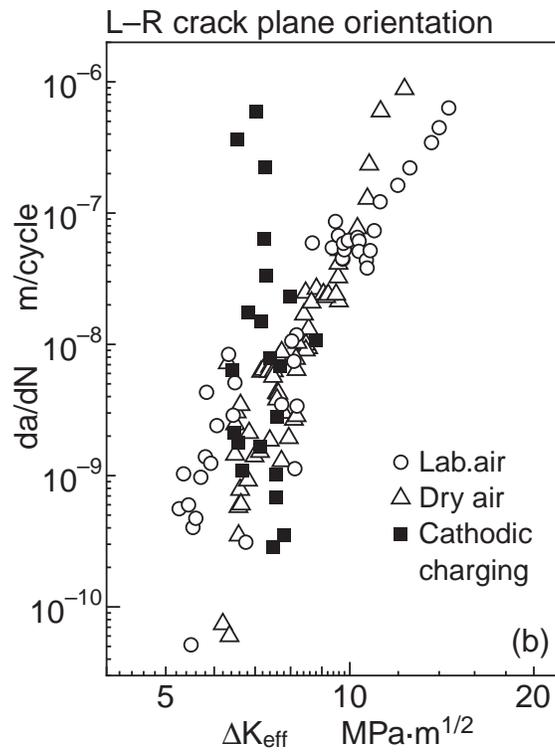


Figure 1: Effect of environment on fatigue crack growth rate in the L-R crack plane orientation in terms of ΔK_{eff}

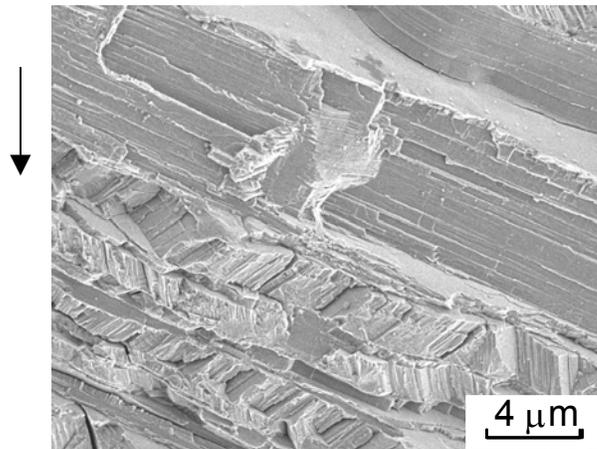


Figure 2: The fracture surface in the L-R crack plane orientation fatigued in laboratory air. Arrow shows crack growth direction

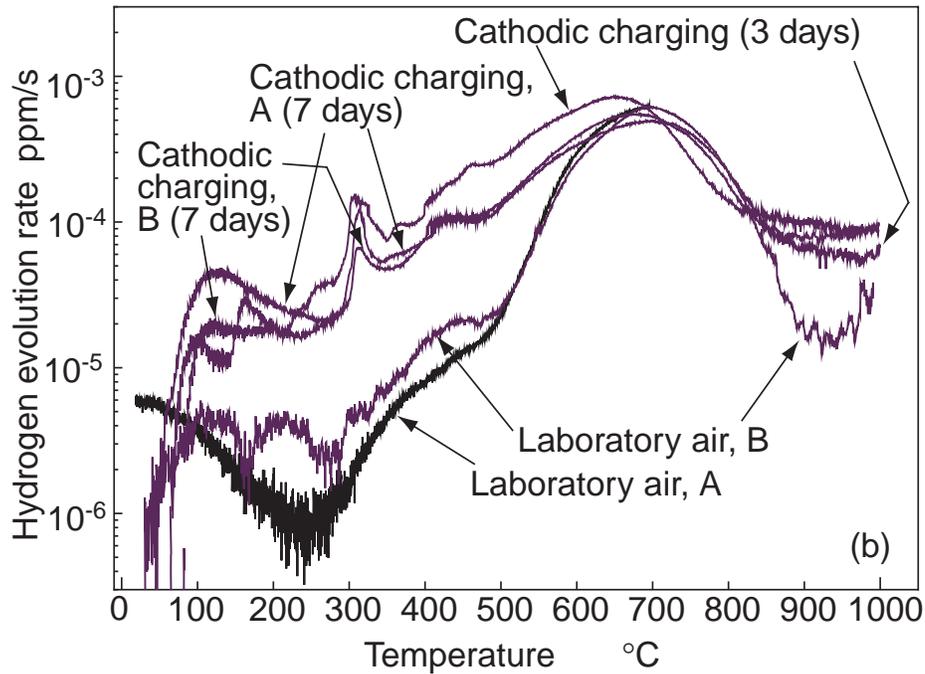


Figure 3: Hydrogen evolution rate as a function of temperature.

accelerated by water molecules in laboratory air. Under the cathodically charged condition, the threshold stress intensity factor range was almost equal to that in dry air. However, the crack growth rate was highly dependent on ΔK or ΔK_{eff} , and the growth rate was much enhanced at higher ΔK (ΔK_{eff}) values compared with those in dry and laboratory air.

In the case of the L-R crack plane orientation, the crack grew with cleavage fracture in the γ phase and translamellar fracture, irrespective of the environment. However, the difference of fracture surface between dry air and laboratory air or under the cathodically charged condition was that the translamellar fracture in dry air was rough and in part with almost equally spaced steps, whereas translamellar fracture in laboratory air or under the cathodically charged condition partly exhibited smooth and featureless zones (see Figure 2).

Thermal Desorption Spectra of Hydrogen

Figures 3 show the hydrogen evolution rate as a function of temperature. The evolution rate of the sample kept in laboratory air was negligible at the lower temperature. However, it increased beyond $T > 300^\circ\text{C}$, and had a peak

temperature of about 600 to 750°C. When the samples were cathodically charged, the hydrogen evolution rate increased from that kept in laboratory air, in particular at a lower temperature range. The evolution rate increased with an increase in cathodic charging time, with two peak temperatures of about 100 and 300°C. Although the evolution rates of the samples charged for three and seven days were almost equal, a substantial increase in the hydrogen evolution rates from that of the sample kept in laboratory air were observed, particularly at the lower temperature range.

As for higher temperatures the maximum hydrogen evolution rate, which was observed at around 600 to 750°C, was almost independent of cathodic charging. The other interesting point is that the hydrogen evolution rate at an extremely high temperature (above 800°C) was higher for cathodically charged samples than that kept in laboratory air.

The lower peaks observed at about 125 to 425°C are considered to be mainly related to hydride decomposition. This is consistent with the results that the sample which was kept in laboratory air had no temperature peak at a low temperature. The several peaks we did observe indicated that the samples used had different structures of hydrides or different sites in a hydride crystal such as octahedral or tetrahedral sites. It is also reported that structural imperfections such as microvoids and internal cracks can be a hydrogen trap site for a duplex TiAl, and these have a lower peak temperature ranging from 150 to 330°C[3]. In general hydrides are a very hard phase, have low ductility, and cause an increase in hardness of the TiAl matrix, in particular within surface layers. This indicates that these hydrides induce micro-cracking under loading. This is consistent with the results that small cracks were formed more in a water environment than in vacuum.

In the case of fatigue crack growth, the role of hydrides formed on the sample surface layers during cathodic charging may be smaller than that required to induce crack initiation. However, the crack growth rates are increased under the cathodically charged condition in comparison to that conducted in dry air. This is considered to be brought about by hydrogen trapped in the matrix alloy. The thermal desorption analyses showed that the hydrogen evolution rate was increased at a higher temperature than that conducted in dry air. The higher peak temperature range agrees with the reported peak temperature for the de-trapping of hydrogen in the alloy matrix [3,4]. The thermal desorption spectra showed that as-received materials also had a similar high temperature peak with a high maximum value, indicating that the hydrogen was already trapped during manufacturing. This leads to the speculation that the crack growth rate may be accelerated also in dry air. This is because the hydrogen diffusion in the

matrix can be enhanced by dislocation motion, and hydrogen concentrates at the crack tips under loading. This is supported by the fact that hydrogen is observed along slip bands in TiAl [5] and Ni₃Al [6] from studies using a hydrogen microprint method.

Under cathodic charging, the hydrogen content in the matrix alloy is increased, and the diffusion can be promoted by dislocation motion under fatigue loading. This may lead to a higher hydrogen concentration in the matrix, particularly in the crack tip region. At the same time hydrogen can enter from the crack tip during fatigue. These are the reason why the crack growth rate was higher under cathodic charging than when conducted in dry air where additional hydrogen entry was extremely small..

CONCLUSIONS

Fracture strength under a slow strain rate and fatigue crack growth behavior was investigated in duplex titanium aluminides. Special attention was paid to the effects of environment on fatigue crack growth. Hydrogen evolution kinetics was analyzed by means of the thermal desorption technique. The investigation gave the following results.

1. The tensile strength of the duplex material decreases in order of water molecule content in an environment: the strength in vacuum being the highest, and decreases in order of laboratory air and water.
2. Under cathodic charging, the crack growth rate is accelerated at higher ΔK (ΔK_{eff}) values, whereas in the low ΔK (ΔK_{eff}) region the crack growth rate is almost equal to the fatigue results in dry air.
3. By means of thermal desorption spectroscopy, analyses of hydrogen trap sites in the titanium aluminides are possible. The hydrogen evolution rate is increased by cathodic charging. The lower peak temperatures observed are about 100 and 300°C and may also relate with the decomposition of hydrides and detrapping of hydrogen from micro structural imperfections such as microvoids. The peak temperature with the greatest evolution rate is around 600 to 750°C. As received materials also shows an evolution peak at a high temperatures, and the evolution rate is almost independent of cathodic charging. In addition, the evolution rate at high temperature (above 800°C) is increased by cathodic charging.. The trapped hydrogen and the one enters from the crack tip during fatigue cycling are considered to have an important role on fatigue crack growth acceleration.

REFERENCES

1. Takasugi, T. and Hanada, S. (1992) *J. Mater. Res.* **7**, 2739.
2. Liu, C.T. and Kim, Y-W. (1992) *Scripta Metallurgical et Materials* **27**, 599.
3. Takasaki, A., Furuya, Y, Ojima, K. and Taneda, Y. (1995) *Journal of Alloys and Compounds* **224**, 299.
4. Numata, H., Amano, M., Tomizuka, I. and Tsujimoto, T. (1997) *J. Japan Inst. Metals* **61**, 105
5. Yoshioka, M., Ueno, A. and Kishimoto, H. (2000) *Committee on Fracture Mechanics, Soc. Mater. Sci., Japan*: **49**, 1.
6. Itoh, G., Haramura, N. and Ihara, T. (1999) *J. Japan Inst. Metals*: **63**, 593.