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## The environment-induced cracking of as-cold rolled Ni<sub>3</sub>(Si,Ti) and Ni<sub>3</sub>(Si,Ti) with 2Mo in sodium chloride solutions



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#### ABSTRACT

The environment-induced cracking (EIC) of as cold-rolled Ni<sub>3</sub>(Si,Ti) and Ni<sub>3</sub>(Si,Ti) with 2Mo has been investigated as functions of applied stress, chloride ion concentration, test temperature, and pH by using a constant load method in NaCl solutions. The EIC susceptibility of both intermetallic compounds increased with increasing Cl<sup>-</sup> ion concentration and test temperature; and increased with decreasing pH. Both intermetallic compounds had the same transgranular fracture appearance and the relationship between  $\log t_{\rm f}$  (time to failure) and  $\log l_{\rm ss}$  (steady state elongation rate) became the identical straight line irrespective of applied stress, Cl<sup>-</sup> ion concentration, test temperature, and pH, which means that  $l_{\rm ss}$  becomes a relevant parameter for predicting  $t_{\rm f}$ . The EIC susceptibility of Ni<sub>3</sub>(Si,Ti) with 2Mo was lower than that of Ni<sub>3</sub>(Si,Ti), which showed the beneficial effect of Mo. From the results obtained, EIC of both intermetallic compounds was presumed to take place by hydrogen embrittlement (HE).

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#### 1. Introduction

Ni<sub>3</sub>(Si,Ti) intermetallic compounds with L1<sub>2</sub> structure have unique strength and ductility properties; that is, (1) an increase in flow strength with increasing temperature and (2) high ductility over a wide range of test temperature [1,2]. In particular, their strength level was extremely high compared to those of other L12 ordered intermetallic compounds developed as advanced materials [3]. On the other hand, Ni<sub>3</sub>(Si,Ti) intermetallic compounds have superior mechanical properties compared to those of conventional alloys such as nickel-base alloys, steels and stainless steels. In addition, this compound has shown an excellent oxidation resistance in air at ambient and elevated temperatures [4]. Furthermore, an improvement in the mechanical property of the Ni<sub>3</sub>(Si,Ti) compound was also attempted with macro-alloying, i.e., addition of molybdenum, which enhanced tensile strength and hardness and improved the elongation at various operational temperatures [5]. On the other hand, the compounds have shown the susceptibility to environmental embrittlement (specifically hydrogen embrittlement) at ambient temperature in moist air and hydrogen gas [6–8]. The addition of boron to Ni<sub>3</sub>(Si,Ti) was carried

out to suppress the environmental embrittlement [8], but led to the intergranular attack for this as-homogenized compound in acidic solutions, due to boron segregation at grain boundaries [9].

However, with regard to as cold-rolled Ni<sub>3</sub>(Si,Ti) and Ni<sub>3</sub>(Si,Ti) with 2Mo, there is little study on environment induced cracking (EIC) in aqueous solutions, Therefore, we have investigated the EIC behavior of those intermetallic compounds in chloride solutions as functions of applied stress, test temperature, Cl<sup>-</sup> ion concentration, and pH by using a constant load method. The objectives of the paper are (1) to elucidate the EIC behavior of those compounds and (2) to presume an EIC mechanism.

#### 2. Experimental

Ni–11 at.% Si–9.5 at.% Ti and Ni–11 at.% Si–7.5 at.% Ti–2 at.% Mo compounds with the addition of 50 wt. ppm of boron were prepared by using an arc melting method under an argon gas atmosphere. Hereafter, the ternary alloy will be written as Ni $_3$ (Si,Ti) and the quaternary alloys will be denoted as Ni $_3$ (Si,Ti) with 2Mo. The nominal compositions of those prepared materials are given in Table 1.

Those intermetallic compounds were homogenized at 1323 K for 48 h under an argon atmosphere and then cooled with a cooling rate of 283 K/min in a vacuum furnace. Homogenized ingot was warm-rolled at 573 K in air to the desired thickness and then cold-rolled to 1.2 mm thickness in 75% reduction. It has been known that Ni<sub>3</sub>(Si,Ti) consists of an L1<sub>2</sub> intermetallic compound phase while Ni<sub>3</sub>(Si,Ti) with 2Mo is composed of two phases of an L1<sub>2</sub> phase and a mixture of L1<sub>2</sub> and fcc Ni [5]. The ultimate tensile stress, yield stress, and strain of as cold-rolled Ni<sub>3</sub>(Si,Ti) were 2004 MPa, 1857 MPa, and 5.8%, while those of as cold-rolled Ni<sub>3</sub>(Si,Ti) with 2Mo

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**Table 1**Nominal compositions of prepared materials.

Compound	С	Si	Mn	P	S	Ni	Cr	Мо	Al	Fe	Ti	В
	at.%											ppm
Ni <sub>3</sub> (Si,Ti)	_	11.0	_	_	_	79.5		_	_	-	9.5	50
$Ni_3(Si,Ti) + 2Mo$	-	11.0	-	-	-	79.5	-	2.0	-	-	7.5	50

were 2059 MPa, 1925 MPa, and 3%, where the low strains of both intermetallic compounds mean that the compounds corresponded to the brittle materials. The specimens were manufactured in gauge length of 10 mm, width of 2 mm and thickness of 1.2 mm, where they were cut perpendicularly to rolling direction. Prior to the experiments, the specimens were polished to 1000 grit emery paper, degreased with acetone in an ultrasonic cleaner and then washed with distilled water. As cold-rolled specimens thus prepared were used for all experiments in this study. The test solutions used were sodium chloride solutions with various chloride ion concentrations (0–5 kmol/m $^3$ ) prepared from distilled water and guaranteed grade reagent. The test temperature was in the range of 333–373 K with an accuracy of  $\pm 1$  K. The pH was in the range of 2–10. All experiments were carried out under an open circuit condition.

A lever-type constant load apparatus with a lever ratio of 1:10 to which three specimens can be separately and simultaneously attached was used with a cooling system on the top to avoid evaporation of the test solution during experiments. The specimens set into the EIC cell were insulated from rod and grip by surface oxidized zirconium tube. Elongation of the specimen under the constant load condition was measured by an inductive linear transducer with an accuracy of  $\pm 0.01$  mm.

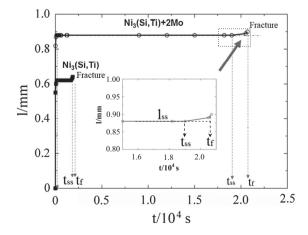
#### 3. Results

#### 3.1. Corrosion elongation curve and three parameters ( $t_b$ $l_{ss}$ and $t_{ss}$ )

Fig. 1 shows an example of the corrosion elongation curves (elongation versus time) up to failure for as cold-rolled Ni<sub>3</sub>(Si,Ti) and Ni<sub>3</sub>(Si,Ti) with 2Mo at a constant applied stress of 1900 MPa in 0.5 kmol/m<sup>3</sup> NaCl solution at 313 K. They consisted of three time intervals with an initial sharp rise of elongation, constant elongation and second rise in elongation to failure. In addition, as shown in the magnified figure inserted in Fig. 1, the three parameters were obtained for each specimen at every applied stress: time to failure ( $t_{\rm f}$ ), steady-state elongation rate ( $l_{\rm ss}$ ) for the straight part of the corrosion elongation curve, and transition time ( $t_{\rm ss}$ ), where  $t_{\rm ss}$  is the time when the elongation deviates from linearity. However, we could get only  $l_{\rm ss}$  from the corrosion elongation curve, when it was not fractured within a laboratory time scale (<10<sup>7</sup> s).

#### 3.2. Applied stress dependence of three parameters

Fig. 2 shows the relationship between applied stress and  $t_f$  in 0.5 kmol/m<sup>3</sup> NaCl solution at 313 K for both Ni<sub>3</sub>(Si,Ti) and



**Fig. 1.** Corrosion elongation curve of Ni<sub>3</sub>(Si,Ti) and Ni<sub>3</sub>(Si,Ti) with 2Mo at a constant applied stress 1900 MPa in 0.5 kmol/m³ NaCl solution at 313 K.  $t_f$ : time to failure,  $l_{ss}$ : steady state elongation rate,  $t_{ss}$ : transition time.

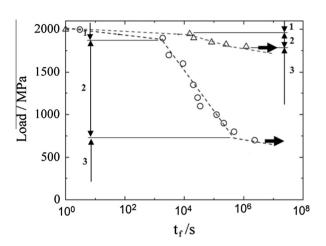


Fig. 2. The relationships between applied stress and time to failure in 0.5 kmol/m<sup>3</sup> NaCl solution at 313 K.  $\bigcirc$ : Ni<sub>3</sub>(Si,Ti), and  $\triangle$ : Ni<sub>3</sub>(Si,Ti) with 2Mo.

Ni<sub>3</sub>(Si,Ti) with 2Mo. The relationships were found to be divided into three regions (1–3 in Fig. 2), where arrows in region 3 mean that the specimens were not fractured within a laboratory time scale ( $<10^7$  s). The maximum applied stress ( $\sigma_{max}$ ) and the minimum applied stress ( $\sigma_{min}$ ) in region 2 become larger for Ni<sub>3</sub>(Si,Ti) with 2Mo than for those of Ni<sub>3</sub>(Si,Ti), and the  $t_f$  of Ni<sub>3</sub>(Si,Ti) with 2Mo was longer than that of Ni<sub>3</sub>(Si,Ti) in region 2.

Fig. 3 shows the relationship between applied stress and  $l_{\rm ss}$  for Ni<sub>3</sub>(Si,Ti) and Ni<sub>3</sub>(Si,Ti) with 2Mo in 0.5 kmol/m³ NaCl solution at 313 K. It was also found that the relationships were divided into three regions (1–3 in Fig. 3), corresponding to those in Fig. 2. The steady-state elongation rate,  $l_{\rm ss}$  of Ni<sub>3</sub>(Si,Ti) with 2Mo was smaller than that of Ni<sub>3</sub>(Si,Ti) in region 2. Furthermore, in region 3, both intermetallic compounds had no fracture with the order of  $10^{-11}$  m/s within a laboratory time scale (<10<sup>7</sup> s). Fig. 4 shows the relationship between applied stress and  $t_{\rm ss}/t_{\rm f}$  for both Ni<sub>3</sub>(Si,Ti) and Ni<sub>3</sub>(Si,Ti) with 2Mo in 0.5 kmol/m³ NaCl solution at

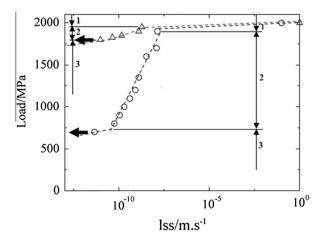


Fig. 3. The relationships between applied stress and steady state elongation rate in  $0.5 \text{ kmol/m}^3 \text{ NaCl}$  solution at 313 K.  $\bigcirc$ : Ni<sub>3</sub>(Si,Ti), and  $\triangle$ : Ni<sub>3</sub>(Si,Ti) with 2Mo.

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