

## Friction welding of pure titanium and pure nickel

A. Fuji\*1, Y. Horiuchi2 and K. Yamamoto3

The joint tensile strength and metallurgical properties of a friction welded joint of commercially pure Ti and pure Ni has been investigated in as welded and post-weld heat treated conditions. While friction pressure did not significantly impinge on joint tensile strength, joint tensile strength was affected by friction time. A 1–1.5  $\mu$  m thick interlayer is essential to join pure Ti and pure Ni using friction welding. A maximum joint tensile strength of 450 MPa was achieved and the joint fractured in the Ti original (not heat affected zone) substrate, i.e. the joint efficiency was approximately 112% relative to Ti substrate and 94.5% relative to Ni substrate. The joint tensile strength abruptly decreased as heating temperature was increased to 873 K and/or the Larson–Miller parameter was increased to approximately 19-20 $\times 10^3$ . The joint tensile strength rapidly decreased with increasing interlayer thickness up to approximately 10  $\mu$  m, and then remained constant for further increase in interlayer thickness. Four layers occurred at the interface of joints heated to more than 873 K, namely Ti<sub>2</sub>Ni, TiNi, TiNi<sub>2</sub>, TiNi<sub>3</sub>. The fracture of heated joints propagated mainly in the Ti<sub>2</sub>Ni layer and/or at the interface between the TiNi and TiNi<sub>3</sub> layers.

### Introduction

Welded joints of dissimilar metals are important because of their considerable advantages in many industrial applications.<sup>1-3</sup> The joints can improve the performance of many types of equipment, and save weight and cost in the transportation field, especially automobile and aerospace. However, welding dissimilar materials has several severe problems at the industrial level. Problems occur when dissimilar joints are welded using fusion welding processes. These processes conventionally produce intermediate layers (hereafter called interlayers) consisting of brittle intermetallic compound phases at the interface of both substrates to be joined. These interlayers degrade the mechanical and metallurgical properties of the joint. Another problem occurs when dissimilar welded joints are operated in an elevated temperature environment; interlayers can grow during operation and cause equipment failures. There have been many and wide ranging studies investigating diffusion phenomena and interlayer growth at the interfaces of dissimilar welded joints and/or diffusion couples.

Titanium (Ti), nickel (Ni) and their alloys have many excellent mechanical and metallurgical properties, therefore, dissimilar joints of pure Ti and pure Ni and/or their alloys may be useful for many industrial applications. There has been some research reported on the diffusion phenomena of Ti–Ni systems; for example, solute elements in  $\beta$ -Ti and in Ti–Ni systems at elevated temperature,<sup>4</sup> to determine the

phase diagram between 'TiNi<sub>3</sub>' and 'Ti<sub>2</sub>Ni'.<sup>5</sup> Honma and Takei clarified the effect of heat treatment on the martensitic transformation in NiTi compound.<sup>6</sup> Although Hinotani and Ohmori reported on the microstructure of diffusion bonded Ti–Ni interface,<sup>7</sup> they did not examine in detail the mechanical properties of the joint. Hanlon et al. reported the effect of martensitic transformation on the electrical and magnetic properties of NiTi.<sup>8</sup> Although much research has been reported regarding the Ti–Ni system, few studies have investigated the dissimilar welding of Ti–Ni. The fusion welding of Ti–Ni and their alloys is actually impossible at the industrial level due to the generation of brittle interlayers as described above. Although one solution to welding dissimilar joints is to apply solid phase joining processes, there have been few research reports to date.

The author has been investigating the joining of Ti and Ni and their alloys using a friction welding method; that is, one of the solid phase joining processes. Generally speaking, friction pressure and friction time are the most important parameters for dissimilar friction welding because they directly produce brittle intermetallic interlayers at interfaces during the friction welding operation. Therefore, the first trial was set up to determine the effect of friction pressure and friction time on the tensile strength and metallurgical properties of pure Ti and pure Ni friction welding joints.

Another problem with dissimilar material joints occurs when they are operated in an elevated temperature environment: interlayers grow during operation and cause equipments to fail. There are very few studies reporting on the welding of pure Ti to pure Ni and/or their alloys. Ti/Ni dissimilar joints are rarely used at elevated temperatures, for example, more than 773 K. However, it is important to clarify the effect of interlayers at the interface of both substrates on the mechanical and the metallurgical properties of dissimilar joints for industrial use. Further investigations were carried out to determine the effect of interlayers on joint tensile strength and metallurgical properties when applying post-weld heat treatments (PWHT), that is, forcibly generating interlayers at the joint interface.

## Experimental procedure

### As welded joint

Commercially pure Ti and pure Ni base materials of 16 mm diameter round bar were used throughout this experiment. The pure Ti bar contained (wt-%) 0.006%H, 0.12%O, 0.006%N, 0.048%Fe, balance Ti, and had a yield strength of 273 MPa, an ultimate tensile strength of 402 MPa, and an elongation of 33.2%. The pure Ni had the chemical composition 0.01%C, 0.10%Si, 0.24%Mn, 0.11%Cu, 0.18%Fe, balance Ni, and was supplied with a yield strength of 476 MPa, an ultimate tensile strength of 515 MPa, and an elongation of 14.5%. All faying surfaces were polished with a buff before joining to minimise the effect of surface roughness on joint properties. A

brake type friction welding machine was employed for joining. According to former research, friction pressure and friction time strongly affect the generation of interlayers consisting of brittle intermetallic compound phases during the friction stage. In this research, friction welding conditions were varied as follows: friction pressures 100, 150, and 200 MPa; friction times from 0.2 to 4 s. Friction speed, upsetting pressure, and upsetting time were fixed at  $25 \text{ s}^{-1}$  ( $1500 \text{ rev min}^{-1}$ ), 250 MPa, and 6 s, respectively. After joining, some joints were machined to 13 mm diameter and 50 mm parallel length with the joint interface at the mid-position, for joint tensile tests. All tensile tests were carried out at room temperature. The specimens for metallurgical testing were derived from the joint interface, and were also of diameter 13 mm. SEM with EDS observation was carried out to observe the interlayer and to analyse elemental chemical distributions across the joint interface.

#### Post-weld heat treated joint

Based on the results of the as welded joint investigation, friction welding parameters were selected for post-weld heat treated joint tests as: friction speed  $25 \text{ s}^{-1}$ ; friction pressure 150 MPa, friction time 2 s, upsetting pressure 250 MPa; and upsetting time 6 s. The friction joint formed under these conditions had the maximum joint tensile strength of 450 MPa, and fractured at the Ti original substrate (not heat affected zone). In order to grow interlayers, PWHT was carried out in a conventional vacuum furnace with infrared heaters. The vacuum environment was approximately  $0.1 \text{ Pa}$  ( $7.5 \times 10^{-4} \text{ torr}$ ). Heating temperatures were 773 K, 873 K, and 973K, and heating times were 21.6 ks, 43.2 ks, 86.4 ks, and 172.8 ks. The joints so formed are hereafter termed: '773 K-43.2 ks joint', for example, for the joint heated at 773 K for 21.6 ks; and/or '773 K-joint' for a joint heated at 773 K. Following PWHT, some joints were machined to 13.0 mm in diameter and 50 mm parallel length with the joint interface at the mid-position for joint tensile tests. All tensile tests were carried out at room temperature. For metallurgical tests, 15 mm diameter specimens were prepared to include the joint interface. SEM with EDS observations were carried out to delineate the interlayers and to analyse the elemental chemical distributions across the joint interface.

## Results and discussion

### As welded joint

Figure 1 shows the effect of friction time on the joint tensile strength welded with friction pressures of 100–200 MPa. The joint tensile strength varied from 408 MPa to 440 MPa, that is, from 101% to 109% of the tensile strength of Ti substrate and from 78% to 84% of the Ni substrate. The joint tensile strength rapidly increased with increasing friction time up to approximately 2 s, and gradually decreased for friction times exceeding 2 s. Friction pressure did not affect the joint tensile strength

as much as friction time. The maximum joint tensile strength of approximately 440 MPa was achieved at friction times of 1 and 2 s. Joint tensile strength at friction time 0.2 s was approximately 410 MPa, and was almost the same as that at 4 s. All tensile test specimens fractured at the interface regions; however, all joint tensile strengths were higher than the tensile strength of Ti substrate, that is, 402 MPa.

Figure 2 shows the relation between the distance from the axial centre and the interlayer thickness on the crosssection of the joints welded with friction times of 1, 2, and 3 s at a friction pressure of 150 MPa (SEM–EDS analysis). Irrespective of friction time, the thickness increased with increasing distance from the centre. The interlayer thickness at the centre region varied from nil (not detected) to approximately 0.5  $\mu$  m, it was approximately 1.6  $\mu$  m at peripheral positions under all conditions. This is due to the heat generation during friction welding, the heat generation at the periphery region being much larger than that at the centre region on account of rotational speed. The interlayer thickness increased slightly with increasing friction time. Maximum thickness was approximately 1.6  $\mu$  m in the peripheral region (6.5 mm from centre) at a friction time of 3 s. This is because of increased heat generation, i.e. long friction time and peripheral location (greater radius) during friction welding.

Only Ti was detected on the Ti fractured surface. A small amount of TiNi with Ni peaks was observed on Ni by X-ray diffraction analysis of the fractured surfaces. Figure 3 shows (a) Ni content and (b) Ti content on the Ti fractured surfaces of joints welded with friction pressures of 100–200 MPa and friction times of 2–4 s. While Ni content on the Ti surface increased to approximately 40 mol.% (at.%) from the centre region to the periphery (Fig. 3a), Ti content decreased to about 60 mol.% (Fig. 3b). The interlayer thickness was approximately 1.6  $\mu$  m, very thin, as described in Fig. 2, so that X-ray diffraction analysis scarcely detected these intermetallic compound phases on both Ni and Ti fractured surfaces. Notwithstanding these results, it can be estimated that some Ti–Ni intermetallic compound phases occurred at the joint interface. The results of Fig. 3a and 3b indicate that fracture propagated mainly between the interlayer consisting of Ni–Ti intermetallic compound phases and the Ti substrate in the central region. However, in the peripheral region, fracture propagated mainly through (within) the interlayer. Taking the data described above into consideration, it can be estimated that an interlayer of approximately 1–1.5  $\mu$  m thickness is necessary to join pure Ti to pure Ni by dissimilar friction welding.

The results suggest optimum joint forming conditions: friction speed 25 s<sup>-1</sup>; friction pressure 50 MPa; friction time 2 s; upsetting pressure 250 MPa; and upsetting time 6 s. The joint achieved a maximum joint tensile strength of 450 MPa, and it fractured in Ti original substrate, not the heat affected region. The joint efficiency was thus approximately 112% of that of the Ti substrate and 94.5% of that of the Ni

substrate.

#### Post-weld heat treated joint

Figure 4 shows the effect of heating temperature on joint tensile strength for heating times from 21.6 ks (6 h) to 172.8 ks (48 h). The specimen heated at 973 K for 172.8 ks autogenously fractured during the cooling stage of heat treatment. The joint tensile strength abruptly decreased with increasing heating temperature, changing from 393 MPa at 773 K to 74 MPa at 973 K for a heating time of 21.6 ks; from 387 MPa at 773 K to 58 MPa at 973 K for 43.2 ks; from 344 MPa at 773 K to 43 MPa at 973 K for 86.4 ks; from 324 MPa at 773 K to 84 MPa at 873 K for 172.8 ks. On the other hand, it slightly decreased with increasing heating time at each heating temperature.

Heat treated joints had two layers at 773 K, and four layers at 873 K and 973 K at the interfaces in comparison with the as welded joint; i.e. the number of layers increased with increasing heating temperature. Although the data are not given here, the direction of growth of the interlayers was determined to be from the Ni substrate side to the Ti substrate side. This arises because of the difference in the mutual diffusion coefficients of Ti atoms in Ni substrate and Ni atoms in Ti substrate. While the diffusion coefficients of Ni in  $\alpha$ -Ti at 973 K is approximately  $1\text{--}2 \times 10^{-13} \text{ m}^2 \text{ s}^{-1}$ ,<sup>9</sup> there are no data reported for diffusion coefficients of Ti in Ni at 973 K.<sup>10</sup> However, the diffusion coefficient of  $\alpha$ -Ti atoms in fcc-Ni is approximately  $3 \times 10^{-17} \text{ m}^2 \text{ s}^{-1}$ , i.e. 300 times larger than that of Ni atoms in a-Ti at 1073 K.<sup>4</sup> The interlayer adjacent to the Ti substrate is hereafter termed II, and then IV for the 773 K joint. The order is I, II, III, IV in the 873 K and 973 K joints. Based on further SEM-EDS investigations, the chemical compositions and estimated phases of each interlayer are summarised in Table 1. At low heating temperature (773 K), interlayers II and IV are estimated to be TiNi and TiNi<sub>3</sub>, respectively. Interlayers I, II, III and IV at elevated temperatures (873 K and 973 K) are estimated to be Ti<sub>2</sub>Ni, TiNi, [TiNi<sub>3</sub>+TiNi], and TiNi<sub>3</sub>, respectively. The Ti-Ni binary alloy phase diagram shows that the mode of intermetallic compounds changes from Ti<sub>2</sub>Ni to TiNi<sub>3</sub> via TiNi with increasing Ni content.<sup>11</sup> The crystal structure changes from hexagonal Ti<sub>2</sub>Ni, to cubic TiNi, monoclinic TiNi and hexagonal TiNi<sub>3</sub> [and Ti(Ti<sub>0.11</sub>Ni<sub>0.89</sub>)<sub>3</sub>] in order.<sup>7</sup> The interlayer III might be estimated as TiNi<sub>2</sub>. The possibility of eutectoid reaction between TiNi and [TiNi<sub>2</sub>+ TiNi<sub>3</sub>] at 823 K has been reported, however, TiNi<sub>2</sub> has not been positively detected and clarified in the Ti-Ni binary system up to now.<sup>6</sup> If TiNi<sub>2</sub> exists, it could be thought of as a metastable phase.

Figure 6 shows the results of X-ray diffraction analysis of the fractured surfaces of (a) the Ti substrate side and (b) the Ni substrate side of a joint heated at 973 K for 172.8 ks. Ti<sub>2</sub>Ni was detected at both fractured surfaces in the 973 K joints. Figure 7 shows the relationship between distance from centre axis region and Ti and Ni

content at (a) the Ti fractured surface, and (b) the Ni fractured surface of a 773 K–21.6 ks joint. Figure 8 shows the same results for a 973 K–172.8 ks joint. While the Ti and Ni contents of the fractured surfaces of 773 K joints were well scattered, contents of 973 K joints were approximately 60–80 mol.%Ti and 40–20 mol.%Ni. These results indicate that the fracture mainly occurred through the interface between TiNi and TiNi<sub>3</sub> for 773 K joints. However, it propagated in Ti<sub>2</sub>Ni and/or at the interface between TiNi and TiNi<sub>3</sub> layers for 973 K joints.

Figure 9 shows the relationship between heating temperature and each interlayer thickness at the interface of joints heated for 172.8 ks. Each interlayer increased with increasing heating temperature. In particular, each layer thickness rapidly increased when the heating temperature exceeded 873 K. The layer thickness order was II, IV, III, I. Figure 10 shows the relationship between interlayer thickness and joint tensile strength. The relationship between Larson–Miller's parameter (LMP) and the interlayer thickness is shown in Fig. 11. The parameter can be described by  $LMP = T(20 + \log t)$ ; where  $T$  is heating temperature (K) and  $t$  is heating time (h). Figure 12 shows the relationship between LMP and the joint tensile strength. The joint tensile strength decreased linearly with increasing interlayer thickness up to approximately 10  $\mu$  m (Fig. 10) and/or LMP up to about 19–20 $\times 10^3$  (Fig. 11). Tensile strength hardly changed when the interlayer width and/or LMP exceeded approximately 10  $\mu$  m and 19–20 $\times 10^3$ , respectively. On increasing heating temperature from 773 K to 873 K, the joint tensile strength rapidly reduced. The interlayer thickness increased rapidly when LMP exceeded about 19–20 $\times 10^3$  (Fig. 12). Generally speaking, increasing interlayer thickness reduced the tensile strength of these dissimilar joints. One of the authors had previously reported the existence of a critical thickness, i.e. joint tensile strength abruptly decreased for several combinations of dissimilar joints. It was a few micrometres for pure Ti and AISI 304L austenitic stainless steel,<sup>12</sup> and approximately 10  $\mu$  m for a pure Al/pure Ti joint.<sup>13</sup> According to Fig. 10, it can be concluded that the critical interlayer width for a pure Ti/pure Ni friction joint is several micrometres.

The results above show the interlayer growth increasing with increasing heating temperature. If the interlayer width  $w$  is calculated from the equation  $W = kx t^{1/2}$ ; where,  $k$  is constant and  $t$  is the heating time, Fig. 13 shows the relationship between heating temperature and  $k^2$  for various interlayer and total layer thicknesses. In descending order, the interlayer growth rate is TiNi<sub>3</sub>, TiNi, Ti<sub>2</sub>Ni, TiNi<sub>2</sub>. Under the supposition that  $k$  depends on the Arrhenius equation, Table 2 summarises the free energies of activation of layers consisting of intermetallic compound phases calculated from Fig. 13. The free energies of activation of Ti<sub>2</sub>Ni, TiNi, TiNi<sub>2</sub>, and TiNi<sub>3</sub> are 110, 138, 120, and 86 kJ/mol, respectively. Although the present authors have not found the free energies of activation, the value is relatively close to that of (Fe,Ni)Ti interlayer, that is, 124 kJ/mol.<sup>14</sup>

## Conclusions

The effect of welding conditions on the mechanical and metallurgical properties of as welded pure titanium (Ti) – pure nickel (Ni) friction welding joints has been investigated. The effect of interlayer thickness at the interface of pure Ti–pure Ni friction welded joints on the mechanical and metallurgical properties has been determined by forcibly applying post-weld heat treatments.

The following conclusions are drawn.

1. While friction pressure did not affect joint tensile strength significantly, friction time did. The maximum joint tensile strength of 450 MPa was achieved for a joint welded with friction pressure 150 MPa, friction time 2 s, and upsetting pressure 250 MPa. The joint fractured in the original Ti substrate (not the heat affected zone); i.e. the joint efficiency was approximately 112% of that of the Ti substrate and 94.5% of that of the Ni substrate.

2. The interlayer thickness increased with increasing friction time, and from the centre region to the periphery at a joint cross-section. The joint tensile strength was a maximum for interlayer thickness between 1 and 1.5  $\mu$  m, this range being essential to join pure Ti and pure Ni using dissimilar friction welding.

3. The joint tensile strength abruptly decreased on increasing heating temperature to 873 K and/or increasing the Larson-Miller parameter from  $16 \times 10^3$  to approximately  $19 - 20 \times 10^3$ . The joint tensile strength rapidly decreased with increasing interlayer thickness up to approximately 10  $\mu$  m, and remained roughly constant for any further increase in the parameter. The joint tensile strength fell from around 400 MPa to approximately 40–60 MPa as the interlayer thickness increased from zero to approximately 10  $\mu$  m.

4. The interlayer growth was from the Ni substrate side to the Ti substrate side. Two or four interlayers occurred at the interface of the joint, as summarised in Table 1. From the Ti substrate to the Ni substrate, TiNi and TiNi<sub>3</sub> interlayers (in that order) existed at the joint interface heated at 773 K. Ti<sub>2</sub>Ni, TiNi, [TiNi<sub>3</sub>+TiNi] and TiNi<sub>3</sub> interlayers (in that order) were detected in the joint interfaces heated at 873 K and 973 K. Joint fracture occurred mainly at the interface between TiNi and TiNi<sub>3</sub> for 773 K joints, however, it propagated in the Ti<sub>2</sub>Ni and/or at the interface between TiNi and TiNi<sub>3</sub> layers in the 973 K joints.

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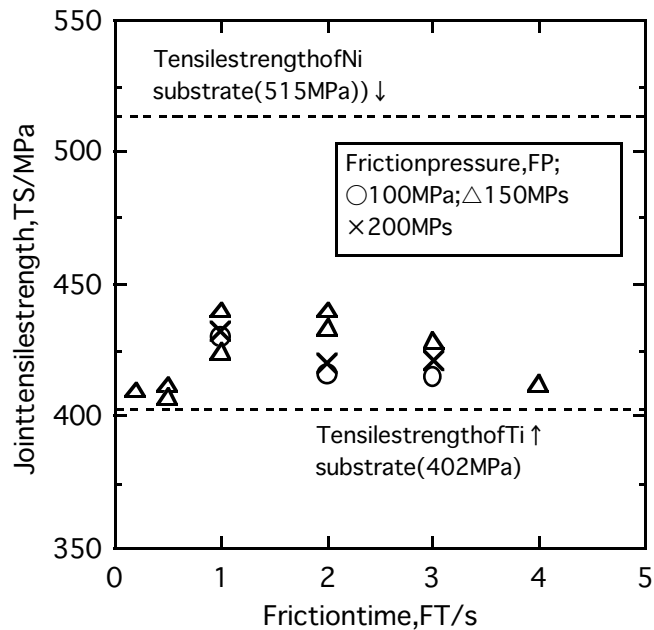


Fig.1 Effect of friction time on joint tensile strength for friction pressures of 100, 150 and 200 MPa.



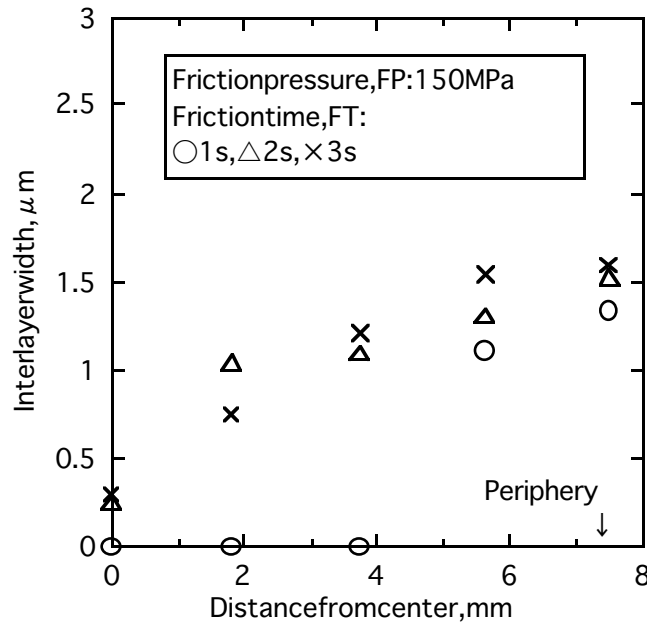
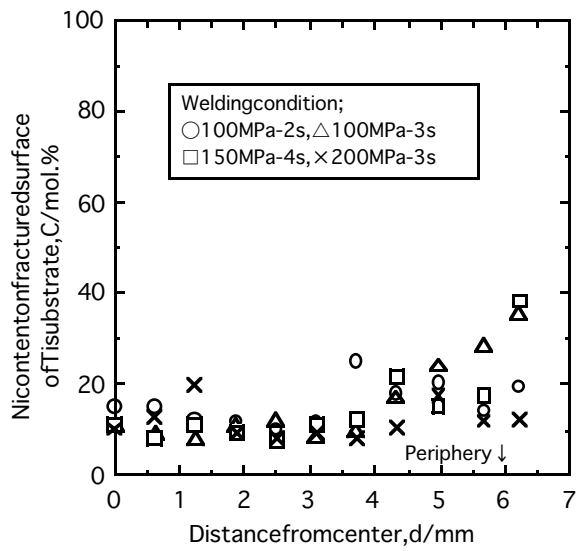
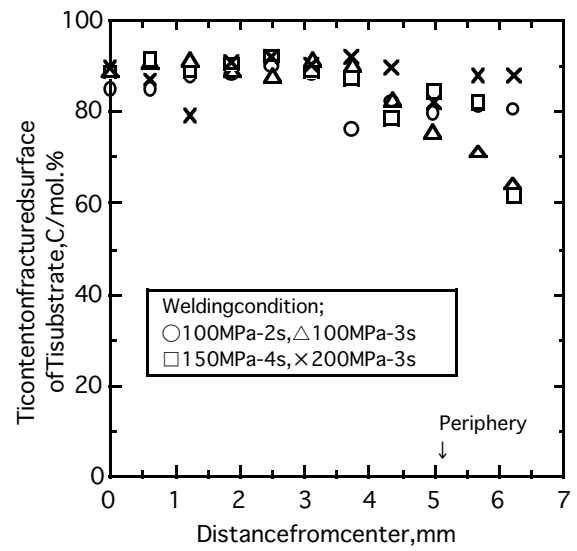


Fig.2 Relation between distance from axial centre and interlayer thickness in cross-sections of joints welded with friction times of 1, 2, and 3 s at friction pressure of 150 MPa (SEM-EDS analysis).



(a) Ni content on Ti fractured surface



(b) Ti content on Ti fractured surface

Fig.3 (a) Ni content on Ti fractured surface, and (b) Ti content on Ti surface of joints welded with friction pressures of 100–200 MPa and friction times of 2–4 s.

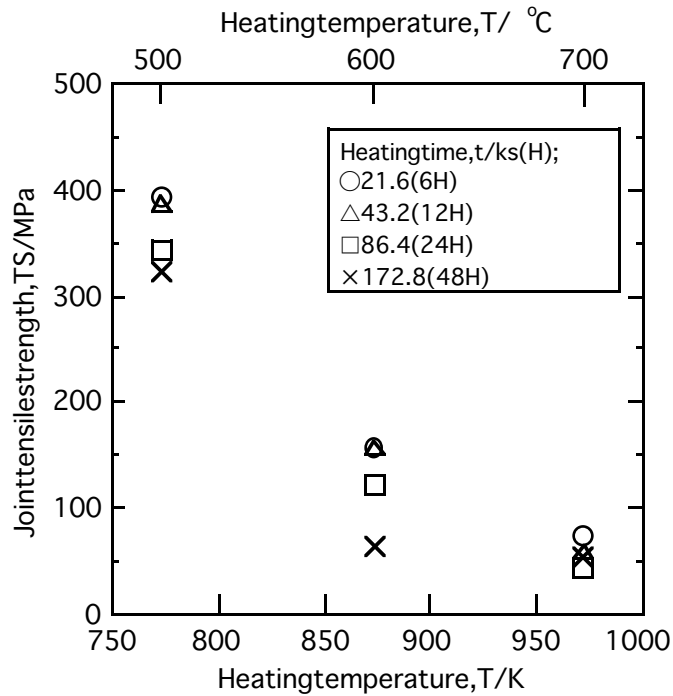


Fig.4 Effect of heating temperature on joint tensile strength for heating times of 21.6, 43.2, 86.4 and 172.8 ks.

Table 1 Chemical compositions and estimated phases of interlayers I, II, III, and IV for joints heated at 773–973 K for 21.6–172.8 ks.

Temperature	Layer	Chemical composition/mol.%		Estimated phases
		Ti	Ni	
773K (500°C)	II	50 - 55	50 - 45	TiNi
	IV	25 - 26	75 - 74	TiNi <sub>3</sub>
873K (600°C)	I	68.3 - 68.7	31.7 - 31.3	Ti <sub>2</sub> Ni
	II	49.4 - 50.5	50.6 - 49.5	TiNi
	III	33.4 - 37.5	66.6 - 62.5	[TiNi <sub>3</sub> + TiNi] or [TiNi <sub>2</sub> ]
	IV	24.3 - 25.7	75.7 - 74.3	TiNi <sub>3</sub>
973K (700°C)	I	64.1 - 67.1	35.9 - 32.9	Ti <sub>2</sub> Ni
	II	48.7 - 50.5	51.3 - 49.5	TiNi
	III	33.4 - 41.1	66.6 - 58.9	[TiNi <sub>3</sub> + TiNi] or [TiNi <sub>2</sub> ]
	IV	24 - 25	76 - 75	TiNi <sub>3</sub>

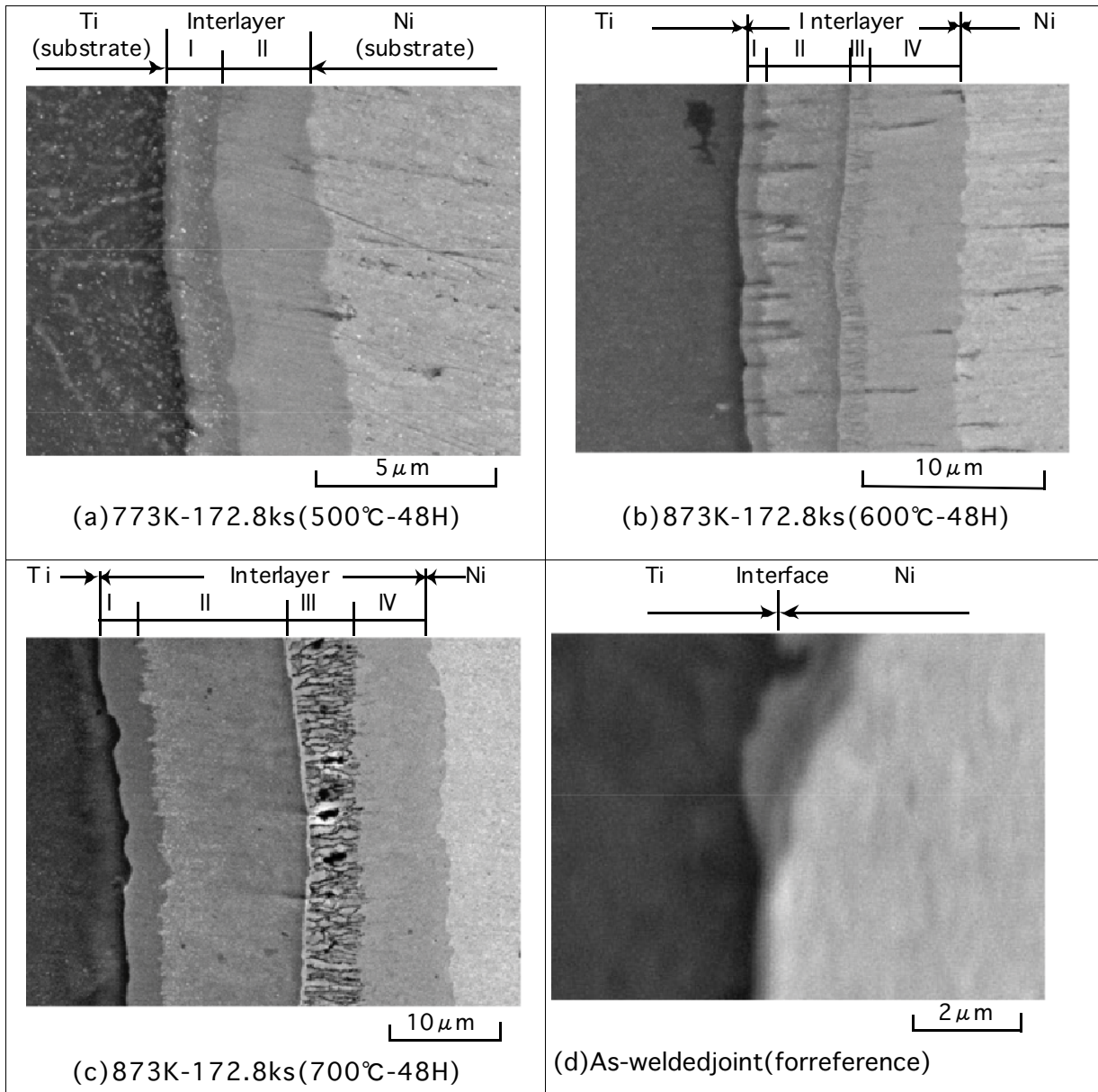


Fig.5 SEM micrographs of interfaces at half radius of joints: (a) 773 K, (b) 873K, and (c) 973 K for 172.8 ks joints; (d) as welded joint for reference.

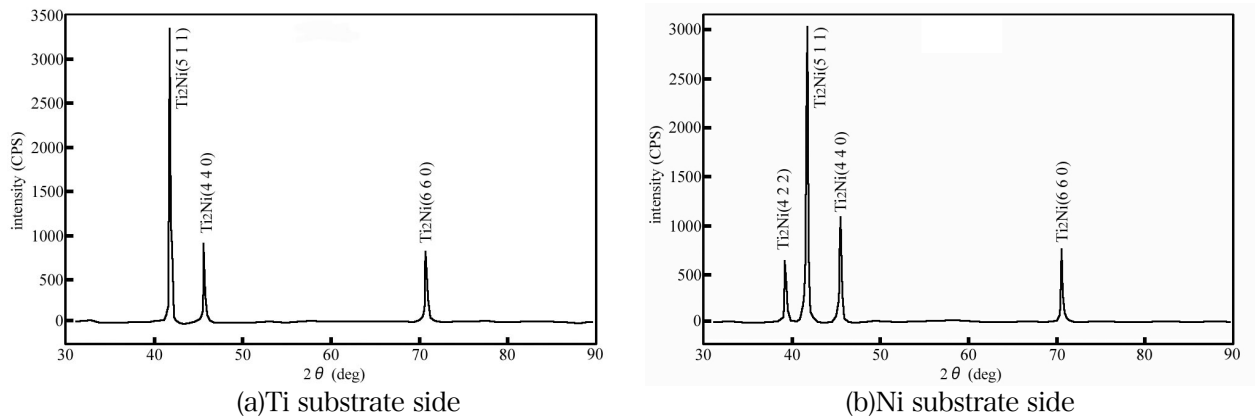


Fig.6 Result of X-ray diffraction analysis of fractured surfaces of (a) Ti substrate and (b) Ni substrate of 973 K-167.2 ks joint.

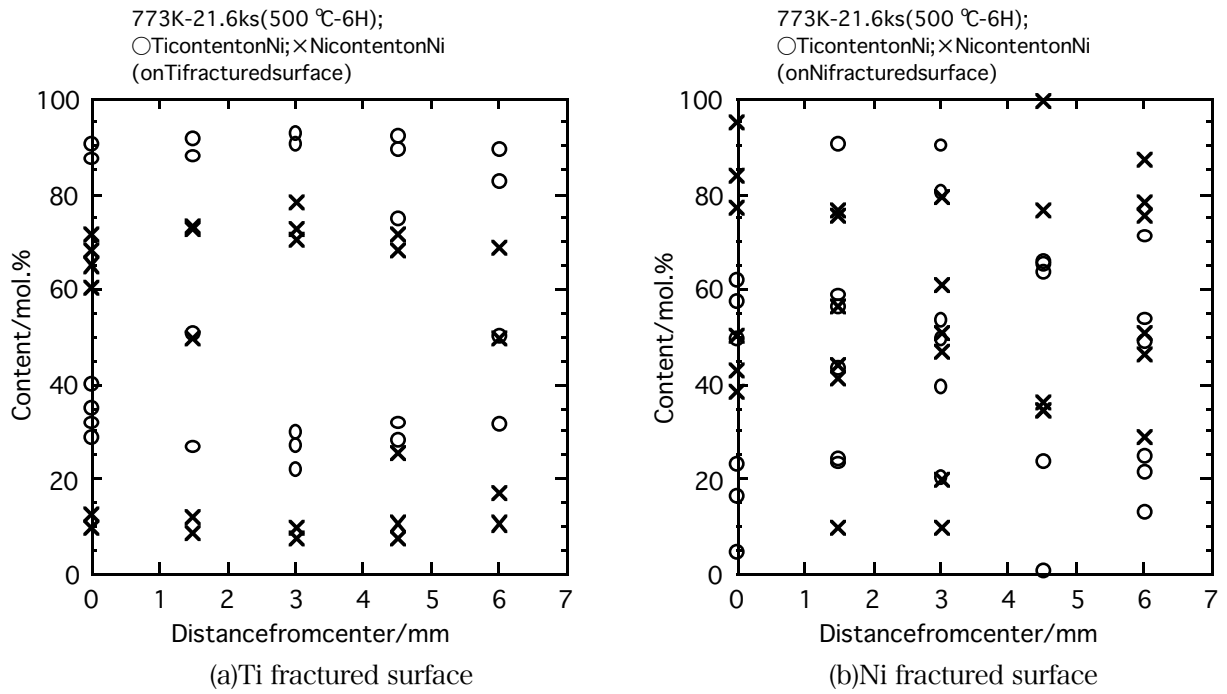


Fig.7 Relationship between distance from centre axis region and Ti and Ni contents at (a) Ti fractured surface, and (b) Ni fractured surface of 773 K–21.6 ks joint.

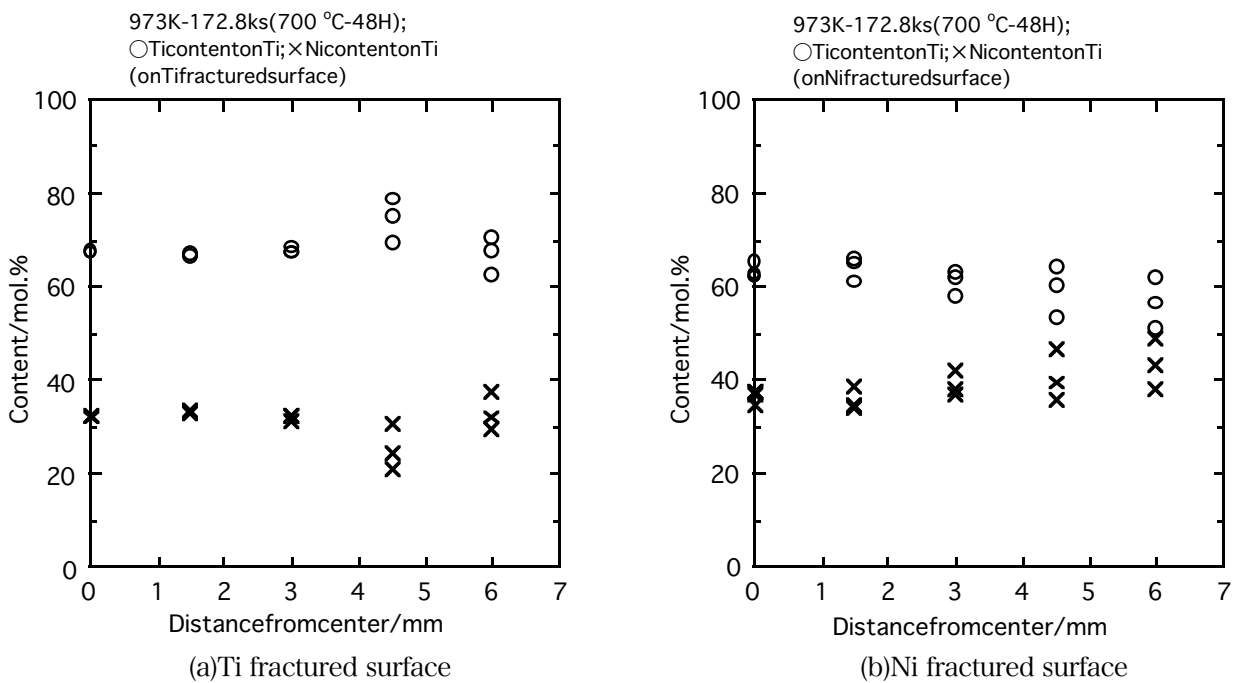


Fig.8 Relationship between distance from centre axis region and Ti and Ni contents at (a) Ti fractured surface, and (b) Ni fractured surface of 973 K–172.8 ks joint.

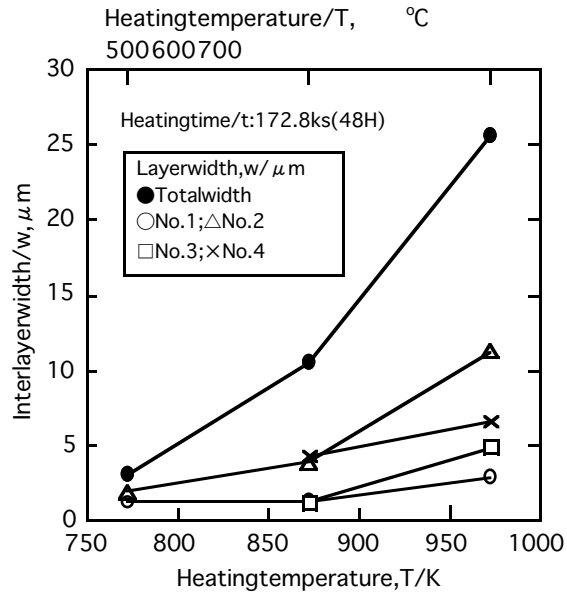


Fig.9 Relationship between heating temperature and interlayer thickness; heating time 172.8 ks.

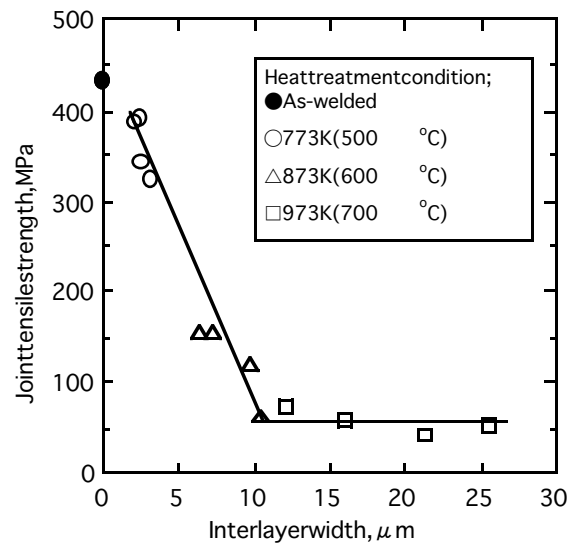


Fig.10 Relationship between interlayer thickness and joint tensile strength.

Table 2 Free energies of activation of interlayers of intermetallic compound phase.

No.	Layer	Activation energy/J/mol
I	Ti <sub>2</sub> Ni	1 0 9.5
II	TiNi	1 3 7.6
III	TiNi <sub>2</sub>	1 1 9.6
IV	TiNi <sub>3</sub>	8 6.0
—	Total	1 2 9.3

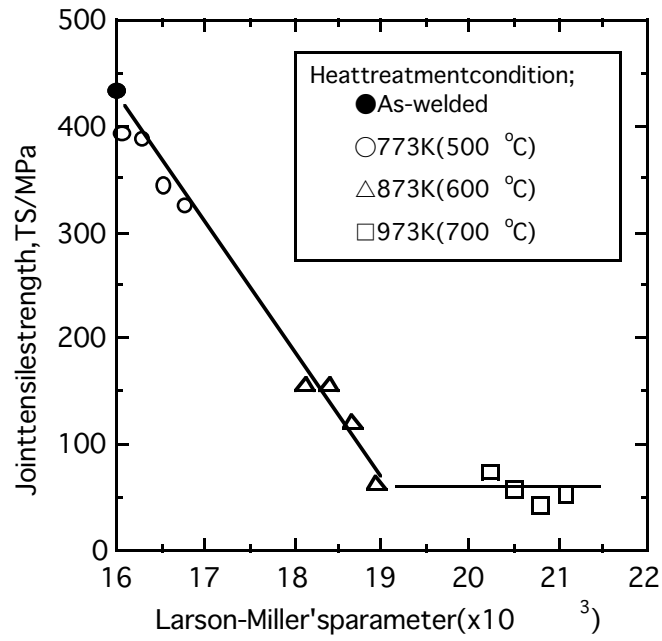


Fig.11 Relationship between Larson–Miller parameter and interlayer thickness.

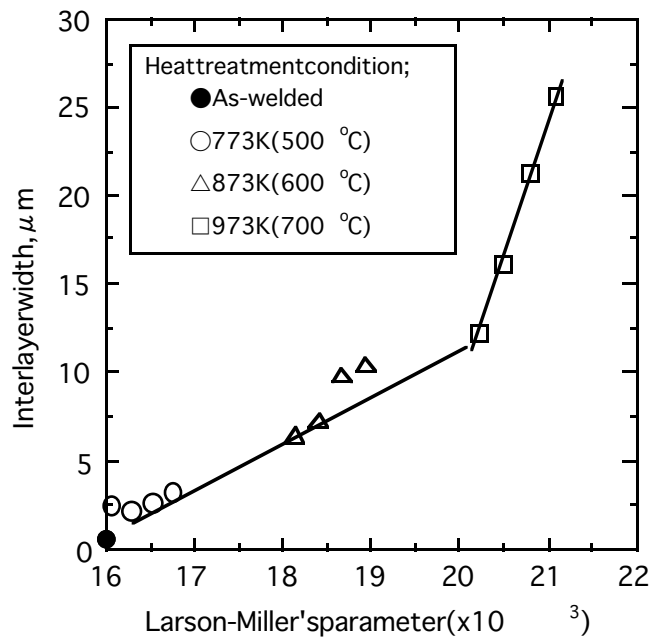


Fig.12 Relationship between Larson–Miller parameter and joint tensile strength.

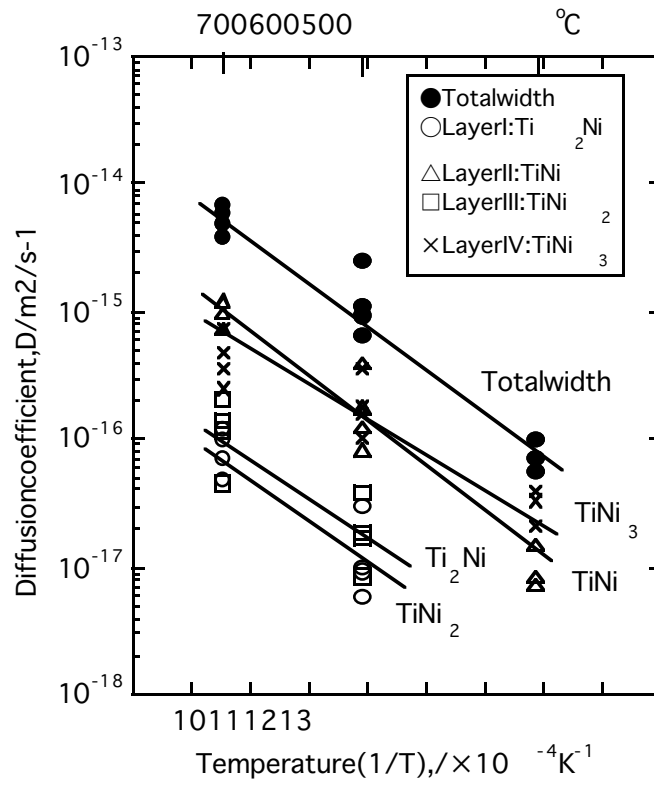


Fig.13 Relationship between heating temperature and  $k^2$  for interlayers of intermetallic phases.