Diffusion Welding of Ti-15%Mo-5%Zr Alloy to Mild Steel (0.06%C) †

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Abstract

Diffusion welding of Ti-15%Mo-5%Zr alloy to mild steel (0.06%C) has been performed in vacuum (~10^{-4} mmHg). Knoop hardness test, microscopic observation, X-ray diffraction analysis, electron prove X-ray microanalysis and scanning electron microscopic observation have been employed in order to clarify the existence of intermetallic compounds and carbides at the bonding interface. The effects of the existence of such compounds on the joint strength are discussed. Results obtained are summarized as follows:

(1) *Intermetallic compounds, Fe_2Ti and FeTi, carbides, TiC and Zr carbide, are found at the bonding interface without insertmetal, under the welding condition at the temperature of 900°C and 1000°C.*

(2) *On the joint containing nickel insertmetal, intermetallic compounds, Ni_3Ti, NiTi and $NiTi_2$, are found in the bonding interface between titanium alloy and nickel insertmetal, under the welding condition at the temperature of 700°C and 800°C.*

(3) *The strength of joints welded with nickel insertmetal is much larger than the joints welded without insertmetal. Maximum strength of joint with nickel insertmetal and without insertmetal was estimated to be 33 ~35 kg/mm^2 and 25 Kg/mm^2 respectively.*

1. Introduction

Diffusion welding is a possible method to obtain a good welded joint without any fusion in base materials. In principle, diffusion welding is quite different from usual welding methods because its process is controlled by thermally activated diffusion of atoms at bonding interface. The diffusion welding is not necessary for base materials to be melted at bonding zone, and so quantity of brittle compounds which are formed frequently in joint between dissimilar materials are small compared with usual welding methods and also welding residual strain is very small. Taking these advantages, diffusion welding has been applied to the weldings of many kinds of metals. Especially diffusion welding is used to obtain good joints between materials which are difficult or impossible by other welding methods. The welding of dissimilar metals is one of the most promising among them[1]. Generally speaking, the weldings of dissimilar metals are quite difficult to obtain good joints by usual fusion weldings, because of the formation of very brittle intermetallic compounds in bonding zone and/or the large difference in melting points of base metals. The reasons why the diffusion welding is favourable to the welding of dissimilar metals are as follows; the distribution of each element in bonding zone can be controlled rather easily than other welding methods and the melting of the base metals is not necessary in diffusion welding.

Diffusion welding between dissimilar metals has been studied in some investigations. But all of them have not resulted in obtaining good joints. Especially, for the joints of dissimilar metals, such as Ti-Fe[1),2),3)], Al-Fe[1),4)] and Al-Cu[1)], which form brittle intermetallic compounds, the obtained bonding strengths have been much lower than the base metals. However, there are only few studies to make clear what kinds of intermetallic compounds are formed in bonding zone and how these intermetallic compounds affect the bonding strength.

It has been reported that the bonding strengths were improved by the use of certain insertmetals[1),2),4)]. But the reasons do not always seem to be clear why the insertmetal strengthens the joint or the insertmetals should be selected.

In this investigation, diffusion welding of Ti-15%Mo-5%Zr alloy to mild steel (0.06%C) has been performed as an example where brittle intermetallic compounds are formed in the bonding zone and the microstructures of the bonding zone have been observed in detail. The relation between the microstructure and the bonding strength has been discussed. As the result of this discussion, nickel foil has been selected as insertmetal. The effects of the insertmetal on the microstructure and the bonding strength have been discussed comparing with the joints without insertmetal.

† Received on Jan. 12, 1976

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2. Experimental details

2.1 specimen

The materials used in this investigation are Ti-15%Mo-5%Zr alloy and mild steel (0.06%C). The chemical compositions are shown in Table 1. The titamium alloy is

Table 1. Chemical compositions.

Specimen	Chemical composition (wt%)					
	C	Si	Mn	P	S	Al
Steel	0.06	0.25	1.20	0.007	0.007	0.002

Specimen	Chemical composition (wt%)					
	Fe	N	O	H	Mo	Zr
Ti-Alloy	0.038	0.0037	0.135	0.0025	14.81	5.09

a metastable beta alloy whose crystal structure is B.C.C.. This titanium alloy is a superior material for the use of chemical industrial field because of its properties as corrosion-resistivity, spring characteristic and strength. The specimen is a cylindrical shape and its diameter and length are about 14mm and 30mm respectively. The end planes of the cylinder were turned to satisfy the flatness (as ▽▽▽ surfaces) and the orthogonarity to the axis of the cylinder. The specimens were welded with the end surfaces as faying surfaces. Nickel foil used as insertmetal is 10μm in thickness.

2.2 Welding procedure

In this experiment, following welding procedure was adopted. The faying surfaces of the specimens were degreased by washing in acetone and then brushed with a stainless steel wire brush before welding. The specimens were set between the pressing anvils in the welding chamber and the welding chamber was evacuated to $1 \sim 2 \times 10^{-4}$ mmHg. Then the bonding zone was heated with high frequency induction heater. The bonding zone was heated to a fixed temperature, and then the pressure of 0.5 kg/mm^2 was applied on the bonding interface. After the bonding zone had been holded in this experimental condition for a desired time, both of heating and pressing were taken off. The bonding zone was promptly cooled to 200°C by introducing argon gas into the welding chamber. The temperature of the bonding interface was monitored by a chromel-alumel thermocouple placed on the surface of the titanium alloy specimen close to the bonding interface. Welding temperatures were chosen from 800°C to 1000°C for welding without insertmetal and from 700°C to 1000°C for welding with nickel insertmetal. The specimens of titanium alloy were slightly deformed in the course of welding at the temperature of 900°C and 1000°C.

2.3 Testing procedure

The microstructure of the bonding zone was observed with optical microscopy with the magnification of ×100 to ×800. The surface of specimen was polished on buff followed by etching. The etchants used were mixed acid solutions (HNO_3 5% and ethylalcohol 95%) for mild steel and (HF 20%, HNO_3 20% and glycerine 60%) for the titanium alloy.

By using scanning electron microscopy (SEM), the secondary electron image from the microstructure in bonding zone, especially in 'd'region (see § 3.1), was observed with the magnifications of more than ×1000. And the distributions of Ti, Fe and Zr in the bonding zone were examined with energy dispersive X-ray spectroscopy attached to SEM. The accelation voltage and spot size of the electron beam was 20kV and about 100Å in diameter, respectively.

The distributions of Ti, Fe, Zr, Mo and C were examined with electron prove X-ray microanalyser (EPMA) in wider region than that examined with SEM. The electron beam spot has a size of about 2μm in diameter.

X-ray analysis was performed with diffractometer. The X-ray diffraction patterns obtained on the fractured surfaces were examined in order to identify the phases formed in the region where the fracture had occured. The intensity of diffraction line of X-ray is recognized to be affected by uneveness in the specimen surface[5]. The fractured surfaces obtained from the joint between the titanium alloy and mild steel were rather even and the effect of the uneveness on the intensity was considered to be rather small. The Cu-Kα radiation was always used in X-ray diffraction analysis.

Knoop hardness and tensile tests were also performed. The specimen for tensile test was prepared by machining after welding procedure and has the shape shematically shown in Figure 1. Instron type machine was used for tensile test and the deformation rate was 2 mm/min.

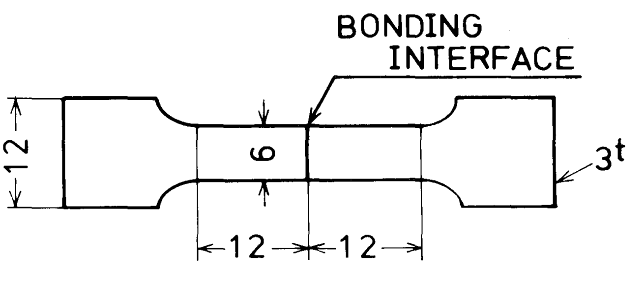


Fig. 1. Specimen for tension test.

3. Results and discussion

3.1 Welding without insertmetal

Photo. 1 shows the microstructures in bonding zones between Ti-15%Mo-5%Zr alloy and mild steel without insertmetal. Microstructures in the bonding zones shown in Photo. 1—i), ii), iii) and iv) are summerized as follows.

(1) The microstructure of mild steel in the joint welded for 30 min. at 800°C is almost the same as that of the base metal as shown in Photo. 1—iii).

(2) As shown in Photo. 1—i) and ii) which were observed in the joints welded for 1 hr. at 1000°C and 15 min. at 900°C, the microstructures of the joints could be divided into four characteristic regions. 'a', 'b' and 'c' regions were found in mild steel and 'd' region was in the bonding interface. The only one region, in which many precipitates appeared, was observed in titanium alloy.

(3) In 'a' region, pearlite structure still remained and grain size was smaller than that in 'b' and 'c' regions.

(4) In 'b' region, pearlite structure had been disappeared and remarkable grain growth had occured. These facts show that carbon atoms in this region have diffused to other regions.

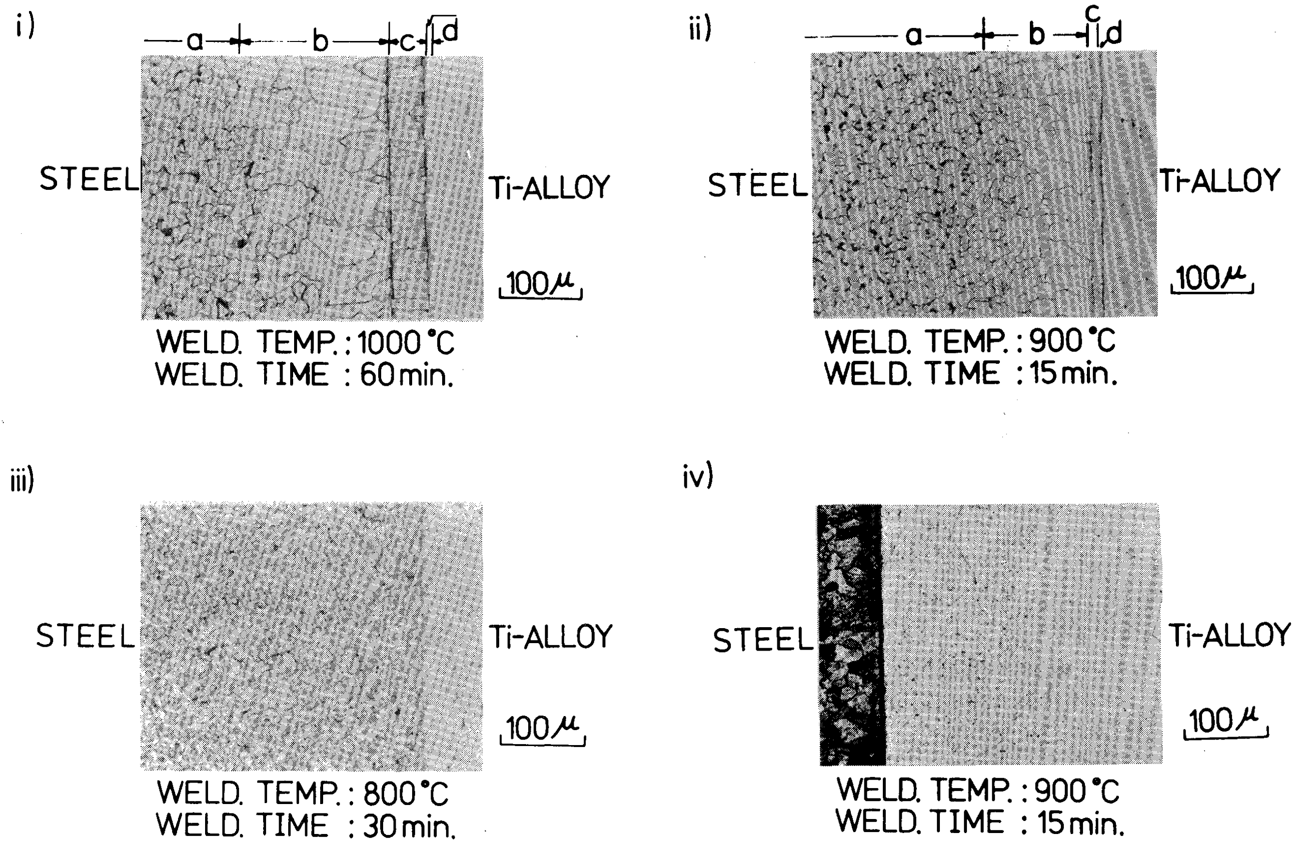


Photo. 1. Microstructures in bonding zones between mild steel and titanium alloy without insertmetal.

(5) In 'c' region, precipitated particles had appeared and grain size was larger than 'a' region but smaller than 'b' region.

(6) In 'd' region, some intermetallic compounds seemed to be formed.

(7) As shown in Phot. 1-iv), there was a relatively large region where many precipitated particles had appeared in the titanium alloy.

The widths of region 'a', 'b', 'c', 'd' and the region in the titanium alloy with many precipitated particles increased with increasing in welding temperature and/or time.

Figure 2 shows the distributions of Knoop hardness number in the joints welded for 1 hr. at 1000°C and for 30 min. at 800°C. As shown in Fig. 2, the hardness of the

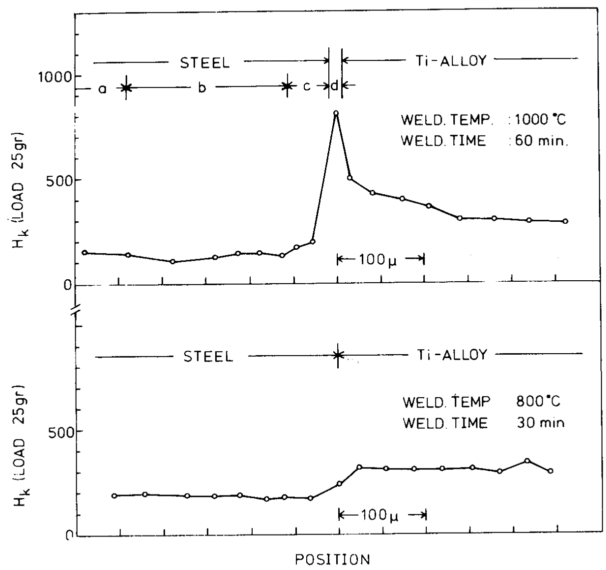


Fig. 2. Distributions of Knoop hardness number in bonding zones. The specimens were welded for 1 hr. at 1000°C and for 30 min. at 800°C without insertmetal.

mild steel and titanium alloy in the joint welded for 30 min. at 800°C underwent little change from those of the base metals. However, in the case of 1000°C and 1 hr. 'd' region had hardened remarkably and the hardening in the tinatnium alloy was also observed. In the case of 900°C and 15 min., the distribution of hardness number had the similar features to that in the case of 1000°C and 1 hr.

The analysis by EPMA was performed to investigate the distributions of Fe, Ti, Mo, Zr and C in bonding zone. Figure 3 shows the results from the analysis by EPMA in the bonding zone welded for 1 hr. at 1000°C. The results obtained from the analysis by EPMA are summerized as follows.

(1) Ti and Fe diffused considerably into the other base metals and the cnick points in each concentration distribution curve were found near the bonding interface.
(2) The concentration of Ti in mild steel decreased gradually from the bonding interface to the base metal and abrupt decrease was found at the region, 40 ~ 50 μm from the bonding interface.
(3) The distribution curve of Zr had a peak near the bonding interface. The gradual decrease in concentration was found near the bonding interface.
(4) The distribution curve of C had two peaks near the bonding interface.
(5) Mo diffused slightly into the mild steel.

The cnick points in the distribution curves of Ti and Fe suggest the formation of intermetallic compounds

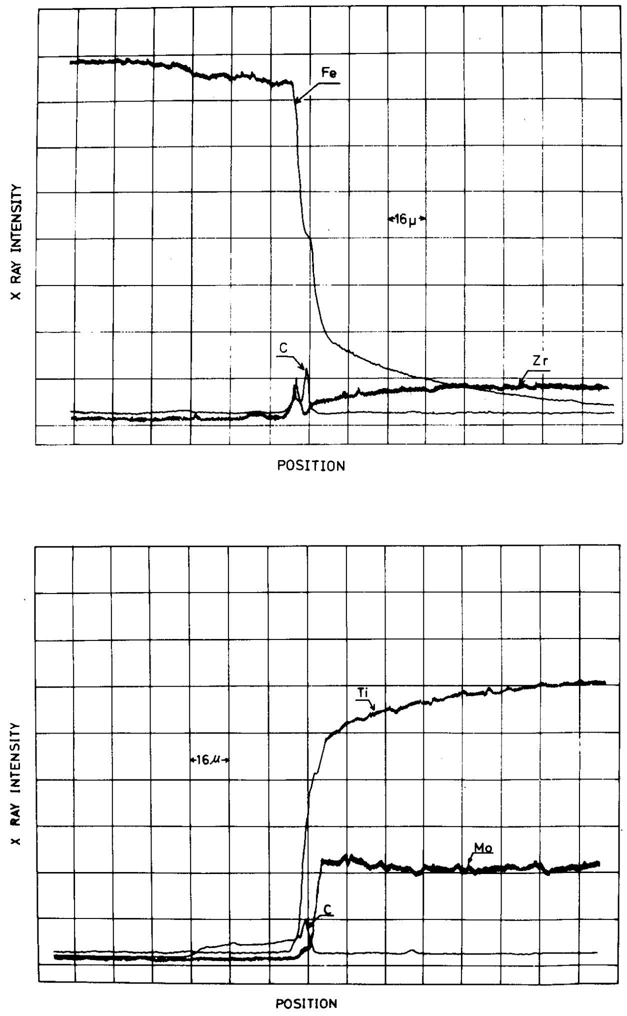


Fig. 3. EPMA analysis for the distribution of each element in bonding zone. The specimen was welded for 1 hr. at 1000°C without insertmetal.

constituted of Ti and Fe. The region, where Ti diffused into the mild steel, as described in above result (2) coincided with 'c' region in Photo. 1–i). One of the peaks of C and that of Zr in their distribution curves were superposed completely in their positions. This fact suggests the formation of Zr carbide near the bonding interface. But the distribution curve of Zr had no peak at the position in which the distribution of C had another peak. This suggests that carbide with any element except Zr was formed near the bonding interface.

Several intermetallic compounds and carbides are considered to be formed near the bonding interface. X-ray analysis was performed to identify these phases. Figure 4 showes the X-ray diffraction patterns obtained from the fractured surfaces of the joint welded for 30 min. at 1000°C. The fractured surface was obtained by the mechanical shock in the course of machining at the stage to prepare the tensile test specimen. Since the fracture of joints occured mainly along the 'd' region as shown in

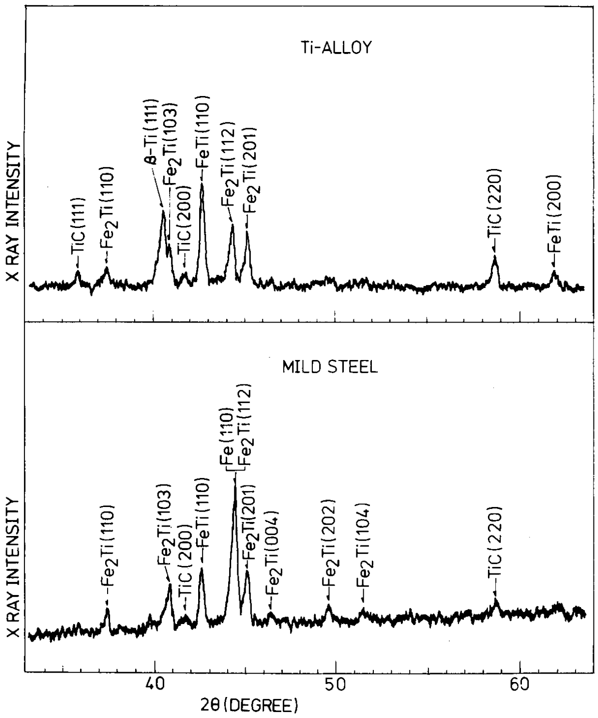


Fig. 4. X-ray diffraction patterns from fractured surfaces obtained from the joint. The specimen was welded for 30 min. at 1000°C without insertmetal.

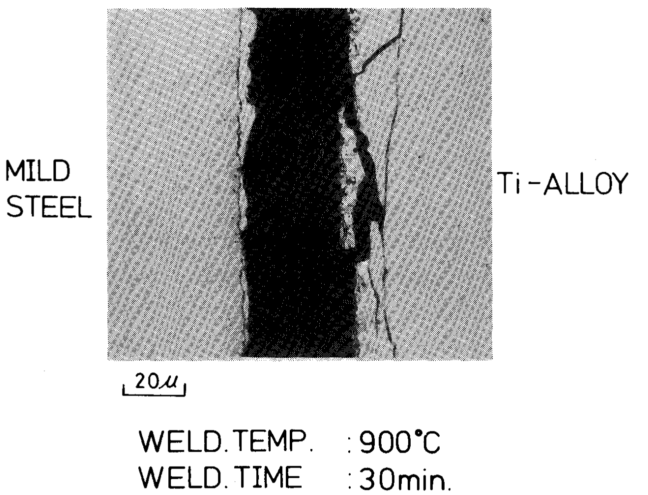


Photo. 2. Cross sectional microstructures for the fractured zone obtained from the joint welded without insertmetal.

Photo. 2, the diffraction patterns shown in Fig. 4 are considered to indicate mostly the phases formed near the 'd' region. As shown in Fig. 4, the diffraction lines of FeTi, Fe_2Ti and TiC were observed from the surfaces of both titanium alloy and mild steel. X-ray analysis was also performed on the fractured surfaces of the joint welded for 15 min. at 900°C. The tensile strength of this joint was estimated to be 25 kg/mm^2. In this case, the diffraction lines of FeTi, Fe_2Ti and TiC were also observed from the surfaces of both titanium alloy and mild steel. Since the joints were broken mainly along the 'd' region, the existence of these intermetallic compounds and carbides in both surfaces suggests that they existed in the 'd' region. The coexistence of intermetallic compounds and carbides is also suggested by the result of the analysis by EPMA in Fig. 3. That is, the cnick points in the distribution curves of Ti and Fe were found quite closely to the peaks in the distribution curves of C. The remarkable hardening of the 'd' region shown in Fig. 2 is considered to be due to the formations of these intermetallic compounds and carbides.

The 'd' region consists of several intermetallic compounds and carbides, such as FeTi, Fe_2Ti, TiC and Zr carbide. The observation of the 'd' region with SEM was performed in order to clarify how these intermetallic compounds and carbides were distributed in the 'd' region. Photo. 3–i) and ii) show the microstructures observed by SEM from the 'd' region welded for 1 hr. at 1000°C and for 15 min. at 900°C. The distributions of Ti, Fe and Zr analyzed with energy dispersive X-ray spectroscopy attached to SEM are also shown in Photo.3. The

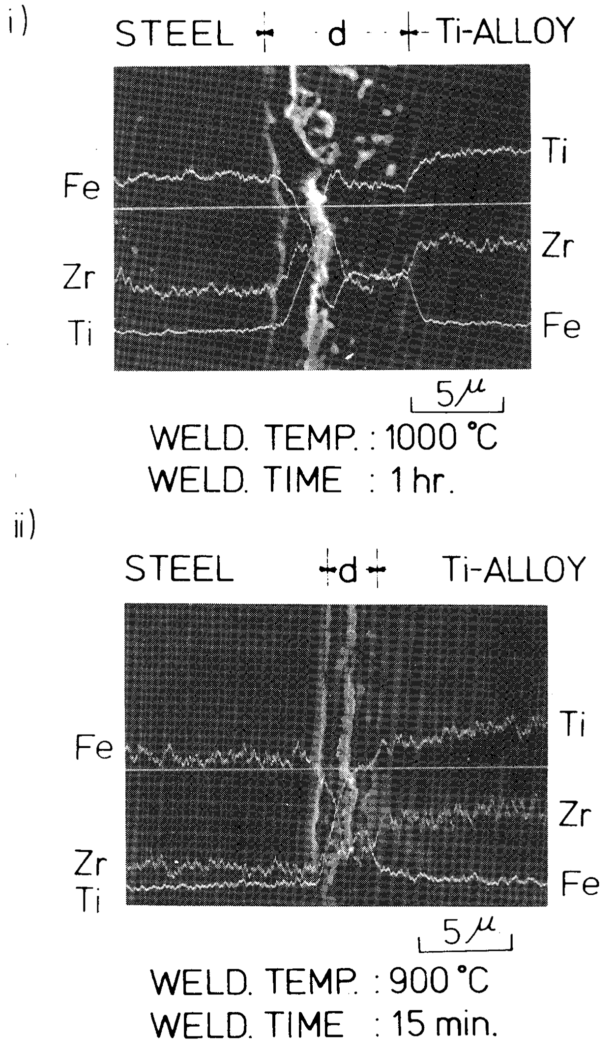


Photo. 3. Scannig electron micrographes obtained on 'd' region and X-ray intensities for each element.

analysis was performed along the white straight lines drawn in the photographs. The results obtained from the observation and analysis with SEM are summerized as follows.

(1) Plate-like compound and granular precipitates were found in the 'd' regions as shown in Photo. 3–i) and ii).
(2) At the plate-like compound, the content of Zr increased, but that of Fe decreased. According to an analysis along a line across granular precipitates, the X-ray intensities of Ti and Zr increased but that of Fe decreased at the granular precipitates.
(3) The contents of Ti and Fe were almost constant in the part of 'd' region between the plate-like compound and titanium alloy.
(4) On the other hand, the content of Ti and Fe decreased and increased respectively in the part of 'd' region between the plate-like compound and the mild steel.

The increase in content of Zr at the plate-like compound is considered to correspond to the peak in the distribution curve of Zr analyzed with EPMA (see Fig. 3). As described in the results of analysis by EPMA, the peak in the distribution curve of Zr was superposed with that of C and the formation of Zr carbide near bonding interface was suggested. Hence, the plate-like compound in the 'd' region is considered to be Zr carbide. It was also pointed out in the result of analysis by EPMA that carbide of any element except Zr was formed near bonding interface. According to the result of X-ray analysis, it was proved that TiC was formed in the 'd' region. From above discussion, the granular precipitates in which X-ray intensities of Ti and Zr were larger than those of other parts in the 'd' region may be considered to contain TiC phase. The region in which the contents of Ti and Fe were almost constant as described in result (3) is considered to consist of a intermetallic compound constituted of Ti and Fe. According to the X-ray analysis, FeTi and Fe_2Ti were formed in the 'd' region. Since the above results and the region was closer to the titanium alloy, the region may be considered to consist of FeTi which contains more Ti atom than Fe_2Ti. Perhaps Fe_2Ti may exist in the region closer to the mild steel where the content of Ti and Fe decreased and increased respectively as described in above result (4). TiC and ZrC are well recognized to be very brittle phases having $2000 \sim 3000$ kg/mm^2 in their hardness number. The 'd' region which contains such carbides in the intermetallic compounds is considered to be very brittle and markedly lower the mechanichal properties of the joint.

The tensile strength of the joint between the titanium alloy and mild steel was examined using Istron type machine. The joint strengths were very low and most joints fractured in the course of machining to prepare the specimens for tensile strength test. The maximum tensile strength obtained was estimated to be 25 kg/mm^2 when the joint was welded for 15 min. at 900°C. However the reproducibility of this maximum strength was not so sufficient.

In this investigation for diffusion welding between Ti-15%Mo-5%Zr alloy and mild steel (0.06%C) without insertmetal, the maximum tensile strength of the joint was much lower than the base metals and the welding condition to get proper maximum tensile strength was limited within a very narrow range. This is considered to be due to the formation of the 'd' region ($2 \sim 10\,\mu$m in width) where TiC and Zr carbide are contained in intermetallic compounds, FeTi and Fe_2Ti. The 'd' region in the joint having the tensile strength of 25kg/mm^2 had almost the same microstructure and phases as those in the joint which had fractured in the course of machining, as shown by the SEM observation and X-ray analysis. The difference between them was only in the width. The width of 'd' region in the joint with higher tensile strength was narrower than the other as shown in Photo. 3–i) and ii). This indicates that in order to obtain a good joint between Ti-15%Mo-5%Zr alloy and mild steel, the formations of intermetallic compounds and carbides should be suppressed as far as the atomic diffusion does not get insufficient.

3.2 Welding with Ni insertmetal

The attempt to obtain good joints by the use of insertmetal was made, since it proved difficult to obtain good joints between Ti-15%Mo-5%Zr alloy and mild steel by diffusion welding without insertmetal. Nickel foil having 10μm in thickness was selected as insertmetal. The reasons why the nickel foil was selected are as follows. The procedure for short time and at low temperature may be enough to weld the nickel foil to both titanium alloy and mild steel, since the velosity of nickel atom diffused into titanium and iron is considered to be faster than that of iron atoms into titanium and titanium atoms into iron.[6)] Therefore, the formation of the intermetallic compounds constituted of Ti and Fe and carbides of Ti and Zr may be suppressed by the use of nickel insertmetal. Besides, Ni and Fe form continuous series of solid solution at high temperature and Ni is difficult to form carbides.

Photo. 4 shows the microstructures of the joint with nickel insertmetal. As shown in Photo. 4, the microstructures of the titanium alloy and mild steel were almost the same as those of the base metals. However, at the interface between titanium alloy and nickel foil some interlayered region could be observed and deduces the

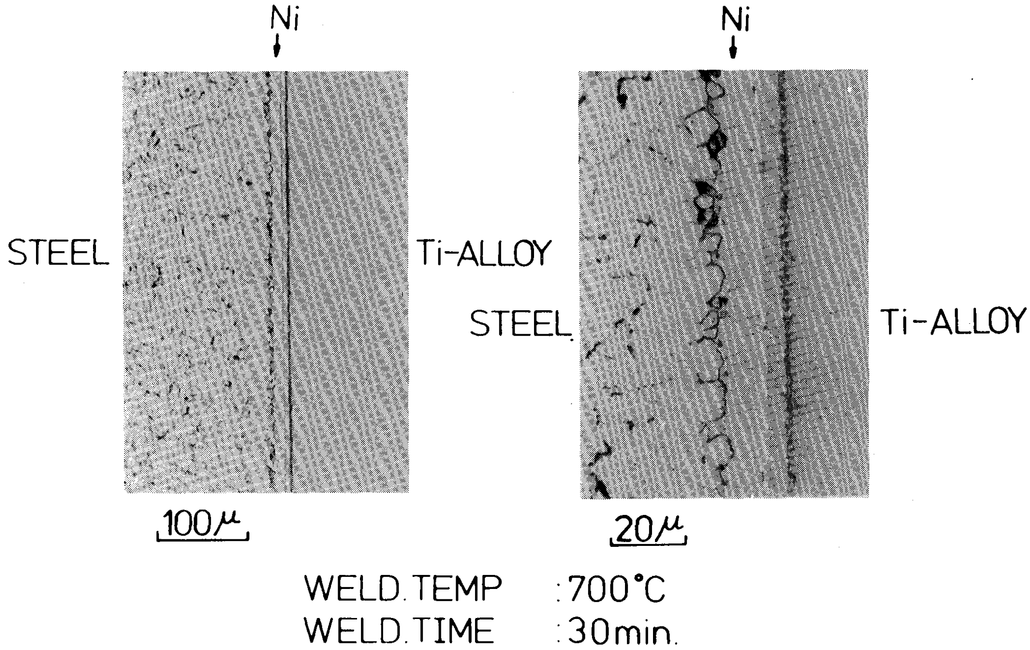
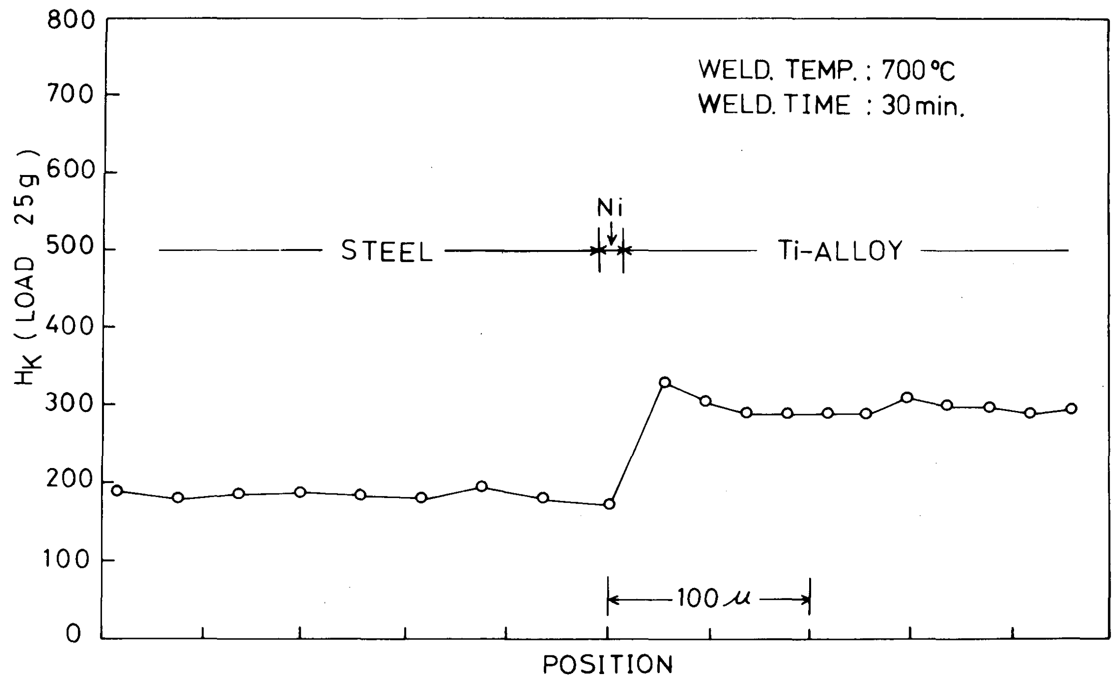


Photo. 4. Microstructure in bonding zone between mild steel and titanium alloy with nickel insertmetal.

formation of intermetallic compounds. When welding temperature and time was 800°C and 10 ~ 30 min. the microstructures were almost the same as shown in Photo. 4.

Figure 5 shows the distribution of Knoop hardness number in the bonding zone with nickel insertmetal. As shown in the figure, no harding was detected in the bonding zone.



Fir. 5. Distribution of Knoop hardness number in bonding zone. The specimen was welded for 30 min. at 700°C with nickel insertmetal.

The distributions of Ti, Fe, Mo, Zr, C and Ni in the bonding zone with nickel insertmetal were examined with EPMA. Figure 6 shows the result of analysis by EPMA in the bonding zone welded for 30 min. at 700°C. The results obtained from the analysis with EPMA are summerized as follows.

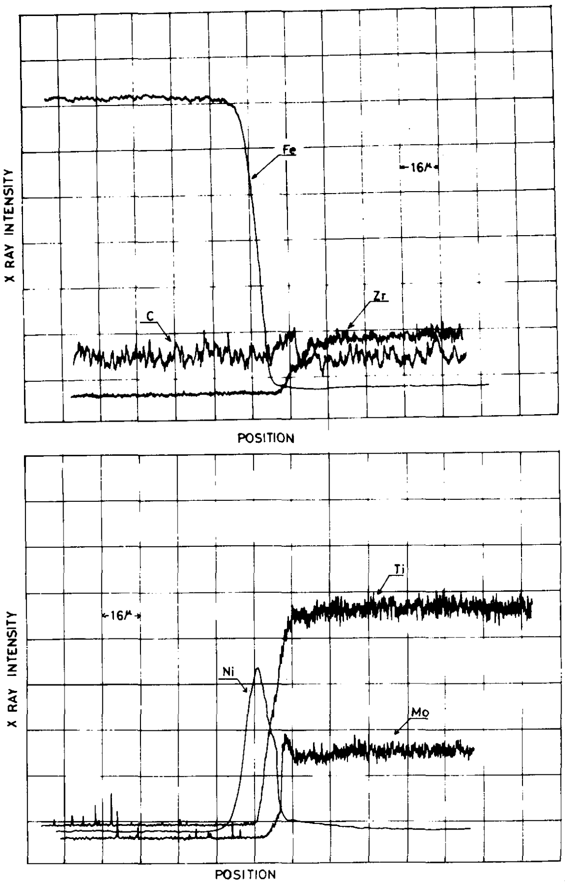


Fig. 6. EPMA analysis for the distribution of each element in bonding zone. The specimen was welded for 30 min. at 700°C with nickel insertmetal.

(1) No peak was found in the distribution curves of Zr and C, while the peaks were found in joints welded without insertmetal.
(2) Cnick point was not observed in the distribution curve of Fe.
(3) Cnick points in the distribution curves of Ti and Ni were observed in the interface between nickel insertmetal and titanium alloy.

The result descrived above in (1) indicates that the carbides of Ti and Zr were not formed in the bonding zone. The result described in (2) indicates that the intermetallic compounds constituted of Ti and Fe were not formed in the bonding zone. The cnick points in the distribution curves of Ti and Ni suggest the formations of intermetallic compounds constituted of Ti and Ni.

X-ray analysis was performed in order to identify the phases formed in the bonding zone welded with nickel insertmetal. Figure 7 shows the X-ray diffraction patterns

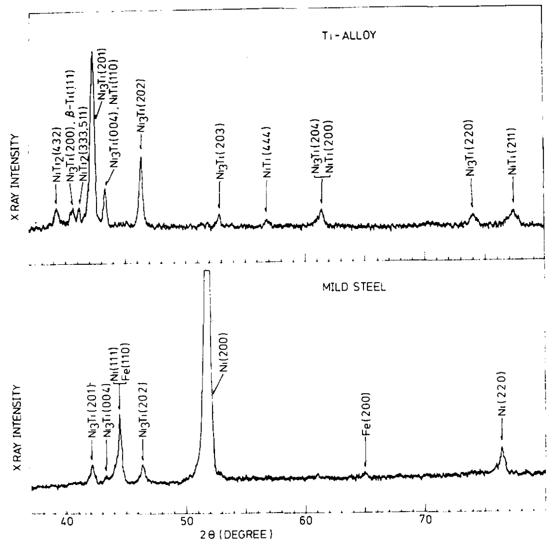


Fig. 7. X-ray diffraction patterns from fractured surfaces obtained from the joint. The specimen was welded for 30 min. at 800°C with nickel insertmetal.

obtained from the fractured surfaces of the joint welded for 30 min. at 800°C. The tensile strength of this joint was estimated to be 35 kg/mm^2. As shown in Photo. 5,

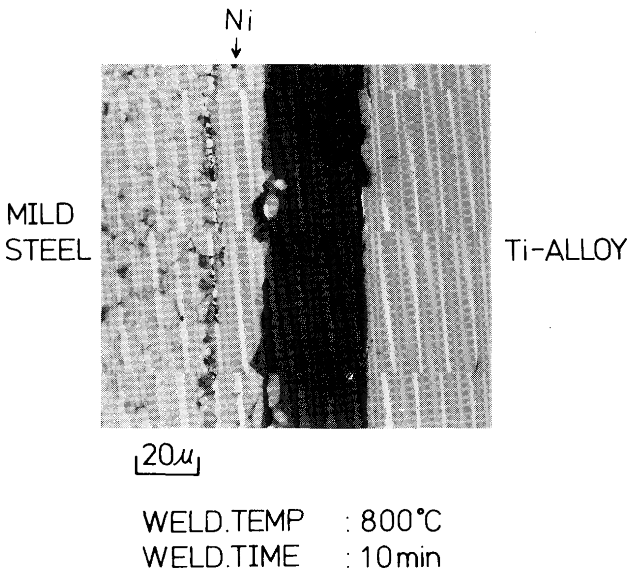


Photo. 5. Cross sectional microstructure for the fractured zone obtained from the joint welded with nickel insertmetal.

the fracture of joints occured mainly along the interface between titanium alloy and nickel insertmetal. Therefore, the diffraction patterns shown in Fig. 7 indicate mostly the crystal structures of the phases formed near the interface between titanium alloy and nickel insertmetal. As shown in Fig. 7, the diffraciton lines of intermetallic compounds, Ni_3Ti, NiTi and $NiTi_2$, were observed. Among these intermetallic ompounds, the diffraction lines of Ni_3Ti were found on the surfaces of both titanium alloy and mild steel. But the diffraction lines of NiTi and $NiTi_2$ were found only on the surface of the titanium alloy. This experimental result indicates that the joint welded with nickel insertmetal was broken down at the region where Ni_3Ti was formed.

To investigate the effects of nickel insertmetal on the strength of the joint, the test of tensile strength was performed. When welding temperature was 700°C and 800°C and welding time was 10 min. and 30 min. at the each temperature, the strengths of the joints were estimated to be 33~ 35 kg/mm^2 much higher than those of any joint without insertmetal. However, even in this case, any joint did not break down at the base metals but at the interface between titanium alloy and nickel insertmetal as shown in Photo. 5. When the welding conditions were for the longer time than 10 min. at above 900°C, the strength of the joint was found to be very low. In the diffusion welding of Ti-15%Mo-5%Zr alloy to mild steel (0.06%C), the joint strength was improved and the welding condition where the maximum strength was obtained was extended by the use of nickel insertmetal.

4. Summary

Diffusion welding of Ti-15%Mo-5%Zr alloy to mild steel (0.06%C) was performed and the microstructures in bonding zones were observed in detail with several metallographic methods. From these observations, the relation between the microstructures and the joint strengths was discussed. According to above discussions, nickel foil was selected as insertmetal and the effects of nickel insertmetal on the joint strength and the microstructure in bonding zone were investigated. Weldings were performed under the following conditions. The degree of vacuum was $1 \sim 5 \times 10^{-4}$ mmHg. The pressure applied on the faying surface was 0.5 kg/mm^2. Welding temperature and time was 800 ~ 1000°C and 15 min. ~ 1 hr. respectively when no insertmetal was used and 700 ~ 1000°C and 10 min. ~ 1 hr. when the nickel insertmetal was used. Results obtained are summerized as follows.

(1) Welding without insertmetal

(i) A remarkably hardened region ('d' region) which consisted of FeTi, Fe_2Ti, TiC and Zr carbide was found in the bonding interface under the welding condition at the temperatures of 900°C and 1000°C.

(ii) In the 'd' region, plate-like compound and granular precipitates which consisted of Zr carbide and TiC were found in the intermetallic ompounds of FeTi and Fe_2Ti.

(iii) The strength of the joint was low and only 25 kg/mm^2 at the maximum when welding temperature and time was 900°C and 15 min. This is due to the formation of brittle 'd' region in bonding interface.

(2) Welding with nickel insertmetal

(i) The intermetallic compounds constituted of Ti and Fe or carbides of Ti and Zr were not formed in the joint with nickel insertmetal welded for 10 ~ 30 min. at 700° ~ 800°C.

(ii) Intermetallic compounds, Ni_3Ti, NiTi and $NiTi_2$ were formed in the interface between the titanium alloy and nickel insertmetal under the welding condition for 10 ~ 30 min. at the temperature of 700°C and 800°C.

(iii) By the use of nickel insertmetal, the maximum joint strength was improved and the range of welding condition where maximum joint strength was obtained was extended. That is, the joint strength of 33 ~ 35 kg/mm^2 was obtained when welding temperature was 700°C and 800°C, and welding time was 10 min. and 30 min. at each temperature. But even in these cases, the joints were not broken at the base metals but at the region where Ni_3Ti was formed.

Acknowledgement

The authors would like to acknowledge the support of the Central Research Laboratory of Kobe Steel Ltd. in supplying the Ti-15%Mo-5%Zr alloy.

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