Proceedings of the 12th International Conference on
Aluminium Alloys, September 5-9, 2010, Yokohama, Japan

pp. 956-960

Nano-scale Evaluation of Interfacial Reaction Layers in Aluminum and Steel Dissimilar Metals Joints with Alloying Elements Using Nanoindentation Technique

Tomo Ogura[1], Keisuke Ueda[1], Yuichi Saito[1] and Akio Hirose[1]

[1] Division of Materials and Manufacturing Science, Osaka University,
2-1 Yamadaoka, Suita, Osaka 565-0871, Japan

Nanoindentation technique was successfully applied to the interfacial reaction layers with additional elements in dissimilar metal joints of 6000 system aluminum alloys to steel in order to characterize the interfacial reaction layers of joints. Nanoindentation hardness of the reaction layer formed at aluminum side was lower than that formed at SPCE side of the investigated joints. At aluminum side, nanoindentation hardness changed by the addition of alloying elements, and the hardness of Al-Fe-Si containing Cu intermetallic compound (IMC) was lower than that of Al_3Fe. It is characteristic that the hardness was monotonously decreased with increasing atomic ratio of iron from Al_5Fe_2 to $AlFe_3$ obtained from specimens with bulk type. By comparison of the hardness result of Al-Fe binary IMCs, hardness changes of interfacial reaction layers are thought to be caused by the crystal system changing from monoclinic to cubic. From the obtained results, it is suggested that mechanical properties of joints relate closely to the crystal structure of reaction layers.

Keywords: *Dissimilar metal joints of aluminum alloys and steels, Interfacial reaction, Nanoindentation, Diffusion bonding*

1. Introduction

Reduction of fuel consumption and curbing carbon dioxide (CO_2) emissions are key issues being tackled by automotive industries to resolve energy problems and global warming. To achieve weight reduction of automobiles at a low cost, hybrid car bodies made from Al alloys and steels are feasible structure, such as dissimilar metal joining of Al alloy and steel [1-5]. The dissimilar metal joining of aluminum alloys and steels has been considerably researched using several joining technique such as spot welding, laser welding, brazing and friction stir welding (FSW), however, it has been a common problem that the formation of a thick Al-Fe intermetallic compound (IMC) layer at the interface during bonding at high temperature causes low strength in aluminum/steel dissimilar metal joints. Therefore, microstructual control of interfacial reaction layers is essential to obtain high reliable dissimilar metal joints. Our previous work reported that the interfacial reaction layer was modified by the addition of Si and/or Cu to 6000 system aluminum alloy and this leads higher strength of the aluminum/low carbon steels (SPCE) dissimilar metal joint [6, 7]. This result suggests that the characteristics of the interfacial reaction layer would be changed by addition of alloying elements, however, the characteristics, especially, nanoscopic mechanical properties of the interfacial reaction layer are still unclear.

As for the method to examine nanoscopic mechanical properties of the interfacial reaction layer directly, nanoindentation techniques have been quite useful because nanoindentation becomes quite effective in investigating local deformation and hardness of alloys in a nanometer-scale [8]. Therefore, in this work, nanoindentation technique was applied to interfacial reaction layers with additional elements in dissimilar aluminum/steel joints in order to understand deeply the reason why higher strength was achieved by the addition of alloying elements to aluminum alloy. From the obtained results, nanoindentaion hardness changes were discussed based on crystal system of interfacial reaction layers.

2. Experimental Procedure

The chemical compositions of the aluminum alloys used in this work are listed in Table 1. For simplicity, the Al-0.6%Mg-0.6%Si alloy (in mass %) is designated as the base alloy, whereas the Al-0.6%Mg-1.5%Si, Al-0.6%Mg-0.6%Si-1.0%Cu and Al-0.6%Mg-1.5%Si-1.0%Cu alloys are designated as the Si-added, Cu-added and (Si+Cu)-added alloys, respectively. A low carbon steel including 0.01C, 0.15Mn and 0.01Si (in mass%) called as SPCE is prepared. After polishing the surface of the samples using emery paper (#2000), the surface was cleaned by acetone. Diffusion bonding was carried out in a vacuum of $<1.0\times10^{-1}$ Pa at heating temperatures (T) ranging from 773 to 848K for a holding time (t) from 150 to 1800s with a pressure of 2.5MPa. The heating rate was 3K/s.

After bonding, the interfacial microstructure was observed with scanning electron microscopy (SEM). The composition analysis was also performed using electron probe microanalysis (EPMA). The thickness of the reaction layer at the bonded interface was measured using SEM images. Tensile test was carries out with an Instron type tensile testing machine. The cross-head speed is 3.0×10^{-2}mm/s. Nanoindentaion measurement was performed toward the interface of joints at 2μm intervals using the instrument of ENT-1100a. The applied load was 50mgf and the load and release times were 10 and 1s, respectively. The error represents one standard deviation.

Table 1 Chemical composition of aluminum alloys used in this work.

material	Chemical composition (mass%)								
	Mg	Si	Fe	Cu	Cr	Mn	Zn	Ti	Al
Base	0.6	0.6	0.18	-	-	0.07	-	0.02	Bal.
Si-added	0.6	1.5	0.18	-	-	0.07	-	0.02	Bal.
Cu-added	0.6	0.6	-	1.0	-	-	-	-	Bal.
(Si+Cu)-added	0.6	1.5	-	1.0	-	-	-	-	Bal.

3. Results

3.1 Nanoindentation measurement at the interface of an Al alloy/SPCE dissimilar joint

Fig.1(a) shows a typical SEM image at the interface of the (Si+Cu)-added alloy/steel joint after nanoindentation measurement. Although no indent was seen at the interfacial reaction layer caused by a very low indent depth (< 50nm), it is clearly seen that some of the indent were successfully located within the reaction layer since indents were located at 2μm intervals. Fig.1(b) and (c) show EPMA mapping results of Si and Cu, respectively. Si and Cu were enriched within the interfacial reaction layer, especially in the aluminum side. Our previous work shows the interfacial reaction layer was Al_5Fe_2 and Al-Fe-Si system containing Cu in SPCE side and aluminum side, respectively by EDX analysis [6, 7]. From this result, the nanoindentation hardness was estimated as a function of the distance from the interface of the joint. The result was shown in Fig.2. The hardness in the interfacial reaction layer of Si and Cu enrichment by EPMA was identified as Al-Fe-Si containing Cu, and other indented points in the interfacial reaction layer were identified as Al_5Fe_2. The hardness of the reaction layer was much higher than that of aluminum and SPCE matrix. Moreover, within the interfacial reaction layer, the hardness of Al-Fe-Si containing Cu was found to be lower than that of Al_5Fe_2.

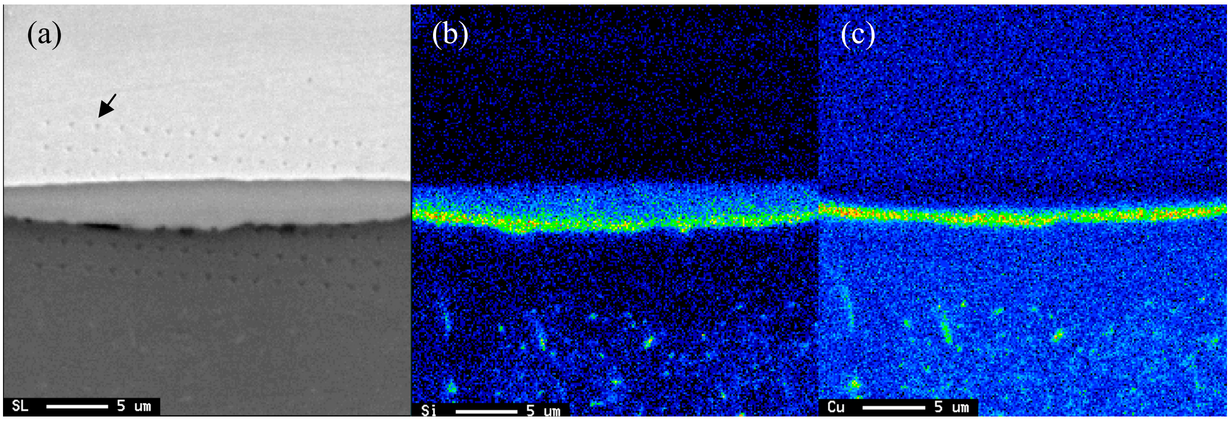

Fig.1 (a) A SEM image of nanoindentation marks around the interface and (b) and (c) corresponding EPMA mappings in (Si+Cu)-added alloy/SPCE joint bonded at 785K for 1.8ks. Nanoindentation marks were indicated by the arrow.

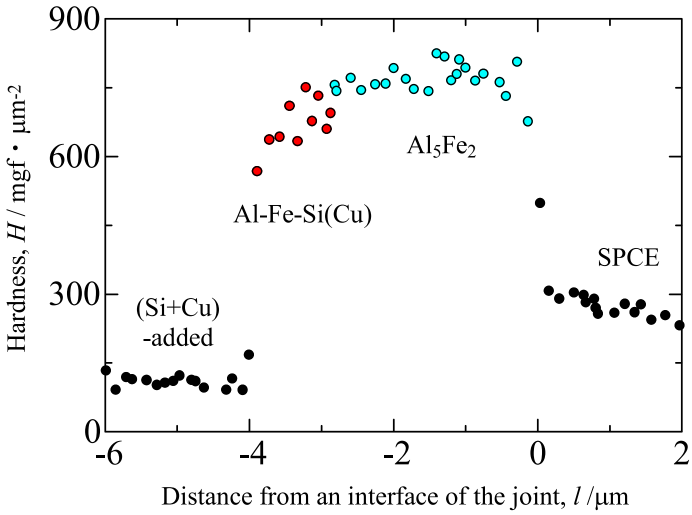

Fig.2 Distribution of hardness around the interface in Cu-added/SPCE joint bonded at 785K for 1.8ks. Corresponding IMCs and matrix are also shown.

3.2 Nanoindentation hardness changes of the reaction layers in Al alloys/Steel dissimilar joints

Nanoindentation hardness of the interfacial reaction layer was evaluated in all joints bonded at 785K for 1.8ks. Fig.3 shows nanoindentation hardness of the reaction layer at SPCE and aluminum side in aluminum alloy/SPCE joints. Corresponding IMCs were also shown, which identified were by EDX analysis [6,7]. At SPCE side, Al_5Fe_2 was formed and the hardness value of the interfacial reaction layer was almost the same regardless of the aluminum alloys. On the other hand, the hardness in the reaction layer at aluminum side was quite different in aluminum alloys. The hardness in the reaction layer of Al_3Fe was lower than that of Al_5Fe_2. Moreover, it was found that the hardness in the reaction layer of Al-Fe-Si was lower than that of Al_3Fe in the Si-added alloy. The effect was larger by incorporating 1% Cu to Al-Fe-Si.

4. Discussion

From the nanoindentation measurement of the reaction layers in aluminum alloys/SPCE joints, it was recognized that the hardness of the reaction layers were much higher than that of aluminum and SPCE matrix (Fig.2). Moreover, the hardness in the reaction layer of Al-Fe-Si was lower than that of Al_3Fe in the Si-added alloy and the effect was larger by incorporating 1% Cu to Al-Fe-Si (Fig.3). This hardness changes are considered to be closely related to atomic ratio of aluminum and iron in IMC. To understand the hardness changes precisely, in this work, nanoindentation measurement was also

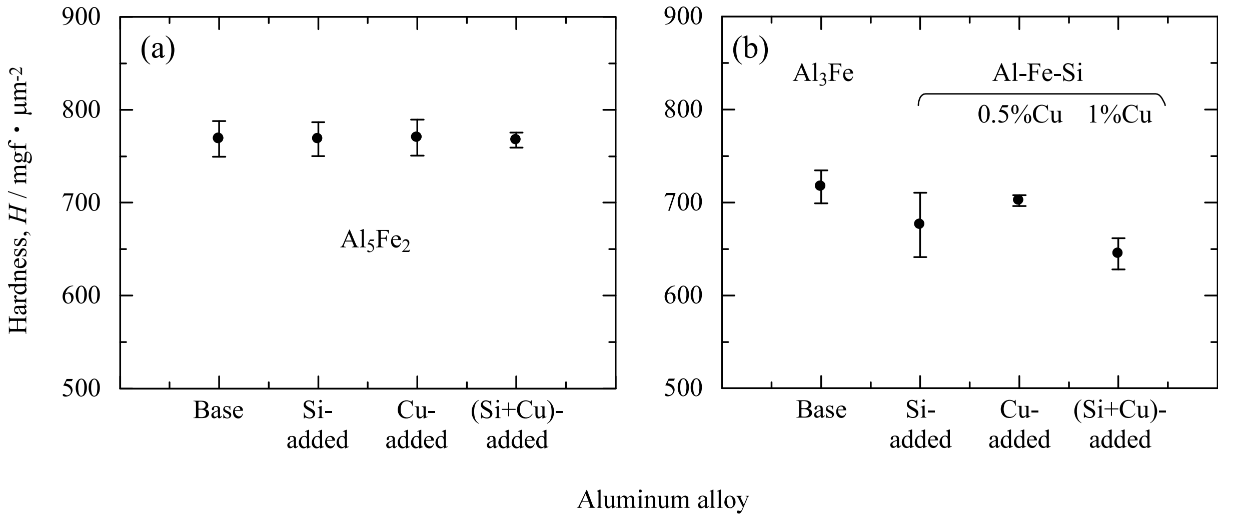

Fig.3 Nanoindentation hardness in the reaction layer at (a) SPCE side and (b) aluminum side in aluminum alloy/SPCE joints. Corresponding IMCs are also shown which were identified by EDX analysis [6,7].

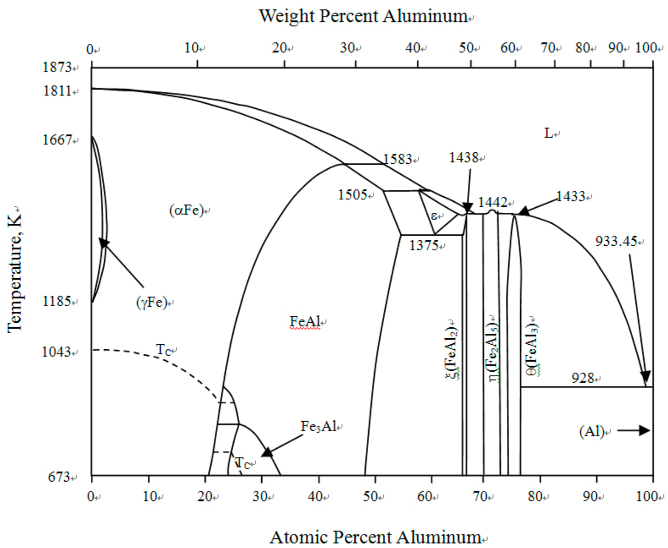

Fig.4 Al-Fe binary system phase diagram [9].

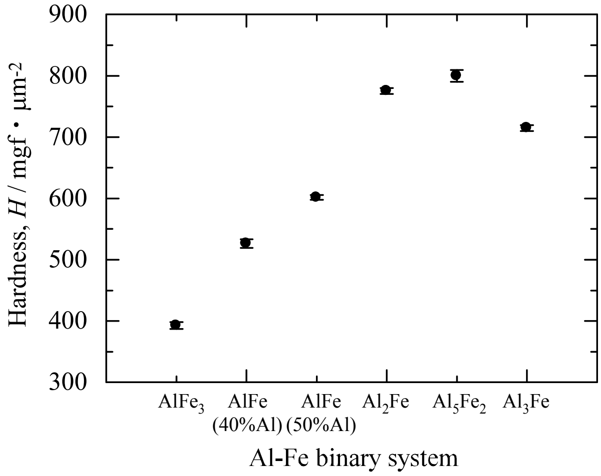

Fig.5 Nanoindentation hardness of Al-Fe binary system IMCs.

Table 2 Crystal structures of Al-Fe binary system [10].

Al-Fesystem	$AlFe_3$	AlFe	Al_2Fe	Al_5Fe_2	Al_3Fe
Cristal structure	cubic($D0_3$)	cubic(B2)	triclinic	orthorhombic	monoclinic

performed to Al-Fe binary system IMCs. Fig. 4 shows Al-Fe binary system phase diagram [9]. There are five types of Al-Fe IMCs ($AlFe_3$, AlFe, $AlFe_2$, Al_5Fe_2, and Al_3Fe) in phase diagram. Fig.5 shows nanoindentation hardness result of Al-Fe IMCs. The results Al-Fe IMCs were obtained not from the reaction layers but from other specimens with bulk type. The hardness of Al_5Fe_2 was the largest in Al-Fe IMCs. The hardness values of Al_5Fe_2 and Al_3Fe are well agreement with the hardness in the reaction layer in Fig.3. From Al_5Fe_2 to $AlFe_3$, it is characteristic that the hardness was monotonously decreased with increasing atomic ratio of iron. The crystal structures of Al-Fe system are shown in

Table 2 [10]. Although aluminum rich-IMCs (Al_2Fe, Al_5Fe_2 and Al_3Fe) have the small number of slip systems and brittle, equivalent or iron-rich IMCs (AlFe and $AlFe_3$) comparatively have the large number of slip systems and ductile.

The hardness decrease by the addition of alloying elements (Fig. 3) is similar to the result the decrease with increasing atomic ratio of iron. The crystal system of Al-Fe-Si in the interfacial reaction layer was identified as cubic by TEM observation [6]. The detail study of crystal structure of Al-Fe-Si and containing Cu IMC is needed, however, the crystal system of Al-Fe-Si containing Cu is thought to be cubic by comparison of the hardness result of Al-Fe IMCs. Therefore, the crystal system change of the interfacial reaction layer is considered to contribute the improvement of joint strength which was obtained in our previous work [6]. Thus, using nanoindentation technique, it is suggested that mechanical properties of joints relate closely to the crystal structure of reaction layers.

5. Conclusions

Nanoindentation technique was successfully applied to the interfacial reaction layers with additional elements in dissimilar metal joints of 6000 system aluminum alloys to steel in order to characterize the interfacial reaction layers. The results are summarized as follows.

1. Nanoindentation hardness of the reaction layer formed at aluminum side was lower than that formed at SPCE side. Moreover, the hardness of Al-Fe-Si containing Cu IMC was lower than that of Al_3Fe.
2. By comparison of the hardness result of Al-Fe binary IMCs, hardness changes of interfacial reaction layers are thought to be caused by the crystal system changing from monoclinic to cubic.
3. It is suggested that mechanical properties of joints relate closely to the crystal structure of reaction layers.

Acknowledgements

The authors would like to thank Prof. T. Nakano, Assoc. Prof. H. Y. Yasuda and Assist. Prof. T. Ishimoto for their significant help with experiments.This work was supported by Grant-in-Aid for Young Scientists (B) No. 21760585, Japan, and Priority Assistance for the Formation of Worldwide Renowned Centers of Research - The Global COE Program (Project: Center of Excellence for Advanced Structural and Functional Materials Design) from the Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan.

References

[1] H. Oikawa, T. Saito, T. Nagase and T. Kiriyama:Tetsu-to-Hagane, 83 (1997), 37-42.
[2] A. Hirose and K. F. Kobayashi:J. Japan Inst. of Light Metals, 56 (2006) 184-188.
[3] K. Mori: Journal of JSAE, 61 (2007), 13-17.
[4] A. Hirose: Journal of JSAE, 61 (2007), 18-23.
[5] S. Sasabe: Journal of JSAE, 61 (2007) , 24-29.
[6] T. Ogura , H. Umeshita , Y. Saito and A. Hirose: Q. J. JWS, 27 (2009), 174-178.
[7] Y. Saito, H. Umeshita, T. Ogura and A. Hirose: Q. J. JWS, 27(2009), 187-191.
[8] T. Ogura, S. Hirosawa and T. Sato, Sci. Tech. Adv. Mater., 5 (2004), 491-496.
[9] *Metals data book*. Ed. by The Japan Institute of Metals (Maruzen , Tokyo, 2004) pp.500.
[10] O.A.bannykh, K. Emani, S. Nagasaki and A. Nishiwaki: *Handbook of phase diagram of iron alloys* (Agune Engineering Center, Tokyo, 2007), pp.3-5.