Friction stir welding of Zr-based bulk metallic glass

Y S Ji¹, H Fujii¹, M Maeda¹, K Nakata¹, H Kimura², A Inoue², K Nogi¹
¹Joining and Welding Research Institute, Osaka University, 11-1 Mihogaoka, Ibraki, Osaka 567-0047, Japan
²Institute for Materials Research, Tohoku University, Sendai 980-8577, Japan

E-mail: fujii@jwri.osaka-u.ac.jp

Abstract. A Zr₅₅Cu₃₀Al₁₀Ni₅ bulk metallic glass plate was successfully welded below its crystallization temperature by friction stir welding. The flash formation and heat concentration at the shoulder edge was minimized using a wider tool and the angle of the recessed shoulder surface was 3°. To analyze the crystallization of the base material and stir zone, the microstructure and mechanical properties were analyzed using DSC, XRD, TEM, and microhardness. As a result, it was found that the amorphous structure and original mechanical properties were maintained in the whole joints.

1. Introduction
Bulk metallic glasses (BMGs) have been produced in various kinds of alloy systems, since the Au₇₅Si₂₅ alloy was the first amorphous alloy using a rapid solidification process in 1960. Bulk metallic glasses (BMGs) have excellent properties that include a high strength, good wear resistance, good magnetic properties, corrosion resistance, good forming ability, etc. [1-5]. These properties can be attributed to the atomic structure of the amorphous phase. Accordingly, BMGs are currently of interest as next generation materials and active research of BMGs are in progress. However, size limitation is a long-standing problem for structural applications because of a high cooling rate (1~100 K/s) requirement for the production of BMGs. To solve this problem, several recent studies on the joining of BMGs have been done [6-12]. In this study, the Zr₅₅Cu₃₀Al₁₀Ni₅ bulk metallic glass was joined using friction stir welding. Mechanical properties and the microstructure of welded joints were then investigated.

2. Experimental
A Zr₅₅Cu₃₀Al₁₀Ni₅ bulk metallic glass master alloy ingot was made by arc melting using high purity Zr (99.9%), Cu (99.9%), Ni (99.9%) and Al (99.9%). Fully amorphous specimens of 75×50×1 mm and 75×50×2 mm metallic glass square plates were prepared by copper mold casting under an Ar atmosphere. The stir-in-plate welding was performed using a position-controlled FSW machine. The tool, which was made of SKD61, had a 25 mm shoulder diameter, 5 mm probe diameter and a 1 mm or a 2 mm probe length with a 3° recessed shoulder surface. The probe was a screw-type and the tool was not tilted. The tool rotation speed was varied between 60 and 170 rpm to control the heat input while the travelling speed was constant at 100 mm/min.
Thermal properties of Zr₅₅Cu₃₀Ni₁₅Al₁₀ bulk metallic glass joints were measured by differential scanning calorimetry (DSC, Bruker, DSC 3300SA) at a heating rate of 0.33 K/s under flowing Ar gas. Microstructures of specimens were examined by X-ray diffraction (XRD) and transmission electron...
microscope (TEM). An XRD analyses were conducted (Bruker, AXS D8 DISCOVER) using Co-Kα radiation. TEM was done at 200 kV using a JEOL2030. Vickers micro-hardness (HV) values were measured using a (Akashi, AAV 501) digital micro-hardness tester with a testing load of 100 gf (=0.98 N).

3. Results and discussion

3.1 Tool shape
To minimize flash formation in this study a tool with a wide shoulder of 25 mm was used although a shoulder of approximately 12 mm is generally used.

On the other hand, the angle of the recessed shoulder surface of the tool is 3°. When the angle of the recessed shoulder surface is 10°, the heat is concentrated at the shoulder edge, causing the surface to become pitted at the shoulder. When the angle of the recessed shoulder surface is 0°, a lot of flash is formed. However, when the angle of the recessed shoulder surface is 3°, a comparatively good surface is obtained.

Figure 1 shows surface appearances of the stir-in-plate specimens of Zr55Cu30Al10Ni5 bulk metallic glass obtained at different rotation speeds. When the rotation speed is 60 rpm defects are formed on the surface, as shown in Figure 1(a), because the heat input is not sufficient. When the rotation speed is between 80 rpm and 170 rpm, large defects and cracks are not formed in the stir zone, as shown in Fig. 1(b), (c) and (d).

3.2 Microstructure and mechanical properties
Figure 2 shows XRD patterns of the stir zone of stir-in-plate specimens. When the rotation speed is between 80 and 150 rpm, XRD patterns of the stir zone shows typical broad patterns. This result confirms that the temperature of the stir zone during the stir-in-plate welding was below the crystallization temperature.

However, when the rotation speed is 170 rpm crystallization behavior is observed in the XRD spectrum of stir zone. In this case, the welding was performed in a short period. Accordingly, the temperature of the stir zone during the stir-in-plate welding was estimated to be above the crystallization temperature.

Figure 3 shows the hardness distribution on a cross section perpendicular to the welding direction of the stir-in-plate specimens. There is no significant change in hardness between the base material and the stir zone when the rotation speed is between 80 and 150 rpm, as shown in Figure 3. The average value of the hardness in the base material and stir zone is 523 HV.
Figure 4 shows DSC results of the base material and stir zone of the stir-in-plate specimens obtained at a 0.33 K/s heating rate. The glass transition temperature \( T_g \), the crystallization temperature \( T_x \) and the super-cooled liquid region range \( \Delta T = T_x - T_g \) are about 680, 750 and 70 K, respectively. As shown in Figure 4, the exothermic peaks are clearly observed without any shift in \( T_g \) and \( T_x \). These values are similar to that of the base material.

Figure 5 shows bright-field TEM images and corresponding selected-area-diffraction (SAD) patterns of the base material and the stir zone in the stir-in-plate specimens at 150 rpm. As shown in Figure 5 (a) and (b), no nanocrystalline particles were observed in the bright-field images of the base material or the stir zone. It is recognized from this TEM result that the base material and stir zone are homogeneous and amorphous at 150 rpm.

3.3 Effect of sample thickness
Figure 6 shows surface appearances and the possible range of the stir-in-plate of Zr_{55}Cu_{30}Al_{10}Ni_{5} bulk metallic glass specimens of 2 mm thickness. At a rotation speed of 80 rpm, defects are formed on the surface because the heat input is not sufficient. At rotation speeds between 100 rpm and 150 rpm, no large defects or cracks are formed in the stir zone as shown in Figure 6. However, the possible welding range for the 2 mm plates is narrower than that for the 1 mm plates because a higher heat input is necessary for thicker plates.
4. Conclusions

Zr\textsubscript{55}Cu\textsubscript{30}Al\textsubscript{10}Ni\textsubscript{5} bulk metallic glass was welded by friction stir welding. The obtained results are summarized as follows:

1. Zr\textsubscript{55}Cu\textsubscript{30}Al\textsubscript{10}Ni\textsubscript{5} bulk metallic glass was successfully welded below its crystallization temperature using friction stir welding.

2. Flash formation was minimized using a tool with a wider shoulder of 25 mm and heat concentration at the shoulder edge was prevented by the 3° angle of the recessed shoulder surface.

3. The microstructure and mechanical properties of welded joints were analyzed. It was found that the amorphous structure and original mechanical properties were maintained.

Acknowledgments

The authors wish to acknowledge the financial support of a Grant-in-Aid for the Cooperative Research Project of Nationwide Joint-Use Research Institutes on Development Base of Joining Technology for New Metallic Glasses and Inorganic Materials, "Priority Assistance of the Formation of Worldwide Renowned Centers of Research-The global COE Programs (Project: Center of Excellence for Advanced Structural and Functional Materials Design) from the Ministry of Education, Sports, Culture, Science and Grant-in-Aid for Science Research from the Japan Society for Promotion of Science and Technology of Japan.

References

[1] Inoue A 2000 Acta. Mater. 48 279
[2] Johnson W L 2002 JOM. 54 40
[3] Pang S J, T Zhang K, Asami and A Inoue 2002 Corrosion Sci. 44 1847
[4] Busch R, Bakke E and Johnson W L 1998 Acta. Mater. 46 4725
[5] Waniuk T A, Busch R, Masuhr A and Johnson W L 1998 Acta. Mater. 46 5229
[6] Wong C H, Shek C H 2003 Scripta. Mater. 49 393
[7] Kawamura Y, Shoji Ohno T and Ohno Y 2003 J. Non-Cryst Solid. 317 152
[8] Kawamura Y, Ohno Y, Chiba A 2002 Mater. Sci. Forum. 553 386.
[9] Kawamura Y, Ohno Y 2001 Scripta. Mater. 45 279
[10] Kawamura Y, Ohno Y 2001 Mater. Trans. JIM. 42 717
[11] Kawamura Y, Ohno Y 2001 Scripta. Mater. 45 127
[12] Kim J H, Kawamura Y 2008 Mater. Proce. Tech. 207 112