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A Novel Approach to Structure Modification of Brasses by Combination of Non-equilibrium Heat Treatment and Friction Stir Processing

AKBAR HEIDARZADEH, ALI CHABOK, VOLKER KLEMM, and YUTAO PEI

A non-equilibrium heat treatment was used to produce secondary phase (β) in the single phase brass (α), which caused the modification of the grain structure and mechanical properties after friction stir processing. For this aim, the single phase brass plate containing 37 wt pct Zn was heated at 810 °C for 1 hour, and then quenched in water, which caused the formation of non-equilibrium secondary β phase. After the non-equilibrium heat treatment, bead on plate friction stir processing was employed. The origin of the modification by β phase was studied using high resolution electron backscattered diffraction and transmission electron microscopy. The results showed that the average grain size, yield strength, and strain-hardening exponent were changed, respectively, from 4.8 μm , 166 MPa and 0.28 to 2.1 μm , 213 MPa and 0.25 in the presence of β phase. The β phase promoted the discontinuous dynamic recrystallization by particle-stimulated nucleation mechanism, and then transformed to α resulting in a finer microstructure with more random texture. In addition, the nanometer-sized β particles were retained at the grain boundaries, which reduced their mobility and hence the grain growth was inhibited.

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I. INTRODUCTION

FRICTION stir processing (FSP) has recently been developed based on the fundamentals of friction stir welding method for modifying the surface structure and properties of metals and alloys.^[1] During FSP, a rotating non-consumable tool inserts into the material and then traverses along the processing line.^[2] It can be employed in two different ways of processing with or without using reinforcements. In the first approach, ceramic particles are injected during FSP into the processed material.^[3] In the second way, no reinforcements are injected, and the properties of the processed materials change by only dynamic restoration mechanisms such as dynamic recrystallization (DRX).

Friction stir processing of Cu–Zn alloys (brasses) has attracted many attentions recently.^[4–8] This is because of the fact that FSW is a solid state process and most of the problems associated with the fusion processes of Cu–Zn alloy systems such as zinc evaporation, color change, gas porosity, shrinkage porosity, distortion, micro and macro segregations, dendritic structures, etc. can be overcome. A few methods have been suggested so far to modify the final microstructure and mechanical properties of the FSPed brasses, which can be divided into three categories. In the first category, low heat input conditions were used to refine the grain structure.^[9–11] For example, Sun *et al.*^[9] obtained a processing map for the friction stir welding (FSW) of commercial 2-mm-thick single phase Cu-30Zn brass alloy plates. They reported that the average grain size ranged from 6.4 to 9.6 μm depending on the different welding conditions, much smaller than the grain size (28.2 μm) of the base metal. However, using low heat input conditions for FSP of brasses causes more defects and reduced mechanical properties. It is notable that in addition to the grain refinement of the brasses, higher dislocation density with tangle structures, lower texture intensity, and larger values of Taylor factor have been reported as other reasons for improved mechanical properties after FSP/FSW.^[6,7]

The second category refers to the methods that use cooling media during or after FSW/FSP.^[12–14] For instance, Xu *et al.*^[14] have used liquid CO₂ as the cooling

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medium during the FSW of 70/30 brass plates. Their results showed that the rapid cooling could prohibit both recovery and static recrystallization, which resulted in enhanced yield strength with adequate ductility. The main difficulty of the second category is the need for the additional instruments for submerged condition, stop action technique, or flowing cooling liquids. In the third category, which has not yet been well studied for brasses, ceramic particles are added into the FSPed material, resulting in the formation of a composite structure.^[15] In this case, achieving a uniform distribution of the particles is the main challenge. The other critical disadvantages of the third category are the problems of grooving on the base material, controlling the volume fraction of the particles, abrasion of the tool by the particles, etc. Due to the mentioned issues, in this study, an alternative approach is originally introduced for the microstructure-properties modification of brasses, which may be either suitable for other alloys with a similar phase diagram.

II. MATERIALS AND METHODS

The base metal (BM) was single phase brass plate with 37 wt pct Zn (SP-63B). The double phase plates (DP-63B) were produced by non-equilibrium heat treatment of SP-63B such that the SP-63B plates were heated at 810 °C for 1 hour, and then quenched in water, which caused the formation of non-equilibrium secondary β phase. It is worth to note that at Zn content higher than 40 wt pct, the microstructure of brasses is double phase containing α (FCC) and disordered β (BCC) phases. However, during cooling of these alloys, at temperatures of 456 °C to 468 °C, disordered β transforms into ordered β' phase. For 40 wt pct > Zn content $\geq$ 37 wt pct, the alloy will be single phase at room temperature because β' cannot form from α . The SP-63B and DP-63B plates with the same dimensions of 150 mm $\times$ 100 mm $\times$ 2 mm were friction stir processed under the equal process condition. The rotational speed, traverse speed, and plunge depth were set constant at 400 rpm, 100 mm/min, and 1.9 mm, respectively. The FSP tool was made of H13 hot work tool steel, which was composed of a cylindrical shoulder with a diameter of 12 mm and a $\phi 3 \times 1.7$ mm cylindrical pin. To record the peak temperature during FSP, type K thermocouples were placed underneath the plates exactly at the center point of the process line.

Nanoindentation tests were performed using MTS XP Nano-indenter equipped with a Berkovich diamond indenter. Minimum 20 indentations were conducted for each samples at the maximum load of 40 mN. The indentations were taken from the stir zones of the samples. The reverse algorithm developed by Dao *et al.*^[16] was used to extract the local mechanical properties (yielding strength σ_y and strain-hardening exponent n) from the nanoindentation data. The microstructure of the FSPed samples was examined on the transvers cross-sections by using a Philips XL30 E-SEM field emission gun scanning electron microscope

equipped with electron backscattered diffraction (EBSD) system for orientation imaging microscopy (OIM). The OIM scans were made on the longitudinal cross sections along the processing direction of the FSPed samples. The specimens for OIM were finalized after mechanical polishing by electropolishing for 30 seconds in a solution containing 250 mL H_3PO_4 , 250 mL ethanol, 50 mL propanol, 500 mL distilled water, and 3 g urea at 10 V and 25 °C. The step size of 70 nm was used for all OIM scans. TEM (JEOL JEM 2010) and scanning transmission electron microscopy (STEM, Hitachi S-4800) were used for more clarification of the microstructural details. For TEM sample preparation, the electrojet thinning was used with a solution of 30 pct H_3PO_4 and 70 pct distilled water at applied potential of 80 V.

III. RESULTS AND DISCUSSION

Figure 1 shows nanoindentation load-displacement curves together with the extracted mechanical properties and average grain size of the SZ of friction stir processed SP-63B and DP-63B, respectively. The average grain size (D_{av}), yield strength (σ_y) and strain hardening exponent (n) are 4.8 μm , 166 $\pm$ 7 MPa and 0.28 $\pm$ 0.01 for SP-63 sample. The corresponding values for DP-63B are 2.1 μm , 213 $\pm$ 5 MPa and 0.25 $\pm$ 0.01, respectively. In fact, the presence of the non-equilibrium β phase in the BM leads to a substantial decrease of 58.3 pct in D_{av} , an increase of 28.3 pct in σ_y , and a little change in n of the sample. The origin of this modification effect of non-equilibrium β phase is scrutinized further.

The inverse pole figure (IPF) map and phase map of the BMs are illustrated in Figure 2. As seen in Figure 2, the SP and DP BMs have an average grain size of 49.5 and 38.5 μm , respectively. The grain boundary character distribution (GBCD) shows similar trend for the BMs. The fractions of high-angle grain boundaries (HAGBs, $\theta \geq 15$ deg), low-angle grain boundaries (LAGBs, 2 deg < θ < 15 deg), and twin boundaries (TBs, $\Sigma 3$) are 44, 29 and 27 pct for SP-63B, and 58, 21 and 21 pct for the alpha phase (α) in DP-63B. Moreover, the amounts of α and β phases in the DP-63B are 85.3 and 14.7 pct,

Fig. 1—The load-unload via displacement curves in conjunction with average σ_y and n for the SP-63B and DP-63B in the SZs.

Fig. 2—(a) IPF map of the SP-63B BM, (b) phase map, and (c) IPF map of the DP-63B BM.

respectively, in which β phase has been formed along α grain boundaries.

The orientation distribution functions (ODFs) of the BMs are illustrated in Figure 3. According to Figure 3(a), the main texture components for SP-63B are Cube $\{001\}100$ and Brass $\{011\}211$. The Cube and Brass components are common recrystallization and rolling texture components in face-centered cubic (FCC) metals and alloys, respectively.^[17] In fact, the SP-63B BM was initially rolled and then annealed, thus the recrystallization and rolling texture components exist in the BM. Figure 3(b) shows that the formation of β phase in the microstructure of DP-63B BM caused the elimination of Brass component and the formation of Goss $\{011\}100$ and Q $\{013\}231$. The origin of this texture transition is not clear.

The phase map, grain boundary (GB) maps, GB CD data of the transition zone from BM to stir zone (SZ), and the pole figures (PFs) of the SZ of the DP-63B sample are illustrated in Figure 4. In Figure 4, the thermomechanically affected zone (TMAZ) is divided into five regions from the point near the BM (Region 1) to the point near to the SZ (Region 5) in order to elucidate the gradual transition of the microstructure from BM to SZ.

As shown, the amount of LAGBs jumps from 21 pct in the BM to 75 pct in the TMAZ Region 1, and then gradually decreases to 16.6 pct in the Region 5. The amount of TBs drops from 21 pct in the BM to 6.4 pct in the Region 1, and then gradually increases to 26.9 pct in the Region 5. In fact, TBs are $\Sigma 3$ coincidence site lattice (CSL) boundaries in FCC materials that are rotated 60 deg about $\langle 111 \rangle$ axis. The strain applied by stirring tool leads to crystallographic rotations of these TBs from

their unique orientations resulting in the reduction of TBs from BM to TMAZ.^[18] Although the initial annealing TBs are disintegrated in TMAZs, some new $\Sigma 3$ TBs are developed in the next regions up to Region 5. Moreover, it is obvious that the amount of β phase decreases from Region 1 to Region 5.

Following the method used by Fonda *et al.*,^[19] the final rotated PFs of the SZ of DP-63B sample are illustrated in Figure 4(c). Obviously, the $A/\bar{A}\{111\}110$ shear texture is the main texture component in SZ, and the texture strength is equal to 2.549.

A comparison between the TMAZ and SZ of the DP-63B (Figures 4 and 5(a) through (b)) reveals that the fraction of LAGBs decreases significantly from the TMAZ to the SZ as opposed to HAGBs that show a large increase in fraction. Moreover, the SZ of the DP-63B alloy contained 99.4 pct α phase and 0.06 pct β phase, indicating that the amounts of β phase drastically decreases from the TMAZ to the SZ.

Reduction of the LAGBs from BM to SZ (Figures 4 and 5(a) through (b)), transformation of LAGBs to HAGBs (black arrows in Figure 4), and the presence of shear texture components in the SZ (Figure 4(c)) confirm the occurrence of continuous dynamic recrystallization (CDRX) during grain structure formation of DP-63B alloy. The change of misorientation angle along the T2, T3, and T4 lines in Figure 4, and the changes in the fraction of different misorientation angle scopes of Regions 2 to 4 in Figure 4 are illustrated in Figure 6. As seen, the point to origin misorientation is larger than 15 deg, which reveals that there is a large orientation gradient inside the deformed grains. The larger orientation gradient leads to easier transition of LAGBs to HAGBs, and hence it promotes the CDRX. However,

Fig. 3—The ODFs of the BMs: (a) SP-63B BM and (b) SP-63B BM at $\phi_2 = 0, 25, 33, 45$ and 63 deg.

the point to point misorientation inside the deformed grains is almost lower than 5 deg confirming that most of the LAGBs have misorientation angles smaller than 5 deg. This is why the amount of LAGBs with misorientation angles larger than 10 deg is very low (about 2 to 3 pct of the whole boundaries) as shown in Figure 6(b), which indicates that the CDRX cannot occur by subgrain rotation. Thus, these microstructural conditions are not suitable for occurrence of complete CDRX.^[20]

On the other hand, some new recrystallized grains in TMAZ as shown by the green arrows in Figure 4 are generated by bulging and twinning, which is the sign of discontinuous dynamic recrystallization (DDR).^[21] It is well documented that the nucleation and growth steps of the DDRX does not inherit the deformation textures, and hence reduces the texture intensity.^[22] Therefore,

CDRX and DDRX both occur in α phase during FSP. The occurrence of CDRX and DDRX is consistent with other investigations during FSP of Cu-Zn alloys.^[11,23]

As illustrated in Figure 4, some new grains nucleate on β phase in some areas as indicated by the blue circles, which are signs of particle-stimulated nucleation (PSN) mechanism as a type of DDRX. DDRX produces new grains with new textures, and hence the misorientation angle between adjacent grains is large enough to form new grains surrounded by HAGBs resulting in finer grains. It is well understood that during dynamic conditions the orientation of nucleus formed by PSN are not random with respect to the deformed matrix. However, the average of orientations around the particles over the whole polycrystalline matrix causes a nearly random distribution of nuclei orientations.^[24] In addition, according to Figures 4 and 5(a), FSP results in

Fig. 4—(a) Phase map of the DP-63B TMAZ in conjunction with GBCD data for different regions, (b) GB maps of the different regions: (b1) region 1, (b2) region 2, (b3) region 3, (b4) region 4, (b5) region 5. The black arrows show the LAGBs transforming to HAGBs. The dashed circles refer to the TB destruction. The green arrows belong to the DDRX TBs. The blue circles indicate the formation of α on β or $\beta \rightarrow \alpha$ transformation. (c) Rotated (111) and (110) pole figures with maximum texture strength of α in the SZ of DP-63B (Color figure online).

drastic reduction in the amount of β from 14.7 to 0.06 pct. From the Cu-Zn binary phase diagram^[25] and the measured peak temperature of 411 °C during FSP of DP-63B alloy, it can be inferred that the DP-63B alloy locates in the single phase area of the phase diagram as shown in Figure 7. Therefore, β could transform to α in this condition. In addition to DDRX and PSN, the $\beta \rightarrow \alpha$ transformation can also contribute to texture randomization. The texture randomization due to phase transformation is a direct consequence of the orientation relationship. For example in Kurdjumov and Sachs orientation relationship,^[26] there are 24 variants as follows:

$$\{110\}_{BCC} // \{111\}_{FCC}, 111_{BCC} // 110_{FCC} \quad [1]$$

During $\beta \rightarrow \alpha$ transformation, such orientation relationship could be created in α phase that results in texture randomization, and hence the intensity of shear texture components decreases. Similarly, Lee *et al.*^[27] reported such a result during thermomechanical process (repeated cold rolling and annealing at 680 °C) of 60Cu-40Zn double-phase brass alloy.

The remained β particles in the SZ are observed as districted nano-sized secondary phase at the grain

Fig. 5—(a) Phase map of the SZ of DP-63B sample. The higher magnification of the area indicated by white rectangle in (a) is shown in (b). The green arrows refer to the β on the grain boundaries of α . (c) IPF map of the SZ of SP-63B sample. (d) Rotated (111) and (110) pole figures with maximum texture strength of the SP-63B SZ.

Fig. 6—(a) Changes of point to point and point to origin misorientation angle along the T2, T3, and T4 lines in Fig. 4. (b) Changes in the percentages of different misorientation angle scopes of Regions 2 to 4 in Fig. 4.

boundaries of the newly recrystallized α grains as shown in Figure 5(b). In fact, after tool passage during FSP, nano-sized β particles pin α grain boundaries. Therefore,

it limits the mobility of the grain boundaries, and hence α grain cannot grow freely. The comparison between the microstructures and PFs of the DP-63B SZ (Figures 4(c)

and 5(a) through (b)) and the SP-63B SZ (Figures 5(c) through (d)) reveals that the presence of β phase reduces the grain size from 4.8 to 2.1 μm and it also reduces the maximum texture intensity from 3.365 to 2.549. In addition, Figure 3(d) shows that the main shear texture components in the SZ of SP-63B are the $A/\bar{A}\{111\}110$, $B/\bar{B}\{112\}110$, and $C\{001\}110$ components. In fact, β reduces the texture intensity by elimination of $B/\bar{B}$ and C components. This comparison confirms both the inhibition of grain growth and the promotion of the DDRX by β . In other words, the large β phases before the $\beta \rightarrow \alpha$ transformation in the TMAZ act as the DDRX nucleation sites, and after the $\beta \rightarrow \alpha$ transformation, the nanometer-sized β phases act as the barriers against the grain growth.

The dislocation structures in the SZ of DP-63B and the BM are illustrated in Figure 8. From Figure 8(a), the grains of the BM had a low density of dislocations, which was due to initial annealing process on the BM. Figure 8(b) discloses that FSP process has a significant

Fig. 7—(a) The Cu-Zn binary phase diagram. The red-dashed line and cross indicates the condition during FSW of DP-63B. (b) The temperature-time curve of the DP-63B sample.

influence on the density of the dislocations, which caused the formation of high dislocation densities in the SZ.

The effect of β on the microstructural evolution during FSP of brasses can be summarized according to the schematic illustrated in Figure 9. The shear stresses caused by the rotating tool deform and elongate the grains of BM (Figure 9(b)). Simultaneously, the deformed grains are subdivided into new subgrains by DRV, which transform to new DRX grains by CDRX (Figure 9(c)). As can be seen, the schematic color of the grains from deformation step to DRX step has not been changed, which is used to show that the governing mechanisms inherit the deformation textures. In addition to the CDRX, the DDRX takes place to form new grains by bulging mechanism at grain boundaries and triple points, which are shown by blue color areas. This mechanism does not inherit the deformation texture and leads to weaken it. Moreover, α nucleates dynamically on the β phase by PSN, which causes more reduction in texture intensity (Figure 9(b)). The amount of β reduces in the microstructure, however nano-sized β remains at the GBs, which prohibits the grain growth (Figure 9(d)). The final dislocations have tangle structures in α grains (Figure 9(d)).

IV. CONCLUSIONS

In this study, the effect of initial non-equilibrium heat treatment and β phase on the microstructure and mechanical properties of the FSPed Cu-Zn plates has been studied. In summary, using non-equilibrium heat treatment to produce non-equilibrium β phase in the brass BM, the microstructure and mechanical properties of the high zinc single-phase brasses can be modified during FSP. The β phase promotes the DDRX by particle-stimulated nucleation mechanism during the process and it transforms to α leading to random texture. More DDRX causes the formation of finer grain sizes. The distribution of the nano-sized β phase at

Fig. 8—(a) TEM image from the BM and (b) STEM image from the SZ of DP-63B.

Fig. 9—Schematic illustration of microstructural evolution during FSP in DP-63B. (a) BM consisting of α and β , (b) TMAZ, (c) SZ, and (d) substructures in the grains of SZ.

the grain boundaries of α is an additional cause for the refined structure, in which β reduces the mobility of the GBs during grain growth stage. Comparison between single phase and double phase FSPed samples indicates that β phase leads to decrease in both average grain size and strain hardening exponent correspondingly from 4.8 to 2.1 μm and from 0.28 to 0.25, and causes an increase in yield strength from 166 to 213 MPa.

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