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High-resolution EBSD characterisation of friction stir welded nickel–copper alloy: effect of the initial microstructure on microstructural evolution and mechanical properties

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ABSTRACT

The effect of the initial structure on the microstructure and texture of the friction stir welded Monel 400 alloy joints was investigated. For this aim, Monel 400 alloy of two initially treated conditions, annealed and rolled, was studied. Orientation imaging microscopy with a step size of 70 nm was used to characterise the microstructures of the joints. The mechanical properties were evaluated using Vickers hardness and nanoindentation tests. In the joint of the initial annealed alloy, continuous dynamic recrystallisation governed the grain structure formation. However, in the case of the initial rolled base material, discontinuous dynamic recrystallisation also occurred to cause more random texture, smaller grains, much more high-angle grain boundaries and higher dislocation densities. In the stir zone, the hardness and yielding strength increased respectively from 177 HV to 192 HV and 215.9 to 238.8 MPa by changing the base metal from the initial annealed to rolled condition. In addition, the microstructural features of the different zones containing the texture components and grain boundaries were scrutinised thoroughly.

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1. Introduction

Nickel–copper solid solution, i.e. Monel, has special characteristics such as high strength and good toughness over a wide range of temperatures and high corrosion resistance in different acidic and alkaline environments. [1,2]. Nowadays, Monel has attracted much attention for marine and petrochemical applications and therefore, the demand for welding of the Monel parts is growing. However, fusion welding procedures result in defects such as segregations, gas and

contraction porosities, residual stresses, etc., which reduces the final mechanical properties [3].

Some researchers have investigated the different aspects of fusion welding of Monel [4–8]. Ramkumar et al. studied the mechanical properties and corrosion behaviour of the Monel 400 joints welded by gas tungsten arc welding (GTAW) [4]. They produced defect-free joints, but the extensive grain growth was seen at the heat-affected zone (HAZ). Ventrella et al. used laser beam welding (LBW) to join the Monel 400 thin foils with lap configuration [5]. They reduced the heat input in order to achieve finer microstructure and to reduce the HAZ width. The other studies were done to reduce the residual stress of the fusion welding joints of the Monel 400 [6–8]. For example, Barsanescu et al. reduced the residual stresses of the conventional welded joints of the Monel 400 using a technique named ‘parallel heat welding’ (PHW) [6]. Javadi used the finite element method to study the clamping effect on the residual stress of the GTA welded Monel 400 joints [7]. Yegaie et al. considered a heat sink during GTAW of Monel 400, and developed a 3D thermo mechanical model to predict the temperature and residual stress of the joints [8]. They showed that using the water heat sink causes lower residual stresses in the vicinity of the joints. According to the literature [6–8], it is clear that conventional fusion welding processes can be used to join the Monel alloys, however, the related problems will be maintained.

Fortunately, friction stir welding (FSW), as a solid-state process, has been proved to be a promising joining method, which can eliminate the difficulties of fusion welding techniques [9]. Until now, a lot of research have reported that FSW can be used to weld different metals and alloys [10,11]. On the other hand, FSW leads to the generation of both the deformed and recrystallised grains, which usually improves the mechanical characteristics of the joints. Consequently, FSW can be a hopeful alternative to the fusion techniques for joining Monel alloys [12].

To our best knowledge, an investigation into the FSW of the Monel alloys is lacking. For the first step of Monel FSW, it is very necessary to have an understanding of the microstructural evolution during the process. On the other hand, the initial microstructure of the base metals (BMs) can influence the microstructural evolution [13]. This study aims to investigate the microstructure evolution during FSW of Monel with different initial microstructures of annealed and rolled conditions and the influence on the mechanical properties of Monel FSW joints.

2. Experimental procedure

The BMs used in this study were the Monel 400 (about 67% Ni–23% Cu) sheets with a thickness of 2 mm. Two different types of annealed and rolled BMs were selected to investigate the effect of initial condition on the microstructural and

textural evaluation during FSW. The FSW tool was made of WC-Co, which had a pin of $\varnothing 3 \times 1.75$ mm, and a shoulder with a diameter of 12 mm. For the both joints, the tool rotation speed and welding speed were set constant at 900 rpm and 25 mm/min, respectively. For the microstructural and textural study, electron back-scattered diffraction (EBSD) technique was used for orientation imaging microscopy (OIM). In order to accurate characterisation of the microstructures in the different zones of the joints, high-resolution OIM maps were acquired with a step size of 70 nm. The TSL-OIMTM software was used to evaluate the EBSD data. A Vickers hardness test was used to measure the microhardness (HV) of the different microstructural zones by applying 1.96 N load for 10 s. A nanoindentation test was employed for conducting at least 20 indentations for each microstructural areas by applying the maximum load of 40 mN. Moreover, yielding strength (σ_y) and strain hardening exponent (n) of the different microstructural zones of the joints were derived from the nanoindentation data, according to the method developed by Dao et al. [14].

3. Results and discussion

It is well documented that the friction stir welded joints are composed of different microstructural zones: the BM, heat affected zone (HAZ), thermo mechanically affected zone (TMAZ), and the stir zone (SZ). In this study, the microstructure and texture of these zones are thoroughly analysed in order to disclose the microstructural evolution [15–18].

3.1. Initial annealed condition

The OIM maps of the BM, including inverse pole figure (IPF) map, misorientation angle distribution (MAD), (111) pole figure (PF), and orientation distribution functions (ODFs) are illustrated in Figure 1. Figure 1(a,b) shows that the annealed BM was composed of large equiaxed grains, which had an average grain size of 17.8 μm . In addition, the BM grain boundaries (GBs) included 60%, 13%, and 27% of high-angle grain boundaries (HAGBs i.e. $\theta \geq 15^\circ$), low-angle grain boundaries (LAGBs i.e. $2^\circ < \theta < 15^\circ$), and twin boundaries ($\Sigma 3$ TBs), respectively. The BM had a nearly random texture with a maximum strength of 1.4 (Figure 1(c)). From ODFs (Figure 1(d)) it was found that the recrystallisation texture components of Q i.e. $\{013\}\langle 231 \rangle$ and R i.e. $\{124\}\langle 211 \rangle$ [19] were formed in BM. The origin of these texture components is the fact that the BM was produced by annealing the rolled material, where the deformation texture has been deteriorated by the recrystallisation components.

In the HAZ (Figure 2), some microstructural features were changed compared to those of the BM. From Figure 2(a,b), the average grain size was increased to 27.76 μm due to the grain growth, whereas the fraction of GBs was not changed considerably. It has been well documented that the HAZ can

Figure 1. OIM of the annealed BM: (a) IPF map, (b) MAD in conjunction with grain boundary characterisation distribution (GBCD) and average grain size (D_{av}), and (c) (111) PF and ODFs with texture strength. The black, white, and green lines in IPF (a) correspond to the HAGBs ($\theta \geq 15^\circ$), LAGBs ($2^\circ < \theta < 15^\circ$), and TBs boundaries, respectively. These colours are used for all the GBs in this paper.

be eliminated or narrowed in the friction stir welded joints made at low heat input condition [20]. In this study, we have selected the high heat input condition (900 rpm and 25 mm/min) to produce wide HAZ, which helps to characterise its microstructure and texture. From (111) PF (Figure 2(c)), the texture intensity was increased to 1.8. According to the ODFs (Figure 2(d)), the R and Q components were intensified. It can be concluded that the generated heat during FSW caused completion of recrystallisation and significant grain growth of the BM.

Inside the TMAZ (Figure 3), the microstructure was clearly changed. Figure 3 (a,c) shows that the amount of LAGBs was intensively increased from 14% in the HAZ (Figure 2) to 45%, which is illustrated by white coloured GBs. On the other hand, the amount of TBs was decreased from 28% (Figure 2(b)) to 9%. The reduction of the TBs is related to the fact that the deformation induced by the rotational tool causes TBs to lose their unique crystallographic orientation (60° about $\langle 111 \rangle$ axis). The grain average misorientation (GAM) map of the TMAZ is illustrated in Figure 3(b). A larger GAM value reveals that the material contains more dislocations, and conversely lower GAM values refer to the recrystallised grains with less dislocations [21]. According to Figure 3(b), the

Figure 2. OIM of the HAZ in the joint with initial annealed condition: (a) IPF map, (b) MAD in conjunction with GBCD and Dav, and (c) (111) PF and ODFs.

TMAZ had a bimodal structure of deformed and recrystallised grains. It is typically believed that the TMAZ contains elongated grains because the plastic flow and temperature are not enough for the occurrence of dynamic recrystallisation (DRX) [3,12]. However, in this investigation, the high heat input during FSW resulted in the partial DRX inside the TMAZ. The DRX grains of the TMAZ are characterised by small GAM and appear in light colour in the GAM map, as indicated by arrows (Figure 3(b)). From the (111) PF shown in Figure 3(d), the texture intensity increased to 7.9. In addition, according to the ODFs (Figure 3(d)), only the shear deformation component of C i.e. $\{001\}\langle 110 \rangle$ was formed in TMAZ. Close inspection to the IPF and GAM map of TMAZ (Figure 3(a,b)) reveals that in some areas the LAGBs are partly transformed to the HAGBs, as indicated by the white arrows in Figure 3(c). Moreover, some new recrystallised grains, which are surrounded by HAGBs, can be detected in the TMAZ (indicated by dashed circles in Figure 3(a,b)). It is clear that these new grains have a low dislocation density (according to the GAM map in Figure 3(b)) and they do not contain $\Sigma 3$ TBs, which are the signs of the continuous DRX (CDRX).

Figure 4 shows the OIM characterisation of the SZ. According to Figure 4, the SZ has an average size of 20.42 μm . The comparison between TMAZ and SZ (Figures 3 and 4) reveals that from TMAZ to SZ, the HAGBs and TBs have

Figure 3. OIM of the TMAZ in the joint with initial annealed condition: (a, and b) IPF and grain average misorientation (GAM) maps, (c) MAD in conjunction with GBCE, and (d) (111) PF and ODFs. The white arrows in (a) and black arrows in (b) show the LAGBs to HAGBs transformation and DRX grains, respectively.

been considerably increased, whereas the LAGBs have been decreased. In addition, the texture intensity has been increased from 7.9 to 12.8 (Figure 4(c)). From ODFs (Figure 4(d)), it is found that the texture of the SZ is composed of the shear deformation components of generally the C type and to some extent the A_1^* i.e. $\{111\}\langle\bar{1}\bar{1}2\rangle$. The generation of the A_1^* in addition to the C components shows that more slip systems have been activated inside the SZ. This is due to the intense slope of the temperature, strain, and strain rate gradients from TMAZ to SZ. In fact, the higher amounts of the deformation and temperature inside the SZ cause more activation of the slip systems. In addition, during FSW, some new grains are formed by a mechanism that has inherited the shear deformation texture of the TMAZ. It is well demonstrated that the CDRX typically inherits the deformation texture [22,23]. During CDRX, the dynamic recovery (DRV) results in the development of subgrains surrounded by LAGBs and then the

Figure 4. OIM of the SZ in the joint with initial annealed condition: (a) IPF map, (b) MAD in conjunction with GBCD and D_{av} , and (c) (111) PF and ODFs.

subgrains transform to the new grains surrounded by HAGBs. The transformation of the subgrains to new recrystallised grains is considered to occur by the gradual absorption of the dislocations to LAGBs, which cause a continuous increase in their misorientation up to those of HAGBs i.e. 15° [23,24]. The texture results and the evidence of DRV and transformation of the LAGBs to HAGBs disclose that the CDRX was the only mechanism for the formation of grain structure during FSW of Monel with initial annealed structure.

3.2. Initial rolled condition

The IPF map, GAM map, MAD, (111) PF, and ODFs of the rolled BM are shown in Figure 5. From Figure 5(a,b), the initially rolled BM had a bimodal microstructure including both deformed and recrystallised grains. According to the GAM map (Figure 5(b)), it is clear that the deformed grains had a high dislocation density or, in other words, high stored energy. In the case of GBs, the LAGBs had a considerable fraction of 73% (Figure 5(c)). The majority of these LAGBs have formed inside the deformed grains on the deformation bands (Figure 5(a)), which is due to the non-uniform stresses induced by the other grains and the intrinsic instability of the grains [25]. TBs had a low value of 5% in the BM, which were developed inside the recrystallised grains.

Figure 5. OIM of the rolled BM: (a) IPF map, (b) GAM map, (c) MAD in conjunction with GBCD, and (c) (111) PF and ODFs.

Figure 5(d) discloses that the texture intensity of the BM was 10.1. It is worthy to note that by omitting the recrystallised grains in the BM, the texture intensity was ever increased to 18.6, meaning that the formation of the recrystallised grains has reduced the average texture intensity. Analysing the ODFs (Figure 5(d)) revealed that the texture of the rolled BM contained the deformation component of Copper i.e. $\{112\}\langle 111 \rangle$, and the recrystallisation component of R i.e. $\{124\}\langle 211 \rangle$. In fact, the production of the R texture component by the formation of the recrystallised grains has reduced the texture intensity of the rolled BM. These features of the BM show that the BM was produced by a warm rolling process.

The OIM results of the HAZ are depicted in Figure 6. Microscopic probing from BM (Figure 5) to HAZ (Figure 6) discloses some important points. First, the amount of equiaxed or recrystallised grains with low GAM value has been increased. This was accompanied by an increase in the amount of HAGBs and TBs, and by a decrease in the fraction of LAGBs. In the HAZ area, due to the heat generated by the FSW and the existence of the deformed grains with high stored energy (high values of GAM), the static recrystallisation (SRX) can occur. In contrast, in the case of the annealed BM (Figure 2), the SRX was not seen in the HAZ. Second, the higher amount of TBs in the HAZ reveals that the SRX grains grow after the formation step. It is well documented that one of the mechanisms leads to the generation of TBs is the growth of the

Figure 6. OIM of the HAZ in the joint with initial rolled condition: (a) IPF map, (b) GAM map, (c) MAD in conjunction with GBCD, and (d) (111) PF and ODFs.

grains [26]. Third, the texture intensity has reduced from 10.1 to 4.7. Lower texture intensity in the HAZ indicates that the amount of recrystallisation texture components has been increased and hence the deformation texture components have been weakened.

Similar to the TMAZ of the annealed BM (Figure 3), the TMAZ of the rolled BM contained both the deformed and recrystallised grains. In this case, two different areas in the TMAZ were selected to be characterised, namely TMAZ I far from the SZ and TMAZ II near to the SZ. The OIM data of the TMAZ I are shown in Figure 7. In the TMAZ I, there were both the recrystallised and the deformed grains with low and high GAM values, respectively (Figure 7(a, b)). In addition, from HAZ (Figure 6) to TMAZ I, the amount of LAGBs was decreased to 35%; however, the amounts of HAGBs and TBs were increased to 39% and 26%, respectively. Figure 7(d) illustrates the (111) PF of the deformed grains in the TMAZ I. Accordingly, there was only the shear deformation texture component of C in the deformed grains with a considerable intensity of 17.5. It is believed that the deformation induced in the material by the rotational tool during FSW has a shear behaviour [27,28]. From the ODFs of the recrystallised grains in TMAZ I shown in Figure 8, in addition to the C type shear texture component, there is a recrystallisation texture

Figure 7. OIM of the TMAZ I in the joint with initial rolled condition: (a) IPF map, (b) GAM map, (c) MAD in conjunction with GBCD, and (d) (111) PF of the deformed areas in (a) or (b). Formation of the DDRX grains by bulging, and CDRX by LAGBs to HAGBs transformation are indicated by the black and white arrows in (a).

component of Cube i.e. $\{001\}\langle 100 \rangle$. Thus, during FSW, a mechanism causes the formation of new grains which do not inherit the deformation texture. Discontinuous DRX (DDRX) is believed to change and weaken the deformation texture [22,23]. Nucleation and growth are two distinct steps of DDRX.

Figure 8. ODF of the recrystallised areas in Figure 7(a or b).

During nucleation, the GBs bulging accompanied by the creation of LAGBs occurs and then the TBs or LAGBs form behind them especially in the triple points [23]. In the IPF and GAM maps of the TMAZ I (Figure 7(a, b)), there are some DDRX grains as designated by the black arrows in Figure 7(a). On the other hand, in the TMAZ I, there are also the evidence of CDRX i.e. LAGBs to HAGBs transformation shown by the white arrows in Figure 7(a). Thus, the CDRX and DDRX mechanisms are competing inside the TMAZ I of the rolled BM. In the TMAZ II (Figure 9(a–c)), the amounts of deformed grains and LAGBs were reduced, while the amounts of HAGBs and TBs were enlarged, compared to the TMAZ I (Figure 7). According to the (111) PF of the TMAZ II (Figure 9(d)) and ODFs of the deformed grains in the TMAZ II (Figure 10), in addition to the C shear texture, the A_1^* shear texture components were generated. Similar to the TMAZ I, the signs of DDRX and CDRX were detected in the TMAZ II, as marked by the black and white arrows in Figure 9(a), respectively.

The OIM results of the SZ of the rolled BM are illustrated in Figure 11. From the IPF map and MAD (Figure 11(a,b)), the SZ compared to that of the annealed BM (Figure 4) consists of a larger amount of HAGBs (58%) and TBs (33%), lower amount of LAGBs (9%) and smaller average grain size (8.19 μm). From the (111) PF of the SZ (Figure 11(c)), the texture intensity of 1.9 is rather low compared to that of the annealed BM (Figure 4(c)). The ODFs of the SZ

Figure 9. OIM of the TMAZ II in the joint with initial rolled condition: (a) IPF map, (b) GAM map, (c) MAD in conjunction with GBCD, and (d) (111) PF. Formation of the DDRX grains by bulging, and CDRX by LAGBs to HAGBs transformation are indicated by the black and white arrows in (a).

Figure 10. ODF of the deformed areas in Figure 8(a or b).

(Figure 11(d)) reveal that in addition to the C component, the recrystallisation texture component of Cube i.e. $\{001\}\langle 100 \rangle$ was existent. The recrystallisation texture components together and nearly random texture confirm that the

Figure 11. OIM of the SZ in the joint with initial rolled condition: (a) IPF map, (b) MAD in conjunction with GBCD and D_{av} , and (c) (111) PF and ODFs.

DDRX had a considerable contribution in the grain structure formation of Monel with the initial rolled structure. Since all the FSW parameters and hence the heat input were constant for the joints, the reason for the finer grain size of the SZ for the rolled BM than that of the annealed BM is related to the microstructural mechanism. So, DDRX causes finer grains compared to CDRX. This can be due to the fact that DDRX changes the deformation textures; however, CDRX inherits them.

3.3. Weld strength

In order to disclose the relationship between the microstructure and weld strength of the different zones, microhardness and nanoindentation tests were conducted on both samples, as summarised in Table 1. In addition, the nanoindentation curves for different zones of the joints are illustrated in Figure 12 from which the σ_y and n are calculated. From Table 1, some important results can be concluded. In the case of the annealed BM, the TMAZ had the maximum hardness of 194 HV, maximum σ_y of 253.4 MPa, and minimum n of 0.32 due to its deformed structure (Figure 3), whereas the other zones had recrystallised structures (Figures 1, 2, and 4). Furthermore, the HAZ had the minimum hardness of

Table 1. Micro hardness, σ_y , and n values of the different zones.

Joints		Mechanical properties			
		BM	HAZ	TMAZ	SZ
Annealed BM	HV	172 ± 5	162 ± 4	194 ± 4	177 ± 5
	σ_y	194.7 ± 7.5	171.5 ± 8.1	253.4 ± 6.3	215.9 ± 4.2
	n	0.43 ± 0.01	0.48 ± 0.02	0.32 ± 0.02	0.39 ± 0.02
Rolled BM	HV	216 ± 5	204 ± 3	198 ± 4	192 ± 4
	σ_y	308.4 ± 11.2	279.2 ± 10.1	261.3 ± 7.8	238.8 ± 7.6
	n	0.25 ± 0.01	0.28 ± 0.01	0.31 ± 0.02	0.33 ± 0.01

Figure 12. The load-displacement curves of the joints at different zones obtained from nano-indentation test.

162 HV, minimum σ_y of 171.5 MPa, and maximum n of 0.48 in the annealed BM sample, because of the growth of the grains (Figure 2). Moreover, in the annealed BM joint, FSW increases the hardness from 172 to 177 HV and σ_y from 194.7 to 215.9 MPa. In fact, FSW has caused the formation of finer grains and higher dislocation density, which have strengthened the annealed BM.

In the case of the rolled BM joint, the hardness and σ_y decrease gradually from the BM (216 HV and 308.4 MPa) to the SZ (192 HV and 238.8 MPa). On the other hand, the n value increases from 0.25 to 0.33. For more quantitative analysis, the fraction of the recrystallised grains was estimated using OIM. The fractions of the recrystallised grains in the BM, HAZ, TMAZ, and SZ of the rolled BM joint were estimated respectively equal to 12.8%, 38.7%, 76.2%, and 100%. Thus, it can be concluded that the gradual replacement of the deformed grains with recrystallised grains by FSW leads to continuing decrease of strength from BM to SZ of the rolled BM joint.

Comparison between the hardness values of the SZs in the joints with different initial BMs reveals that when the BM had rolled condition, the resulted joint had higher strength. Two kinds of strengthening mechanisms can be suggested for this effect. When the BM had an annealed initial structure, CDRX was the dominant mechanism and leads to larger grain sizes. In addition, dynamic recovery during CDRX mechanism can reduce the dislocation density. Therefore, CDRX is responsible for the lower strength of the joint in the sample with an initial annealed structure. On the contrary, in the sample with initial rolled structure, DDRX causes higher strength by grain boundary and dislocation strengthening mechanisms.

4. Conclusion

In this study, the effect of initial conditions of Monel 400 BMs on the microstructure, texture components and strength of the different zones of the friction stir welds was investigated using high-resolution EBSD and Vickers hardness test. The following conclusion can be summarised. When the Monel 400 alloy with an annealed structure is friction stir welded, the grain growth in HAZ, DRV and CDRX in TMAZ, and pure CDRX in SZ occur. These mechanisms cause a lower fraction of HAGBs, larger grains, and higher texture intensity in the final structure of the joint. On the other hand, in the case of the Monel 400 alloy with an initial rolled structure, SRX in HAZ, and CDRX and DDRX in TMAZ and SZ of the joints are the key mechanisms, governing the microstructural evolution during FSW. The higher contribution of the DDRX causes the formation of a higher fraction of HAGBs, finer grain sizes, higher dislocation density, and higher texture intensity in the final microstructure of the joint. Therefore, it is better to choose the Monel 400 BM with an initial rolled structure for FSW because

the strength of the joint will be improved by grain boundary and dislocation strengthening mechanisms.

Disclosure statement

No potential conflict of interest was reported by the authors.

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