Effect of FSP process parameters with air blowing on microstructure and hardness of NiAl Bronze alloy

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Abstract

The present work investigates the effect of friction stir processing on microstructure evolution—grain refining and phase transformation— and hardness of NiAl bronze alloy. NiAl bronze alloys have a wide range of industrial applications especially in the marine field. The technique is based on applying friction using a rotating tool causing heat generation and material mixing. The effect of process parameters, such as; rotation speed, traverse speed, forced convection were investigated in this research. At constant traverse speed, the effect or rotation speed was studied at 800, 1000, and 1200 rpm. At constant rotation speed, the effect of traverse speed was studied at 40, 64, and 93 mm min−1. Minimum grain size was achieved at FSP conditions 800 rpm/93 mm min−1. Highest average hardness value was achieved by FSP 1200/93. At constant rotation and traverse speeds, the application of forced convection using air blowing at a rate of 4.5 m3 min−1 was studied and it was found to reduce the grain size within the SZ and to increase the average hardness value.

1. Introduction

The NiAl bronze (NAB) alloy is one of the several commercial aluminum bronze (AB) alloys with Al ranging between 9 wt% to 12 wt%, in addition to both Ni and Fe up to 6 wt%, and 1wt% Mn [1]. This alloy is commonly used in marine applications such as; screw propellers due to its pronounced resistance to corrosive atmospheres, in addition to its acceptable mechanical properties [2]. However, poor cast properties of NAB alloy is considered a major limitation since casting defects, such as shrinkage porosities and segregation of alloying elements, may impair the mechanical and corrosion properties of the cast alloys therefore limiting the service life of the components [2–5]. In addition, ship propellers are designed with various thicknesses leading to microstructural inhomogeneity given that thick sections cool slower than thin ones, resulting in the generation of relatively coarse microstructure within thick sections compared to thin sections. Hence, in applications where casting process is preferred yet other properties like, strength, wear resistance, corrosion resistance, and cavitation resistance are still required, a surface hardening process may offer a convenient and satisfying solution [6].

Hot deformation is one of the optimum approaches to refine grains and eliminate casting defects, which significantly improves the mechanical properties of cast metallic materials. Several plastic deformation techniques have been used to improve the mechanical properties of cast NAB alloys such as equal channel angular extrusion (ECAE), friction surfacing (FS) and friction stir processing (FSP) etc. It is reported that the plastic deformation significantly improves the mechanical properties of as-cast NAB alloy [7]. Friction stir processing (FSP) is a recent solid-state process that aims to improve surface properties of the material using a non-consumable rotating tool. It was first developed by Mishra who upgraded the use of friction stir welding (FSW) technique [8]. FSP induces localized severe plastic deformation and dynamic recrystallization to the surface of the material. FSP gained its competitiveness from generating heat high enough to achieve structural recrystallization yet not too high to melt the metal thereby maintaining the processing in the solid state condition [9].
Recently, FSP has been extensively used by US navy to repair and enhance the properties of the NAB propellers which consequently increased its performance and service life [10, 11]. Further research studied the effect of FSP on microstructural evolution and mechanical properties of as cast (AC) NAB [3, 12, 13]. It was reported that FSP processing parameters, such as tool rotation speed, tool traverse speed, tool geometry, and number of passes, significantly influenced the microstructure and mechanical properties of NAB alloys [13].

Previous literature discussed the effect of including a cooling system during FSP on magnesium alloys, such as; AZ31 [14], and AZ91 [15]. However, studying the effect of cooling on the properties of NAB alloy was rarely studied. Yang et al [16] applied the FSP to modify cold-sprayed AA2024/Al2O3 metal matrix composites and investigated the effect of rotation speed on tensile and microhardness using FSP technique and found that a larger rotation speed of 1500 rpm resulted in a maximum increase of 25.9% in ultimate tensile strength and 27.4% in elongation in addition to an effect of rotational speed on fluctuations of friction coefficients. In a recent review by Patel et al [17] of the latest progress in FSP, the process was also found to show a great potential in repairing and or modifying the weld or coating structure by refining the microstructure. It has also illustrated a potential in inducing a superplastic behavior in aluminium alloys [18] where the tool rotation and travel speed affect the stirring and mixing of the material which highly influence the microstructure.

The purpose of this work is to study the effect of rotation speed, transverse speed, and cooling effect on hardness profiles along, across and in-depth in the FSP zone of C95500 cast NAB alloy.

2. Experimental work

Cast NiAl bronze UNSC95500 alloy with thickness of 10 mm was the sample material used in this study. The chemical composition of the alloy is shown in table 1. Samples were extracted from a cast gear and machined to dimensions of 100 × 30 × 10 mm. The FSP tool shape and dimensions are given in figure 1. In order to avoid tool softening, breakage or excessive wear during the process, the FSP tool was made of hard carbide material used as cutting tips in milling processes.

2.1. Friction stir processing

FSP took place on a BEMATO vertical milling machine (model: pBM-GVS250) from Benign Enterprise Co., Ltd

Different rotational speeds (ω) of 800 rpm, 1000 rpm, and 1200 rpm were applied keeping a constant traverse speed (v) of 93 mm min⁻¹ while three different traverse speeds of 40, 64, and 93 mm min⁻¹ were applied keeping a constant rotation speed (ω) of 800 rpm. In order to reduce the temperature of the tool, forced convection using air blowing applied. This was achieved using an air blower delivering 4.5 m³ min⁻¹. The blower was fixed 250 mm away from the processed region. Its nozzle was oriented directly towards the FSP tool.

The tool shoulder used in treatment was made of hardened H13 steel, while the tool pin was tungsten carbide, figure 1. The pin diameter, length, and shoulder diameter were 6 mm, 3 mm, and 25 mm, respectively. The depth of penetration was defined by the depth at which the pin will fully sink in, and the shoulder would fully touch the sample surface, then penetration at 0.2 mm below sample surface. The samples will be identified in the following text by rpm/traverse speed (for example 800/93) and as FSP–Only and FSP–Air in case of presence or absence of air blowing.

2.2. Temperature measurement

The temperature profile was determined using three K-type 0.4 mm wire sheathed thermocouples (TCs) with the data recorded, converted to a digital signal, and transferred through a MAX6675, finally sent to ARDUINO UNO micro-controller to be recorded and stored. TCs were protected with a 4 mm diameter ceramic sheath and fixed at penetration, middle, and exit areas and labelled; A, B, and C, respectively, figure 2.

2.3. Microstructure investigation

Samples were ground to 1200 grit SiC paper and polished using up to 1 μm diamond paste suspension. Micrographs were acquired using Imager A1m Zeiss Microscopy throughout the stir zone. Scanning electron microscope SEM InspectTM 550 was also used for much higher magnifications of each treatment. Phase fraction

| Table 1. Chemical composition of UNSC95500 alloy. |
|-----------------|------|------|------|------|------|
| Element         | Cu   | Al   | Ni   | Fe   | Mn   |
| %               | Balance | 10.1 | 4.83 | 4.277 | 1.52 |
| Others          | 1.2915 |      |      |      |      |
Figure 1. FSP Tool and dimensions (a) phot, (b) drawing.

Figure 2. Thermocouple Distribution; TC-A is the penetration point, TC-B is the middle point, and TC-C is the exit point.
and average grain size within the stir zone was measured using the image processing program ImageJ on at least 3 locations in each sample.

2.4. Hardness measurement
Hardness values were measured on Zwick ZHV10 Vickers hardness testing machine. Hardness was measured on the surface in several points along the FSP pass, across, and in-depth in FSP zone of NAB alloy and the average values were calculated. Samples were ground to 1200 Grit SiC paper, prior to measurements. Indentations were measured after applying a load of 5 kg and 15 s dwell time.

3. Results and discussion

3.1. Temperature investigation
The temperature distribution in FSP using a tool rotating at 1200 rpm and moving in feed direction at 93 mm min⁻¹ is shown in figure 3. The effect of forced convection using air cooling was also investigated and identified as (FSP-Air) while the case without air cooling as (FSP-Only). Results are based on four identical trials.

In case of FSP-Only, during penetration (position A) the rotating tool pin and shoulder caused a gradual rise in temperature due to gradual increase in penetration depth until reaching its maximum value, figure 3(a). Steady and smooth temperature evolution was observed within positions B and C during both heating and cooling stages. Hence, any cross-section within the FSP path is expected to have a steady temperature evolution pattern during heating and cooling stages, except for the penetration region. The peak temperature value, heating and cooling rates within points A, B and C were shown in table 2.

In case of FSP-Air, during penetration (A-air) there was retardation in temperature rise for all points A, B and C relative to the case with no air blowing, figure 3(b). A relatively higher peak temperature in points A and B was noticed in case of air blowing which could be due to the fact that air was blown on the hot tool, table 2.

The effect of air blowing on the peak temperature value, heating and cooling rates were also shown in table 2. It can be observed that cold air blowing helped dissipating the heat energy in points B and C, hence, increasing the cooling rate. Increasing the cooling rate is expected to affect the microstructure of the NAB alloy and consequently its mechanical properties such as hardness. During pin exit region, points C-only and C-air reached about 414 °C for both cases with and without air blowing. Air blowing had no significant effect neither on temperature uniformity nor the peak temperature values within the pin exit point C.

Analyzing the temperature profile throughout the FSP pass was rarely studied. Temperature results were commonly recorded only within the middle of FSP pass, ignoring the penetration and exit regions. The temperature profile patterns for the middle region, B-only and B-air, in present work were confirmed by previous temperature evolution results [19].

3.1.1. Temperature estimation
The peak stir zone (SZ) temperature could be estimated using two different methods. Since measuring the temperature directly is not usually convenient, analytical methods could be used in order to estimate the peak temperature based on FSP process parameters, or microstructural evolution within SZ.

3.1.1.1. Estimated temperature based on FSP process parameters
First, given the rotation speed, traverse speed, and (T_melt) of the material, the peak temperature values could be estimated using the following computational model in equation (1), where, K is a constant reported by about 0.65–0.75, while α is an exponent that ranges from 0.04–0.06 [20], and finally (T_melt) range for NAB alloys is 1060 °C–1075 °C [21]. According to equation (1), the estimated peak processing temperatures are expected to range between 961 °C and 994 °C. Estimated maximum temperature (T_max) is expected to decrease at higher traverse speeds (v), while increase at higher rotation speeds (w).

\[
\frac{T_{\text{max}}}{T_m} = K \left( \frac{\omega^2}{v \times 10^4} \right)^\alpha
\]  

(1)

3.1.1.2. Estimated temperature based on quantitative analysis of \(\beta^\prime\) Martensitic phase
Second, based on quantitative analysis of NAB alloy constituents, peak SZ temperatures could be estimated based on microstructure since the constituents of the alloy are affected by the FSP-induced thermo-mechanical history. The percentage of \(\beta^\prime\) phase within SZ microstructure may reflect the peak temperature. Using equation (2), peak temperature can be conveniently estimated through the volume fraction of \(\beta^\prime\) phase \(V_{\beta^\prime}\) determined from microstructural investigation [14]. The \(V_{\beta^\prime}\) data used within this study was based on the microstructural analysis which will be discussed later in details. Figure 4 indicates the peak SZ temperature
Figure 3. Temperature distribution corresponding to TCs readings during FSP at 1200rpm and 93 mm min$^{-1}$ (a) FSP-Only and (b) FSP-Air.
estimated based on $V_\beta$ and on process parameters is lower than that estimated from equation (1) reaching a value of 800 to 830 °C.

$$T = 244V_\beta + 758 \quad (^\circ C)$$

(2)

It is noticed that the peak temperatures estimated based on quantitative analysis appeared to follow the same pattern as the peak temperatures estimated based on FSP process parameters. Increasing traverse speed from 40 to 93 mm min$^{-1}$ reduced the peak temperature using both equations (1) and (2) while increasing rotational speed from 800 to 1200 rpm raised the peak temperature due to increase in thermal energy due to friction. The highest peak temperature was also observed at highest traverse speed and least rotation speed. However, a difference was noticed within each treatment.

Previous studies reported the variation in peak temperatures results from thermocouples according to their placement with respect to the stir zone. If TCs were placed outside SZ, peak temperatures were found to be 0.6 to 0.8 $T_{\text{Melt}}$ during the FSP/FSW of various metals and alloys [22]. While if they were placed inside SZ peak temperatures were found to be $\geq 0.9 T_{\text{Melt}}$ during the FSP of NiAl bronze. Also, quantitative analysis of the microstructural constituents predict temperatures to exceed 0.9 $T_{\text{Melt}}$ [19].

Heating during FSP is attributed to a combination of adiabatic deformation within the material surrounding the tool pin in addition to the friction due to the interface between the tool and sample surface. The equilibrium of forces and moments, traversing of the tool, and localized plastic deformation induced by tool rotation result in rapid transients and steep gradients in the SZ strain, strain rate, and temperature [19]. At high transient temperatures, the uncertainty within K-type TCs’ readings may occur as mentioned in previous literature [23, 24]. The mentioned uncertainty may be attributed to: (1) thermal inertia (2) electrical amplifiers (3) analog-to-digital converters (4) surrounding temperature compensation (5) presence of electrical/magnetic fields, or (6) random ground currents [24]. This may explain not only the deviation within thermocouple measurements, but also the peculiar effect of air cooling in increasing the peak temperature by about 50 °C. This is probably due to the orientation of blown air towards the hot tool end. The data processed from the transient thermal system may have been delayed by the thermal inertia of the thermocouple, and due to the traverse speed, thermocouples might had insufficient heat exposure equivalent to that received by the sample. Also, application of air cooling may have decreased the thermal inertia of both the sample surface and the thermocouples which enhanced the overall heat transfer coefficient due to forced convection to the surface of the sample thereby increasing the results of heating and cooling rates.

### Table 2. Thermocouple data.

|         | FSP-only |         | FSP-air |         |
|---------|----------|---------|---------|---------|
|         | A        | B        | C        | Aair    | Bair    | Cair    |
| $\frac{dT}{dt}$ $\text{Heat} \quad ^\circ \text{C} / \text{s}$ | 28.29    | 22.08    | 11.76    | 94.67   | 24.75   | 17.43   |
| $\frac{dT}{dt}$ $\text{Cool} \quad ^\circ \text{C} / \text{s}$ | $-4.29$  | $-14.26$ | $-10.78$ | $-25.71$| $-19.6$ | $-17.69$|
| $T_{\text{peak}}$ $\quad ^\circ \text{C}$ | 498      | 518      | 414.45   | 553     | 572     | 414.3   |

Figure 4. Estimated peak temperature indirectly based on FSP process parameters and Vol% of martensite phase.
3.2. Microstructure investigation

3.2.1. As-cast microstructure

The microstructure of UNSC95500 NAB as-cast alloy was investigated using different magnifications. The microstructure mainly consists of coarse α grains (light phase color), β′ martensitic phase (dark phase), and κ-phase (dark particles), figure 5, with a Widmanstätten morphology. The eutectoid phase $\alpha + \kappa_{\text{II}}$ appeared within the boundary line of coarse α grains. Relatively large $\kappa_{\text{II}}$ rosettes appear inside α grains. The smaller rosette-shaped $\kappa_{\text{III}}$ particles appeared within α grains. Finally, $\kappa_{\text{IV}}$ appeared as very fine spherical particles at the center of α grains. This description was confirmed by previous literature [3, 9, 11, 13].

3.2.2. Effect of FSP on surface features of NAB alloy

Generally, the surface of the samples treated with FSP had a unique pattern observed regardless the process parameters, sample material, or the tool parameters. A linear pattern of wavy cycles joined together was observed starting from the pin insertion until exit area, as shown in figure 6. The tool rotation direction was kept counter clockwise for all samples. Hence, we can define the two sides to the FSP line as; advancing side AS (right side), and retreating side RS (left side). Some excess material was observed at the AS, namely ribbon flash, which is attributed to the relatively smoother and faster material flow in comparison to the RS. At the AS, the tangential
velocity vector of the rotation speed is in the same direction as that of the tool traverse direction, while at the RS, the tangential velocity vector of the rotation speed is opposite to the tool traverse direction. This relative velocity may explain the excess in material flow noticed at the AS. A pin exit hole was observed at the end of the FSP path where the tool left the work-piece. It is one of the most unique characteristics of FSP and FSW since the pin is always the last part leaving the sample surface. This pin hole is considered a defect. However, in real applications, the path of FSP can be selected so as to avoid such defect, for example, by overlapping FSP lines together so that the FSP path would omit any previously existing pin holes.

3.2.3. Macroscopic investigation of samples treated with FSP
Due to FSP, four zones were induced to the cast microstructure; stir zone SZ, thermo-mechanically affected zone TMAZ, heat affected zone HAZ, and finally the base metal BM, as shown in figure 7. In the stir zone SZ, FSP had a general effect of achieving significant grain refinement and phase transformation in comparison to the as-cast microstructure, which is attributed to dynamic recrystallization caused by the presence of intense plastic deformation and heat exposure. The thermo-mechanically affected zone TMAZ has relatively less heat exposure with lamellar grain morphology. The heat affected zone HAZ had relatively coarser grains with relatively more $\beta'$ martensite, and less $\kappa$-phase precipitates in comparison to the base material BM, which will be discussed later.

3.2.3.1. Effect of FSP process parameters on the treated region
The FSP treated region, shown in figure 8, represents FSP operated at 1000 rpm and 93 mm min$^{-1}$. FSP process parameters, such as: rotation speed, traverse speed, and cooling rate are considered from the major parameters that can significantly affect the microstructure of the FSP treated region, and consequently the surface properties of the sample, as found in previous work [25, 26]. FSP with different cooling rates has been demonstrated as a one of the variants of FSP to enhance superplastic behavior with grain refinement and equiaxed grains. In order to clarify the size of FSP treated region with respect to the FSP tool pin and shoulder, a schematic view of the FSP tool was presented with equivalent scale in figure 8. Generally, it can be observed that the maximum depth within the treated area is located at the center of the sample cross-section corresponding to the position where tool pin rotates causing material mixing, which is called the stir zone SZ. At the terminals of the FSP region,
relatively less treated area appeared due to the effect of friction between the tool shoulder and the sample top surface. In between these regions, due to the combined effect of surface friction, between tool shoulder and sample surface, and material mixing, caused by the tool pin; the FSP treatment tends to gradually diminish from the center of the SZ to the end of the tool shoulder. The rotating tool affects not only the zone where the pin rotates but also a much bigger zone. This zone is extended downwards below the pin and laterally beyond the tool shoulder similar to a parabolic pattern observed in figure 8. This indicates that a much larger volume of material is affected by FSP due to heat and mass flow.

The dimensions of the stirred zone (depth and width) and total width, illustrated in figure 9, indicate that at constant traverse speed, the area of the FSP treated region increased with increasing the rotation speed. This increase could be attributed to the increase of both heat generation and material stirring mechanisms. According to the estimated peak temperatures shown in figure 4, the peak temperature was expected to increase with the increase of the rotation speed, which explains the increase in FSP treated region. Therefore, the FSP zone for a rotational speed of 1200 rpm was larger than that obtained at a rotational speed of 800 rpm. On the other hand, the traverse speed had only a slight effect on the dimensions of the FSP region in comparison to the rotation speed. It is interesting to note that with the same tool penetration depth, the SZ depth increased from 4.5 to 5.5 mm with increasing the tool rotational speed, and the total width increased from 18 mm to 35 mm and to 44 mm with increased rotational speed from 800 to 100 and to 1200 rpm. The mostly affected increase in size was the total SZ width. The edges of processed zone appeared to become smoother at higher rotation speeds, which indicates that the material mixing became more uniform. This could be attributed to the combined effect of increased softening caused by the increased heat input and the increased stirring mechanism, which are all...
correlated with the rise in rotation speed. According to the estimated peak temperature plotted in figure 4, the generated heat is expected to decrease with the increase of the traverse speed. The higher the traverse speed, the shorter the contact time between rotating FSP tool and sample surface will be, resulting in earlier free convection and conduction which limits the temperature rise.

Generally, it can be observed that the variation in rotational speed had a larger effect on the dimensions of the FSP regions compared to the traverse speeds, hence the macrostructure of the FSP zone, figure 9. It can also be observed that FSP/1200/93 achieved the maximum treated region with maximum SZ area indicating that applying FSP in surface hardening will require less number of paths by processing larger areas in one path. This will definitely decrease the processing time.

Air blowing was introduced during FSP treatment in order to reduce over heating of the FSP tool. Air blowing at 4.5 m³ min⁻¹ acted as a forced convection cooling unit which resulted in reducing the propagation of generated heat throughout the base material. The results in figure 9 also indicates that the application of air cooling during FSP treatment caused 61% decrease to the FSP treated area. With application of air cooling in FSP1200/93/Air, the FSP treated region was significantly minimized making it nearly equivalent to the FSP treated region achieved by FSP800/93.

3.2.4. Microscopic investigation of samples treated with FSP

Studying the effect of FSP process parameters on the microstructure indicates the significant microstructural modifications resulting from the changes in tool rotation speed, tool traverse speed, and application of air cooling. A high magnification optical micrographs were taken of the SZ subsurface, figure 5. The light-etching areas represent the Cu-rich α grains while the dark-etching areas apparently included fine Widmanstätten α as well as bainite α + β' and martensite (darkest features). The lamellar structure associated with α + 3\(\alpha_{III} \) was not apparent in any of these micrographs. Hence, it is suggested that the lamellar α + 3\(\alpha_{III} \) within as-cast material has reverted to β during severe material deformation and heat exposure of FSP. However, the primary α formed during equilibrium cooling has not been completely retransformed to β and, so, the microstructure during FSP mainly consists of α and β in varying amounts, depending on the local peak temperature. Both phases deform and then the β transforms on subsequent cooling, giving Widmanstätten α, or bainite or martensite, depending on the cooling rates following passage of the tool. Such cooling rates are expected to be of the order of 10⁸ °C s⁻¹, i.e., much faster than associated with equilibrium cooling [14].

The effect of the rotation speed and traverse speed on the microstructure within SZ can be illustrated in details in figures 10, 11 and 12 for FSPed samples under constant traverse speed of 93 mm min⁻¹, different rotation speeds, with and without the application of air blowing. Figure 1 illustrates the percentage β martensite phase and average grain size within SZ which were measured using ImageJ software. Grain refinement and phase transformation were generally achieved in comparison to as-cast alloy due to dynamic recrystallization caused by localized severe plastic deformation and heat exposure. The percentage of β martensite phase and average grain size within SZ at rotation speeds 800, 1000, 1200 rpm were found to be 22.3%, 29.2%, 30.1%, and 4.5, 11.6, 18.1 μm, respectively. Maximum percentage of β martensite phase and grain size were achieved at 1200 rpm while finer grains were found at lower rotation speeds. Previous study on pure copper in cryogenic temperature by Wang et al [27] indicate that the average grain size first increased as the rotational speed increased from 400 to 600 rpm then decreased with increasing rotational speed from 600 to 1200 rpm. In the present study the grain size increased as rotational speed increased from 800 to 1200 rpm; the difference in behavior could be due to difference in alloy composition, traverse speed and processing temperature. The authors in [27] explained the refined grains at low speeds due to dislocation cells while at high speeds dynamic recovery further refined the grains. With application of air blowing, the percentage of β martensite increased and the average grain size decreased in comparison with no air blowing. By increased rotational speeds the β martensite increased gradually to be 30.7%, 32.7%, 33%, with an increase in grain size of 1.9, 6.9, 7.3 μm, respectively.

The effect of traverse speed on microstructure was also investigated. Figure 12 presents SEM images of SZ under constant rotation speed of 800 rpm, different speeds, with and without the effect of air blowing. SEM micrographs illustrate the grain size of the SZ with respect to the different traverse speeds used. Figure 1 illustrates the percentage of β phase and average grain size within SZ of FSP samples treated at constant rotation speed of 800 rpm and traverse speeds of 40, 64, 93 mm min⁻¹, giving values of 33.9%, 29.3%, 22.3%, and 21.8, 13.2, 4.5 μm, respectively. The maximum percentage of β phase and largest grain size were observed at the lowest traverse speed of 40 mm min⁻¹ due to the increased heat exposure. Increasing the traverse speed from 40 to 93 mm min⁻¹ achieved 79.6% reduction in grain size, i.e., grain refinement. On the other hand, the minimum phase fraction and grain size were found at the highest traverse speed. Increasing the traverse speed from 40 to 93 mm min⁻¹ achieved 56.3% reduction in grain size. In other words, increasing the traverse speed appeared to have the opposite effect as increasing the rotation speed. The grain morphology changed from elongated to equiaxed grains associated with increasing the traverse speed. At lower traverse speeds, the excess in thermo-mechanical exposure may cause not only grain growth, but also formation of recrystallized grains.
Considering the effect of air blowing, larger % of $\beta'$ and finer grains were obtained so that the percentage of $\beta'$ phase and average grain size were 48.1%, 38.4%, 30.7%, and 4.4, 2.6, 1.9 $\mu$m, respectively. Therefore, FSP treatment itself caused grain refinement and formation of hard $\beta'$martensite phase. Increasing the rotation speed increased peak temperature which increased the grain size, decreased the grain refinement, and increased the retransformation of more $\alpha$ to $\beta$ during heating range, which increased the subsequent percentage of $\beta'$ martensite phase formed during subsequent cooling. The peak temperature was expected to increase for higher rotation speeds due to increasing the friction and stirring intensity. Also, higher peak temperatures were expected at lower traverse speeds due to increasing the time exposure to friction and heat generation. Application of air blowing is expected to raise the thermal inertia due to forced convection, consequently increasing heating and cooling rates. Figure 13 illustrates the summarized view of the effect of FSP process parameters, such as; rotation speed, traverse speed, and air blowing on the microstructural features of the SZ represented by phase transformation and grain refinement within microstructure. In comparison to 201.5 $\mu$m which is the average grain size the as-cast sample, the achieved grain refinement indicates that recrystallization throughout SZ was successfully achieved due to heat exposure and SPD. Excessive heat

Figure 10. Microstructure of SZ using different process parameters.
exposure either by higher rotation speed or lower traverse speed led to grain growth. In general, the minimum grain size was obtained at 800/93/Air condition and the maximum% of \( \beta' \) martensite at 800/40/Air condition. These results confirm previous studies in applying FSP on NAB alloy. Lv reported that in addition to the rise in peak temperature, the strain rates also increased within SZ causing dissolution of \( \kappa \) precipitates and significant increase in the formation of \( \beta' \) martensite phase [15]. Therefore, increasing heat exposure generally increased the rate of phase transformation thereby the dissolution of more \( \kappa \) particles and formation of more \( \beta' \) martensite phase.

**Figure 11.** SEM images of (a) as-cast, and after FSP at 93 mm min\(^{-1}\) and (b) 800, (c) 1000, (d) 1200 rpm, (e) and (f) air blowing at 1000 and 1200 rpm respectively.
3.3. Effect of FSP on hardness profile in FSP pass

3.3.2. Effect of FSP process parameters on hardness values

Generally, enhancement in hardness values were achieved in FSP samples in comparison to as-cast due to formation of wrought structure attributed to localized recrystallization, grain refinement, phase transformation, and elimination of casting defects; such as porosities. Significant increase in the percentage of the relatively harder $\beta'$ martensite phase and the finer grain size in SZ were strongly correlated with higher hardness, as would

Figure 12. SEM images of (a) as-cast, and after FSP at 800 rpm and (b) 40, (c) 64, (d) 93 mm min$^{-1}$. (e) And (f) with air blowing at 64 and 93 mm min$^{-1}$ respectively.
be expected from the Hall-Petch relationship [10]. However, some process parameters appeared to be relatively more significant than others on hardness.

3.3.3. Effect of rotation speed and traverse speed on hardness values

The increase in rotation speed from 800 to 1200 rpm at constant traverse speed of 93 mm min$^{-1}$ raised the hardness in the SZ, figure 14(a). The hardness values were 264, 242, and 270 HV5, corresponding to rotational speeds of 800, 1000 and 1200 rpm with an improvement of 75.8%, 61.7%, and 80% in comparison to as-cast hardness. Increasing the rotation speed appeared to have a combined effect on the microstructure of the alloy through increasing the rate of formation of the hard $\beta'$ martensite phase and grain growth due to rise in peak temperature. This may explain the decrease in hardness values within the FSP/1000/93 treatment. Lv also reported that the thermo-mechanical exposure attributed to increasing the rotation speed will also lead to the dissolution of more $\kappa$ hard precipitates. In addition, increasing rotation speed on FSP treated NAB alloys is expected to display higher content martensite nanotwins and to produce more dislocations within the microstructure [15]. Hence, increasing the rotation speed caused the strengthening mechanisms to gradually change from grain refinement and secondary phases strengthening to solid solution, dislocations and nano-twin strengthening [15].

The effect of traverse speed on the average hardness values was also studied. Figure 14(b), illustrates that the average hardness values at 40, 64, and 93 mm min$^{-1}$ at constant rotational speed of 800 rpm, were 240, 262, and 264 HV5, hence, achieving hardness enhancement compared to as-cast alloy by 60%, 74.9%, and 75.8%, respectively. Increasing traverse speed from 40 to 64 mm min$^{-1}$ raised hardness from 240 to 262 HV5 achieving 9.1% enhancement. It can be seen that, the maximum hardness values were obtained because of the full dynamic recrystallization caused by FSP, in agreement with previous observations [28]. On the other hand, raising the traverse speed above 64 mm min$^{-1}$ slightly affected the hardness values. It is also noticed that the maximum hardness values were achieved at highest traverse speed. Higher traverse speed caused the decrease in peak temperature due to less heat exposure, therefore lowering the rate of transformation rate to martensite phase.
However, the increased relative linear motion at higher traverse speeds increased the cooling rate in addition to restraining grain growth. Studying both effects; rotation and traverse speed, \textit{FSP/1200/93} treatment achieved relatively highest hardness values.

![Graphs showing hardness variations](image)

**Figure 14.** (a) Average hardness at 93 mm min$^{-1}$ and different rotation speeds, (b) average hardness at 800 rpm and different transverse speeds.

**Figure 15.** Hardness values at 93 mm min$^{-1}$, air cooling, and different rotation speeds showing (a) hardness distribution under air cooling, (b) comparison between FSP-Only and FSP-Air.
The effect of air blowing on the hardness was studied for different rotational speeds and for different traverse speeds. Figure 15(a), indicates that the hardness values for rotation speeds of 800, 1000, and 1200 rpm were 263, 252, 277HV5, with enhancement 75.1%, 68.1%, 85%, respectively relative to the as-cast alloy. The highest average hardness was again achieved at 1200rpm. This behavior is similar to that without air blowing in figure 14(a). The effect of air blowing is illustrated in figure 15(b) where FSP-Air appeared to follow the same pattern as FSP-Only but with higher hardness values at 1000 and 1200 rpm. This increase in hardness was probably due to the higher percentage of $\beta'$ martensite phase and finer grains.

The hardness was also affected by traverse speed under air blowing condition as shown in figure 16(a) where the average hardness values at 800 rpm, and traverse speeds of 40, 64, and 93 mm min$^{-1}$ were 259, 250, 263 HV5, achieving enhancement of 72.3%, 66.9%, 75.2%, respectively relative to as-cast alloy. Maximum hardness values were achieved by FSP800/93/Air. However, the comparison between FSP-Only and FSP–Air in figure 16(b) shows that the maximum effect of air blowing was achieved at the slowest traverse speed of 40 mm min$^{-1}$.

The hardness distribution in the FSPed zones was taken in the center of the samples in the transverse direction and in the depth directions, as plotted in figure 17. The results indicate that FSP increased the hardness in comparison with that of the as-cast alloy and that a higher hardness is obtained in the region where the pin rotates in both transverse direction and in-dept directions.
4. Conclusions

Surface treatment of Nickel-Aluminum Bronze alloy using friction stir processing was successfully achieved using carbide tip tool under different process parameters. The effect of process parameters including rotational speeds, traverse speeds and using forced air blowing on the temperature evolution, microstructure and hardness have been investigated.

1. It was found that the cast structure composed of the Widmanstätten morphology of NAB alloy- consisting of coarse $\alpha$ grains, $\beta'$ martensitic phase, and $\kappa$-phase with eutectoid phase $\alpha + \kappa_{III}$ at the boundary of coarse $\alpha$ grains- transformed after FSP to $\beta'$ martensite phase and recrystallized fine grains with $\beta'$% increasing with rotational speed.

2. With decreasing rotation speed or increasing traverse speed, finer grains and lower $\beta'$ phase were achieved. The same was achieved with application of air blowing.

3. Temperature profile measurements indicate same peak temperature from tool entry point till before the tool exit. At tool exit point the peak temperature was relatively lower.

4. Application of air blowing raised the cooling rate at all points along the FSP pass. It resulted in more homogeneous and higher hardness values compared to the case without air blowing.

5. FSP treatment significantly enhanced the average hardness of the NAB alloy in comparison with the as-cast with an increase of about 100%.

6. Increasing the tool rotation speed from 800 rpm to 1200 rpm had a slight effect on the hardness while increasing the tool transverse speed from 40 to 93 mm min$^{-1}$ had a more sensitive effect.

7. The maximum hardness reached 280 VHN for 1200/93/Air and 270 VHN for 1200/93 while the as cast hardness was 150 VHN.

Figure 17. (a) Hardness distribution across SZ in as cast and after FSP and (b) effect of Hardness distribution in the in-depth SZ direction.
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