Characterization of AZ91/alumina nanocomposite produced by FSP

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**A B S T R A C T**

The microstructures, wear property and micro-hardness of AZ91 Mg alloy/alumina particle reinforced nano-composite produced by friction stir processing (FSP) were investigated. The initial microstructures of the AZ91 were composed of irregularly distributed \( \alpha \)-phases \( (\text{Al}_12\text{Mg}_{17}) \), while the FSPed specimens were characterized by the homogeneous distribution of alumina particles, the recrystallized grain structure and the dissolution of \( \alpha \)-phase. The results showed an improvement in the hardness, wear property of the FSPed zone as results of more grain refinement and pinning effect of nano-alumina particles as compared to those of the base metal. The hardness of the FSPed zone was a higher and more homogeneously distributed and the wear resistance as evaluated by Dry sliding wear tests, was superior, as compared with the base metal.

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1. Introduction

The magnesium alloys, showing admissible mechanical properties and considerable weight saving, have fueled research activities recently [1]. Magnesium alloys are used instead of aluminum and steel in the aerospace and automobile industries and instead of plastic in the electronic and computer industries for their weight saving and good thermal and electrical conductivity [1]. However, Mg alloys have not traditionally been used for high performance applications due to their low mechanical properties and wear resistance. Therefore, some processes were investigated to produce magnesium based composites in order to improve the mechanical properties and wear resistance [1–12]. In FSP of magnesium alloys, for enhancing the mechanical properties, many attentions are paid on AZ31 alloy [2–6]. Meanwhile, working on AZ91-base composite manufacturing by FSP is limited due to the difficulties in FSP of AZ91 Mg alloy and lack of AZ91 sheet form. Therefore, researchers have used different casting methods for manufacturing AZ91/ceramic particle composites [8–11].

Since in certain applications, lightweight alloys and composites are subjected to sliding motion, sliding wear is also an important consideration in material processing. For most of the applications the components require surface modifications as magnesium alloys exhibit a poor wear resistance [13]. The FSP is expected to be efficient for the structural materials to confer special mechanical properties, especially higher wear resistance, on the stirred zone (SZ). Lee et al. [14] have added the SiC particles in the friction stir welding of magnesium alloy. They reported an improvement in the hardness and wear property of the weld zone. Chen and Alpas [15] by investigation of the wear properties of AZ91 magnesium alloy identified two main wear regimes, namely a mild wear regime and a severe wear regime. Based on Hiratsuka et al, [16] investigations on dry sliding wear of pure magnesium, magnesium exhibited an oxidation wear mechanism when tested in air.

In this study, aluminum oxide is used as reinforcement. Aluminum oxide possesses strong ionic interatomic bonding giving rise to its desirable material characteristics. It can exist in several crystalline phases which all revert to the most stable hexagonal alpha phase at elevated temperatures. This is the phase of particular interest for structural applications. Alpha phase alumina is the strongest and stiffest of the oxide ceramics. High hardness, excellent dielectric properties, refractoriness and good thermal properties make aluminum oxide as a material for use in a wide range of applications. Nano-sized \( \text{Al}_2\text{O}_3 \) particles were dispersed into AZ91 magnesium alloy in order to reveal the effect of FSP on the microstructure, hardness and wear properties of the fabricated AZ91/\( \text{Al}_2\text{O}_3 \) nano-composite layer. Effects of the FSP parameters such as tool geometry and rotational and traverse speeds on defects formation and quality of the AZ91/\( \text{Al}_2\text{O}_3 \) surface nano-composite are also investigated. Optimum FSP parameters are revealed based on powder dispersion, microstructure, micro-hardness, and wear properties.
2. Experimental procedure

Commercially available nano-sized (30 nm) $\text{Al}_2\text{O}_3$ particles with the purity of 99.9% were used in volume fraction of 8% to produce nano-composite layer on the surface of AZ91. The $\text{Al}_2\text{O}_3$ powder was filled into a groove of $0.8 \text{ mm} \times 2 \text{ mm}$ machined on the 3 mm thick AZ91 plate before the FSP was carried out. The as-received AZ91 composition (in wt.%) was Al, 9.1; Zn, 0.68; Mn, 0.21; Si, 0.085; Cu, 0.0097; Ni, 0.001; Fe, 0.0029 and Mg, bal. The circular and square tools were used in this work. Tools were made of hardened H13 tool steel. Tools consisted of a pin with 5 mm in diameter, 3 mm in height, and a shoulder with 15 mm in diameter.

The FSP tool was rotated in 900 and 1200 rpm in the clock-wise direction. The traverse speed was changed from 40 to 80 mm/min. The tilted angle was $3^\circ$. Transverse sections of the FSPed samples were cut for metallographic investigations. Surfaces were prepared by standard metallographic techniques and etched with a solution of 5 ml acetic acid, 6 g picric acid, 10 ml water, 100 ml ethanol, 5 ml HCl and 7 ml nitric acid for 1–2 s.

The distribution of the $\text{Al}_2\text{O}_3$ particles and microstructure of the FSPed samples was considered using optical and scanning electron microscopy (SEM). The micro-hardness was measured using a micro-Vickers hardness tester with a load of 200 g for 15 s. Dry sliding wear tests of the specimens were performed by a pin-on-disc test machine. Specimens were prepared by wire cut in $5 \text{ mm} \times 3 \text{ mm} \times 10 \text{ mm}$ dimension from as-cast AZ91 and FSPed specimens. A load of 50 N was applied with slide speed of 1 mm/min for a distance of 500 m.

3. Results and discussion

Typical optical micrograph of the cross section the FSPed specimen by the square type tool, 900 rpm rotational speed and 80 mm/min traverse speed is shown in Fig. 1a. The figure shows that there are no defects and porosities in the cross section of the specimen but there is agglomeration of the $\text{Al}_2\text{O}_3$ particles. Agglomeration of alumina particles is shown in Fig. 1b in higher magnification. Particle distribution in the specimen FSPed in a high traverse speed is not proper.

Fig. 2 shows the cross section of the specimen produced by the square tool and rotational and traverse speeds of 900 rpm and 40 mm/min, respectively. From Figs. 1a and 2, one can see that the particle distribution in the specimen fabricated by 40 mm/min traverse speed is better than that produced by 80 mm/min. High traverse speeds decreases the rotational speed/traverse speed ($\omega/v$) ratio and the stirring of material. Therefore, alumina particles are not mixed well in the AZ91 matrix. Consequently, in order to achieve a better distribution of nano-sized alumina particles, the traverse speed should be decreased or the rotational speed increased. Totally it can be concluded that the higher $\omega/v$ ratio results in a better particle distribution.

Fig. 3 shows the cross section of the specimen produced by the circular tool and rotational and traverse speeds of 900 rpm and 40 mm/min, respectively. As can be seen, despite the square tool, the circular tool does not result in a uniform distribution of par-
Fig. 3. Optical macrograph of cross section of FSPed specimen by the circular tool, rotational speed: 900 rpm, traverse speed: 40 mm/min.

Particles in the traverse speed of 40 mm/min. Elangovan et al. [17] reported that the square tool produces a pulsating stirring action in the material flow; however there is no pulsating action in the circular tool. Indeed, pulsating stirring action intensifies the mixing of particles in the matrix. Therefore, acquiring a uniform distribution of particles by circular tool is very difficult even in low traverse speeds.

3.1. Microstructure

Fig. 4a and b shows the optical and SEM micrographs of the as-received AZ91 magnesium alloy. Grain size (GS) in the AZ91 is about 150 μm. Microstructure of this alloy is characterized by a coarse eutectic \( \beta - \text{Mg}_17\text{Al}_{12} \) network distributed at the grain boundaries, which is a typical microstructure for as-cast AZ91. The maximum solid solubility of aluminum in magnesium is as high as 12.9 wt.% at eutectic temperature of 437 °C [18]. However, the equilibrium concentration of aluminum in magnesium at room-temperature is about 1.5 wt.%. When aluminum-supersaturated magnesium solid solution is cooled, \( \beta - \text{Mg}_17\text{Al}_{12} \), with a stoichiometric composition of 44 wt.% Al will precipitate. The energy dispersive spectroscopy (EDS) analysis results are summarized as follows. Inside the grains in the as-received AZ91 magnesium matrix contains ~4.5 wt.% Al (point c in Fig. 4b). This value is higher than the room-temperature equilibrium concentration of aluminum in magnesium, indicating that the as-received AZ91 is a little supersaturated. On the other hand, point (d) was composed of ~40 wt.% Al and ~60 wt.% Mg, suggesting that the white phase in Fig. 4b is \( \beta - \text{Mg}_17\text{Al}_{12} \).

The SZ of the FSPed AZ91 is characterized by fine grains (~15 μm) (Fig. 5a). No fine particles (related to the \( \beta \) phase) were found within the grain interior by SEM examinations even under a high magnification. The coarse eutectic of \( \beta - \text{Mg}_17\text{Al}_{12} \) network in the as-received AZ91 disappeared after FSP [19]. EDS analysis in point b of Fig. 5a (Fig. 5b) indicated that the interior of grains contains ~8.5 wt.% Al. This value is much higher than that in the as-received alloy (5 wt.% Al), indicating significant dissolution of the eutectic \( \beta - \text{Mg}_17\text{Al}_{12} \) phase into magnesium matrix during FSP. From Fig. 5, it is clear that FSP resulted in significant breakup and dissolution of the coarse eutectic \( \beta - \text{Mg}_17\text{Al}_{12} \) which was previously distributed at the grain boundaries. EDS analysis in the grain boundary of FSPed specimen (Fig. 5c) indicates that the amount of Al in the grain boundaries are almost similar to that in the interior of grains.

Based on the Mg–Al phase diagram, a heating temperature of 437 °C causes the dissolution of the eutectic \( \beta - \text{Mg}_17\text{Al}_{12} \) phase into the magnesium matrix. For Mg–Al alloys, it takes up to ~40 h to achieve the complete dissolution of the eutectic \( \beta - \text{Mg}_17\text{Al}_{12} \) phase due to low diffusion rate of aluminum in magnesium matrix [20]. In friction stir welding/processing, heating and cooling rates are quite high. For example, for the A356 aluminum alloy, the duration that the temperature stays above 200 °C during the FSP is only 25 s [21]. It seems impossible to achieve a complete dissolution of the eutectic \( \beta - \text{Mg}_17\text{Al}_{12} \) phase in such a short period of time for a conventional thermal cycle. However, severe plastic deformation...
in the SZ with a strain rate of $10^{0}–10^{2}$ s$^{-1}$ and a strain of up to 0.4% [22,23] in the friction stir welding/processing facilitates significantly the dissolution of the $\beta$-Mg$_{17}$Al$_{12}$ phase, thereby generating an aluminum-supersaturated solid solution. FSPed AZ91 alloy with fine and homogenous grain structure is expected to exhibit an improved ductility and formability due to the dissolution of coarse precipitated $\beta$ phase.

While the average GS is about 150 $\mu$m in the base metal, it is about 20 $\mu$m in the SZ that is much finer. Homogenous microstructure, including very fine and equiaxed grains, is obtained via dynamic recrystallization.

Fig. 6a shows the boundaries of the SZ in the FSPed specimen in 50 mm/min traverse speed. It can be seen that the width of the thermomechanically affected zone (TMAZ) in the side of the SZ is smaller than that in the bottom of the SZ.

Fig. 6b and c shows the SEM micrographs of bottom TMAZ in low and high magnifications, respectively. As could be seen, grains in this zone are elongated and continuously aligned in a direction and severely squeezed. Stretched $\beta$-Mg$_{17}$Al$_{12}$ phases are clearly seen in these figures. In particular, with regard to the behavior of the $\beta$-phase, the TMAZ is characterized by a lower fraction of $\beta$-phase, while the SZ is characterized by the disappearance of the $\beta$-phase, due to the dissolution into the matrix. Subjected strain in TMAZ besides the high temperature which is experienced by TMAZ causes the slight dissolution of $\beta$-phase in TMAZ.

Fig. 7 showing the bottom TMAZ width in the specimens produced by the square tool in 50 and 80 mm/min traverse speeds exhibits that the width of bottom TMAZ in 80 mm/min traverse speed is wider than that in 50 mm/min traverse speed. Decreasing the traverse speed increases the heat input and causes more softening in material. More softening of material eases the movement of tool in the process path which results in a decrease in the TMAZ width. It is better to say that the $\omega/v$ ratio is the dominant factor in the discrimination of TMAZ width. The high the $\omega/v$ ratio, the more the material softening and the less the TMAZ width. Fig. 8 shows the effect of $\omega/v$ ratio on the width of bottom and side TMAZs.

From Fig. 6a, one can see that the TMAZ width in the side is very smaller than that in the bottom. The quantitative values are shown in Fig. 8. This may be because of better heat transfer in sides rather than in bottom. Moreover, the type of contact of tool pin with the base metal in the side is linear but in the bottom is planer. So, the amount of plastic deformation in the bottom is higher than that in the side.

Each weld zone experiences a different temperature and consequently a temperature gradient must have produced throughout the weld zone. Since the experienced temperature by heat affected zone (HAZ) is very lower than 400 °C [24,25] and no plastic deformation occurs in this zone, course $\beta$-Mg$_{17}$Al$_{12}$ phase was not dissolved in the HAZ. Therefore, there is no noticeable difference between the HAZ and base metal microstructures.
Presence of uniformly distributed Al_2O_3 particles in the composite layer leads to a severe grain refinement. Besides the grain refinement due to the severe plastic deformation occurred in SZ, the grain refinement of matrix in the case of FSP with Al_2O_3 particles can be attributed to the coupled effects of: (i) capability of Al_2O_3 particles to nucleate magnesium grains during recrystallization and (ii) restricted growth of recrystallized magnesium grains as a result of presence of Al_2O_3 particles (pinning effect of Al_2O_3 particles) [26]. The fundamental principles behind the ability of inclusions in the metallic matrix to nucleate recrystallized grains and to inhibit grain growth have been already established [1] and will not be discussed here. Relatively higher tendency of clustering of nano-sized reinforcing particles might be the plausible reason of limited grain refinement effect in the case of composite with nano-sized Al_2O_3 [1].

Dispersion of the nano-sized reinforcements in a uniform manner is a critical and difficult task [27]. Fig. 9 shows SZ of the specimen produced by square tool and rotational speed of 900 rpm and traverse speed of 80 mm/min. In this figure, dark regions are agglomerated nano-sized alumina particles that were not...
distributed well in the field. It is clearly seen that the grains are finer where the alumina clusters exists and much finer where the particles are separately dispersed.

Assuming that the alumina particles are distributed uniformly in the AZ91 matrix, the theoretical GS of the composite layer can be calculated by the Zener–Holoman parameter, Eq. (1).

\[ dz = \frac{4r}{3V_f} \] (1)

where \( r \) and \( V_f \) are the radius and volume fraction of clustering \( \text{Al}_2\text{O}_3 \) particles, respectively [27].

The measured Mg matrix GS can be related to the Zener limiting grain size. This matter suggests that the presence of finely dispersed nano-sized \( \text{Al}_2\text{O}_3 \) particles can limit the grain growth and result in an ultrafine GS in the AZ91 matrix. According to Eq. (1), decreasing the radius or increasing the volume fraction of the reinforcing particles, decreases the GS of the produced composite. For instance, assuming all of the \( \text{Al}_2\text{O}_3 \) particles are separated, the GS should be 250 nm, where the radius and volume fraction of the alumina particles in the AZ91 matrix are 15 nm and 8%, respectively. However, from Figs. 9–11 it can be seen that the grains are very larger than that estimated GS (250 nm). Hence, the existence of local clustering of alumina particles is unavoidable and not all nano-sized particles can restrict the grain growth. Assuming that the alumina clusters’ size is \( 7 \mu \text{m} \) the GS will be the \( \sim 60 \mu \text{m} \). This inconsistency between \( 60 \mu \text{m} \) and 250 nm reveals that some alumina particles are distributed separately and some are agglomerated.

Fig. 10 shows the microstructure of the FSPed specimens with alumina particles at 900 rpm rotational speed and 40 mm/min (a), 50 mm/min (b), 80 mm/min (c) traverse speeds, respectively. It is observed that although there is much grain refinement after FSP, the grain structure is clearly more equiaxed and more homogenized. FSP refined the average GS from about \( \sim 150 \) to \( \sim 5–10 \mu \text{m} \) by statistical analysis on 300 grains. From Fig. 11, it can be inferred that decreasing the traverse speed leads to smaller GS and better particle distribution. Fig. 11 shows the effect of rotational speed on the microstructure of FSPed specimens with alumina particles. It is seen that increasing the rotational speed or decreasing the traverse speed has the same effect on the grain refinement and particle distribution. Stirring action of tool pin in SZ increases as the rotational speed increases or traverse speed decreases. Higher stirring of material results in better distribution of particles in matrix. Totally it can be mentioned that the higher \( \omega/v \) ratio leads to better distribution of nano-sized particles (smaller alumina clusters’ size) and consequently smaller GS of composite layer.

![Fig. 8. Effect of the \( \omega/v \) ratio on the TMAZ width in side and bottom of the SZ.](image)

![Fig. 9. Optical micrograph of the FSPed specimen with \( \text{Al}_2\text{O}_3 \) particles. Rotational speed: 900 rpm, Traverse speed: 80 mm/min.](image)

![Fig. 10. Effect of traverse speed on GS and alumina cluster size of FSPed specimens; traverse speed: (a) 40 mm/min, (b) 50 mm/min and (c) 80 mm/min. The rotational speed in all FSPed specimens was 900 rpm.](image)
Fig. 11. Effect of rotational speed on GS and alumina cluster size of FSPed specimens; rotational speed: (a) 900 rpm, (b) 1200 rpm. The traverse speed in all FSPed specimens was 40 mm/min.

Fig. 12. Effect of the traverse and rotational speeds on the microhardness of SZ.

Fig. 13. Hardness profiles of the specimens produced with one- and two-pass FSP. Rotational speed: 900 rpm, traverse speed: 40 mm/min.

Fig. 14. Wear rate in the base metal and the friction stir processed specimens in different conditions.
Fig. 15. SEM micrographs of the worn surfaces in the (a) base metal, (b) one-pass FSPed specimen with square tool and (c) two-pass FSPed specimen square tool. (d)-(e) The higher magnifications of (a)-(c), respectively.
3.2. Microhardness study

Fig. 12, showing the hardness profiles across the FSPed sample away from the center of the SZ, exhibits that the average hardness increases as the traverse speed decreases or the rotational speed increases. Darras et al. [6] and Morisada et al. [2] reported that increasing the rotational speed or decreasing the traverse speed decreases the hardness value. This result is expected since the peak temperature increases as the rotational speed increases or the traverse speed decreases. High temperature leads to more softening due to the grain growth. According to Darras et al. and Morisada et al. results it could be mentioned that the higher $\omega/v$ ratio results in higher GS and consequently lower hardness. In the case of FSP without reinforcing particles the dominant factor in determination of GS is heat experienced by the material during recrystallization. However, in the case of FSP with reinforcing particles the circumstances change. In this work, in the case of presence of alumina particles, it is concluded that the higher $\omega/v$ ratio causes better particle distribution and smaller alumina cluster size which results in a smaller grain size. According to the well-known Hall–Petch relationship, increase in GS decreases the hardness value. Therefore, more refinement and higher hardness can be achieved at higher $\omega/v$ ratios.

Lee et al. [14] reported that the hardness of AZ91 magnesium alloy reached to 98 HV by addition of 10% SiC particles. In the present study, the maximum hardness value of SZ in reached to about 110 HV.

Fig. 13 shows the effect of second FSP pass on the hardness of composite layer produced with square tool. The second FSP pass homogenizes the particles’ distribution, decreases the alumina clusters and consequently decreases the grain size. Therefore the hardness increases by the second FSP pass. Furthermore, the hardness profile of the specimen produced with two-pass FSP is smoother than that in the one-pass FSPed specimen. Uniform distributed alumina clusters results in a uniform hardness profile.

3.3. Wear properties

Wear rate of the base metal and the specimens FSPed in different conditions, shown in Fig. 14, reveals that the wear resistance is severely increased in the FSPed specimens. While the wear rate of the base metal is about $17 \times 10^{-5}$ (mm$^3$/Nm), it is less than $5 \times 10^{-5}$ (mm$^3$/Nm) in the FSPed specimens with nano-sized $\text{Al}_2\text{O}_3$ particles. On the other words, addition of nano-sized alumina particles decreased the wear rate more than three times. Habibnejad et al. [28] reported that wear rate reduced from 2.5 to $2 \times 10^{-5}$ g/m by addition of 2% $\text{Al}_2\text{O}_3$ in the matrix of AZ31 magnesium alloy (sliding rate was 0.5 m/s). Also, Lim et al. [29] reported that, at the sliding rate of 1 m/s, the wear rate in the magnesium decreased from $10 \times 10^{-4}$ to $8 \times 10^{-4}$ mm$^3$/m by addition of 8% $\text{SiC}$ (14 $\mu$m) particles. In a better condition, Lee et al. [14] reported that addition of 10% $\text{SiC}$ particles in AZ91 matrix decreases the wear loss from 5.5 to 2 mm$^3$/Nm (near to three times).

Owing to higher hardness value, the specimens with $\text{Al}_2\text{O}_3$ particles have higher wear resistance compared to the base metal. Also, it can be mentioned that presence of the alumina particles decrease the direct load between the specimen surface and the disk. In fact, load bearing component action of the hard alumina particles decreases the direct load [30]. Decreasing the direct load decreases the wear rate [15].

Better distribution of $\text{Al}_2\text{O}_3$ particles, which leads to a higher hardness value in the composite layer, can help the wear resistance. As shown in Fig. 14, the wear rate in the FSPed specimen with square tool is lower than that in the FSPed specimen with circular tool. Also, the wear rate in the two-pass FSPed specimen is lower than that in the one-pass FSPed specimens.

SEM micrographs of the worn surfaces of the base metal and one- and two-pass FSPed specimens with square tool are shown in Fig. 15. Existence of the straight deep plows in Fig. 15a shows the severe adhesive wear occurred on the surface of the base metal. It seems that the brittle and coarse Mg$_7$Al$_{12}$ phase does not impressively act as an obstacle to the wear [14]. Additionally, existence of loose metallic debris on the worn surface of the base metal (Fig. 15a and d) indicates that the mild abrasive wear also occurred. Even though the metal-to-metal contact between the magnesium surface and the steel disk is expected to result in just adhesive wear, it seems that resulted in a combination of adhesive and abrasive wear [13]. Severe adhesive wear, which is characterized by massive surface damage, transfers the removed material from the surface of the AZ91 base metal to the steel disk. These transferred materials might have oxidized during sliding [13] and hence the
large metallic debris particles, which are identifiable by the naked eye on the disk surface, are produced [15]. These debris particles cause the abrasive wear and increase the wear rate in the base metal.

It is shown in Fig. 15 that the wear mechanism in the FSPed specimens with Al2O3 particles is different from that in the base metal. The equiaxied grains and homogeneously distributed Al2O3 particles positively affect the wear resistance [14]. Distributed alumina particles in the AZ91 matrix act as a barrier to the wear and prevent from the severe adhesive wear and consequently decrease the wear rate. Decrease in direct contact load between the magnesium surface and steel disk due to the presence of alumina particles changes the severe wear to mild wear. Therefore, the dominant wear mechanism in these specimens is abrasive wear by the loose debris particles. Generation of the loose debris particles was due to both removing and oxidizing the AZ91 matrix, and the alumina particles pulled out from the AZ91/Al2O3 composite layer during the sliding. Debris particles are clearly shown in the worn surfaces of FSPed specimens with Al2O3 particles (Fig. 15e and f).

It is worth mentioning that in the one-pass FSPed specimen delamination of substrate layers of AZ91 matrix is also seen in pass number. It is found that, in the case of presence of nano-sized FSP parameters such as traverse speed, rotational speed and FSP size and the particle distribution pattern are mainly affected by the surface layer were achieved by square tool and rotational speed of tle precipitates. The optimum conditions for producing the sound produces fine and homogenous grain structure with dissolved brit- ment. FSP is an effective microstructural modification process that the delamination [31].

Al2O3 particles and higher hardness value in the two-pass FSPed spec-

particles positively affect the wear resistance [14]. Distributed alumina particles not the heat experienced by the material. Since the better particle distribution can be achieved in higher \(a/v\) ratios and subsequent FSP passes, grain size reduces and hardness increases as the \(a/v\) ratio and FSP pass number increases. Furthermore, FSP of AZ91 with alumina particles increases the wear resistance significantly and changes the wear mechanism from severe wear in the as-received AZ91 to mild wear in the FSPed specimens.

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References