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An investigation on the hardness and corrosion behavior of MWCNT/Mg composites and grain refined Mg

N. Saikrishna[a,e], G. Pradeep Kumar Reddy[b], Balakrishnan Munirathinam[c,f,*], Ravikumar Dumpala[d], M. Jagannatham[c], B. Ratna Sunil[a,*]

[a] Department of Mechanical Engineering, Rajiv Gandhi University of Knowledge Technologies (AP-IIIT), Nuzvid 520201, India
[b] Department of Mechanical Engineering, Vignana Bharathi Institute of Technology, Hyderabad 501301, India
[c] Department of Metallurgical and Materials Engineering, Indian Institute of Technology Madras, Chennai 600036, India
[d] Department of Mechanical Engineering, Visvesvaraya National Institute of Technology (VNIT), Nagpur 440010, India
[e] Reactor Engineering Division, Bhabha Atomic Research Centre, Mumbai - 400085, India
[f] Department of Materials Science and Engineering Department, Delft University of Technology, Delft, Netherlands

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Abstract

In the present work, multi walled carbon nanotubes (MWCNT) reinforced magnesium (Mg) matrix composite was fabricated by friction stir processing (FSP) with an aim to explore its mechanical and electrochemical behavior. Microstructural observations showed that the thickness of the produced composite layer was in the range of 2500 μm. FSP resulted uniform distribution of CNT near the surface while agglomerated layers in the subsurface. Grain refinement of Mg achieved by FSP improved the hardness but significant enhancement in the hardness value was observed for FSPed MWCNT/Mg composites. Potentiodynamic polarization studies revealed that the increase in corrosion current density was observed for MWCNT/Mg composite compared with grain refined Mg and pure Mg, implying the significance of secondary phase (MWCNT) in decreasing the corrosion resistance of the composite.

Keywords: Friction stir processing; MWCNT/Mg composite; Hardness; Basal texture; Corrosion resistance.

1. Introduction

Magnesium (Mg) and its alloys are now attracting a great attention for wide range of applications due to their promising properties such as light weight, high specific strength and good machinability [1,2] Composites are a class of materials which are being well considered for several structural applications in various industries [3]. In this context, as modern engineered materials, Mg based composites can offer hybrid properties compared with their counter parts. Liquid state methods such as stir casting, squeeze casting, laser melting etc. are well known routes in producing the metal matrix composites [3]. However, handling liquid magnesium, formation of oxides, reaction between Mg and secondary phases and distribution of the dispersing phase are the major issues which make the liquid state methods as complex in developing Mg based composites.

Recently, friction stir processing (FSP), a solid state processing method has arrived as a promising tool to develop surface composites without melting the matrix material [4]. In addition, FSP also results grain refinement in the processed region and, therefore, structure dependent properties also can be altered. Mishra et al. initially demonstrated producing AA5083-SiC composites using FSP [5]. Later, several material systems were considered to develop surface composites by FSP as reported in the literature [6,7]. Magnesium and Mg alloy based composites reinforced with several secondary

* Corresponding authors.
E-mail addresses: blkrish88@gmail.com (B. Munirathinam), bratnasunil@rgukt.in (B.R. Sunil).

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phases including SiO_2, B_4C, TiC, SiC, Al_2O_3, carbon fibers, graphite and hydroxyapatite were successfully produced by FSP [7–10]. Recently, carbon nanotubes (CNT) have attracted the attention of the material scientists due to their extraordinary mechanical properties [11]. By and large, CNT are utilized as reinforcement in several metal matrix composites (MMCs) [12]. Morisada et al. [13] successfully introduced multi walled carbon nanotubes (MWCNT) into AZ31 Mg alloy by FSP and investigated the effect of process parameters and presence of MWCNT on the mechanical behavior and microstructure particularly grain size of the composite. Mertens et al. [14] developed composites by introducing carbon fibers into AZ31 and AZ91 Mg alloys by FSP and reported improved mechanical properties for the composites due to the presence of carbon fibers. Yang and Schaller [15] produced Mg-CNT composites by mechanical alloying followed by sintering route and demonstrated increase in high temperature shear modulus by 20% due to the addition of CNT. Similarly, Mg-CNT composites produced by hot pressing exhibited improved hardness, compressive and bending strength as reported in the literature [16]. However, it is worth noting the deteriorating corrosion resistance due to the presence of CNT in Mg-CNT composites developed by disintegrated melt deposition technique [17]. Information on developing MWCNT/Mg composites by solid state methods in particular by FSP is limited in the literature. From the structural application perspective, it is of cardinal importance to investigate their mechanical as well as corrosion behavior. In the present study, an attempt has been made to fabricate MWCNT/Mg composites by FSP and the effect of MWCNT on the microstructure, microhardness and corrosion behavior was investigated.

2. Experimental details

Cast billet of pure magnesium of composition 0.003% Al, 0.001% Zn, 0.002% Fe, 0.008% Mn and remaining being Mg (by wt.%) was purchased from Exclusive Magnesium, Hyderabad. Purified MWCNT synthesized by arc discharge method were used in the present work. The detailed synthesis and purification processes of carbon nanotubes can be found elsewhere [18]. Pure magnesium sheets of 10 mm thickness were sliced from the Mg cast billet across the cross section (100×80 mm^2) using a power hack saw. Hence, the size of Mg sheets used in the present work is $100 \times 80 \times 10$ mm^3. FSP was carried out using an automated universal milling machine (Bharat Fritz Werner Ltd., India). FSP tool made of H13 tool steel was used to process the samples. FSP tool has a shoulder diameter of 20 mm and a tapered pin having root diameter of 3 mm, end diameter of 1 mm with 3 mm length. Initially, trial experiments were conducted to optimize the process parameters to get defect free stir zone. Then, FSP was carried out at optimized process parameters with a tool travel speed of 100 mm/min at a tool rotational speed of 1100 rpm. The penetration depth of 3.1 mm was adopted such a way that the tool shoulder touches the surface of workpiece. The sample was named as FSPed Mg after friction stir processing.

In order to produce MWCNT reinforced Mg matrix composites, rows of double line zig-zag blind holes of depth 2 mm and diameter 1 mm were produced on the surface of the sheets maintaining 25 mm distance between the rows as shown in Fig. 1. These holes were filled with purified MWCNT. Pinless FSP tool (with a flat shoulder diameter of 20 mm and a shoulder length of 25 mm) was used to close the holes to avoid the escape of MWCNT during the process as shown in Fig. 1b. Based on the preliminary trial experiments with different process parameters, better processed zone was observed at 1400 rpm with 25 mm/min travel speed. Therefore, friction stir processing was carried out (one pass) by completely overlapping on the region produced by pinless tool, at a tool rotational speed of 1400 rpm and traverse speed of 25 mm/min. The processed region is now distributed with MWCNT in Mg matrix due to the stirring action of FSP tool and material plastic flow and named as MWCNT/Mg. The entire surface of the workpiece was not processed to produce the composite. Only the regions with holes as shown in Fig. 1 were processed and developed the surface composite. Representative specimens were cut from the processed regions for the further investigations.

Specimens of size $25 \times 10 \times 10$ mm^2 were cut from the pure Mg (named as Mg), FSPed Mg and MWCNT/Mg samples and polished according to standard metallographic procedures by using different grades of emery papers. Then, disc polishing with alumina as abrasive agent was carried out on disc polishing machine followed by using polishing with diamond paste. At each level, the samples were cleaned with distilled water and dried. Picric acid solution was prepared as etching agent [19]. The samples were immersed in the etchant for 15 s, rinsed in distilled water followed by rinsing in ethanol and dried. Microstructural observations (Leica, Germany) were carried out on the cross section of the samples at different areas of interest and the images were acquired using the accompanied software. X-ray diffraction (XRD, X'Pert PRO, PANalytical) analysis was performed using an incident Cu Kα radiation ($\lambda = 1.5418$ Å), at a tube voltage 30 kV with a step size of 0.05 and a counting time of 20 s/step. Raman spectra was recorded using micro-Raman spectroscopy (Horiba Jobin Yvon, model HR800UV) instrument with He–Ne laser (632.81 nm) with an acquisition time of 5 s. Purified MWCNT were characterized using a Philips CM-12 TEM, with LaB_6 filament, operated at a voltage of 120 kV. Microhardness measurements (Omnitech, India) were carried out across the FSPed Mg and MWCNT/Mg samples and compared with pure Mg sample. During measuring, a load of 100 g was applied for 10 s (dwell period). The indents were placed across the FSPed regions.

Electrochemical studies were carried out in an electrochemical cell with a three-electrode configuration coupled to a potentiostat (Gill AC – ACM instruments, USA). Potentiodynamic polarization measurements were performed in 3.5% NaCl medium at a scan rate of 1 mV s^{-1}. During the measurements, graphite rod was used as the counter electrode and a saturated calomel electrode (SCE) was used as a reference electrode. An area of 1 cm^2 of the sample (considered as

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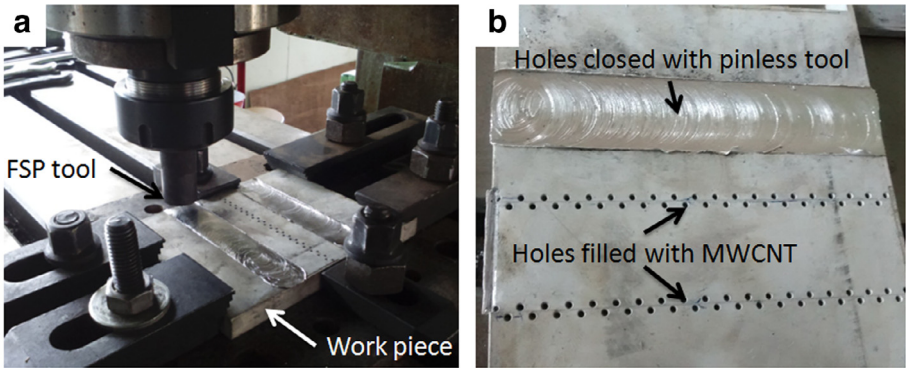

Fig. 1. (a) Photographs showing FSP process and (b) workpiece with CNT filled holes.

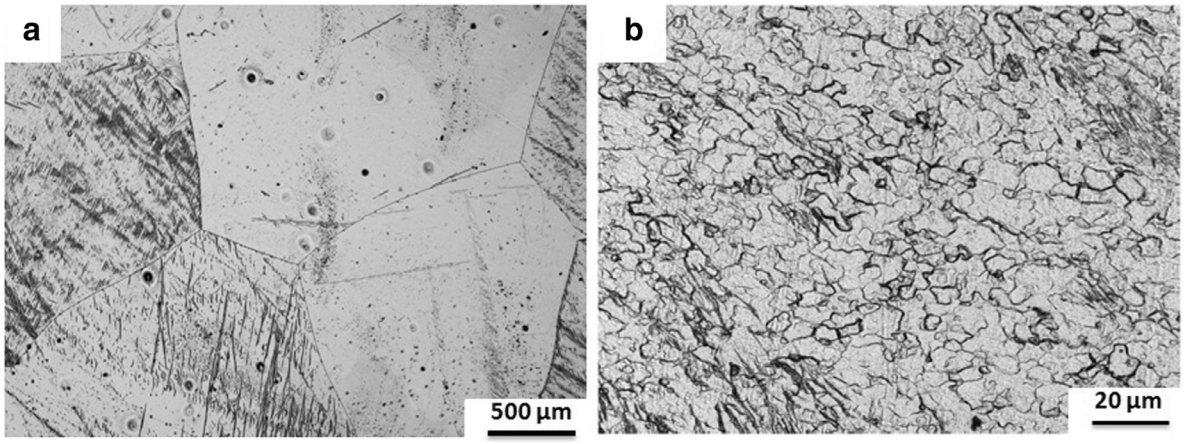

Fig. 2. Optical microscope images of (a) pure Mg and (b) FSPed Mg.

working electrode) was exposed to the electrolyte. All tests were conducted at room temperature (25 °C). Prior to the beginning of the polarization tests, the samples were exposed to the electrolyte to attain stable open circuit potential (OCP). From the polarization curves, corrosion current density and corrosion potential of the samples was measured using Tafel extrapolation and the corrosion rate (CR) was calculated using the following equation [20]:

$$\mathrm{CR(mpy)} = 0.129 * a * i_{\mathrm{corr}}/n\,D \quad (1)$$

where CR is the corrosion rate in mils per year, a is the molar mass (for magnesium 24.3 g mol^{-1}), i_{corr} is the corrosion current density (μA cm^{-2}), n is the valance (2) and D is the density (1.74 g cm^{-3}).

3. Results and discussion

Fig. 2 shows the optical microstructure of the starting Mg material and FSPed Mg. The pure Mg sheets cut from the cast billet have a grain size distribution from 500 to 1000 µm (Fig. 2a). After FSP, the grain size was observed as reduced to ≈5−10 µm as shown in Fig. 2b. Friction stir processing was reported to refine grain structure of Mg and its alloys and it is also clearly evident from the present work. TEM image of purified MWCNT used in the present study are shown in Fig. 3. The diameter of CNT is in the range of 18–23 nm and the length of the CNT is around 500 nm. The magnified TEM image Fig. 3b shows the multi walls of the CNT.

The high surface area of CNT tends to form agglomerates very easily and hence the distribution of CNT in metal matrix is a serious issue in developing composites by using melting practices. In the present work, CNT were observed as distributed within the solid state up to a depth of 2.5 mm in the stirred zone as shown in Fig. 4a. In the fabricated composite, the CNT were distributed as agglomerated layers. The layer like appearance signifies the flow of CNT along with metal flow during FSP. This type of distribution was resulted due to the flow of CNT as a group from each hole into the material. They could not have diffused deep into the Mg matrix because of which some regions were identified with agglomerated CNT. Thermo Mechanical Affected Zone (TMAZ) (Fig. 4(c)) was appeared with bimodal grains as usually observed in friction stir processed Mg [8,19,21]. Adjacent to TMAZ, heat affected zone (HAZ) has number of twins as shown in Fig. 4(d). Being a metal of *hcp* crystal structure, Mg necessarily needs twinning to initiate plastic deformation. Due to the applied load and the heat dissipation during FSP, several twins have been developed in the HAZ which is similar to the earlier report [21]. The CNT distribution at the retreating size is marginally higher compared

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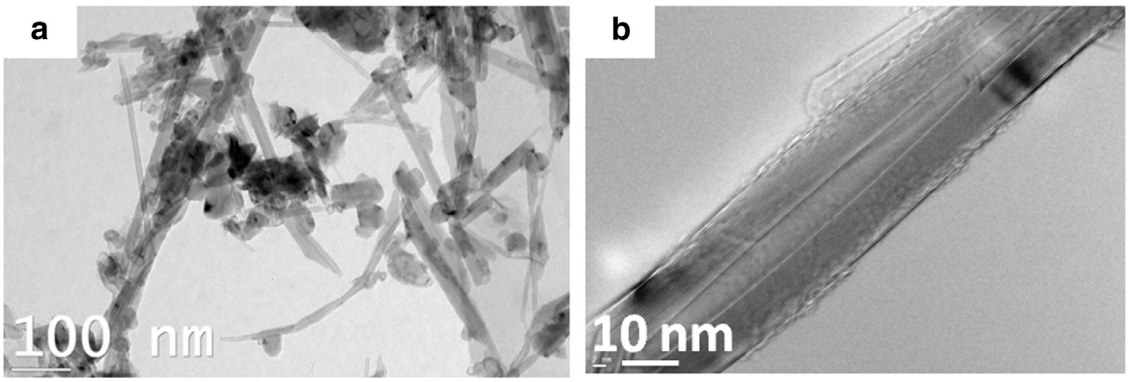

Fig. 3. TEM images of MWCNT: (a) low magnified and (b) high magnified image.

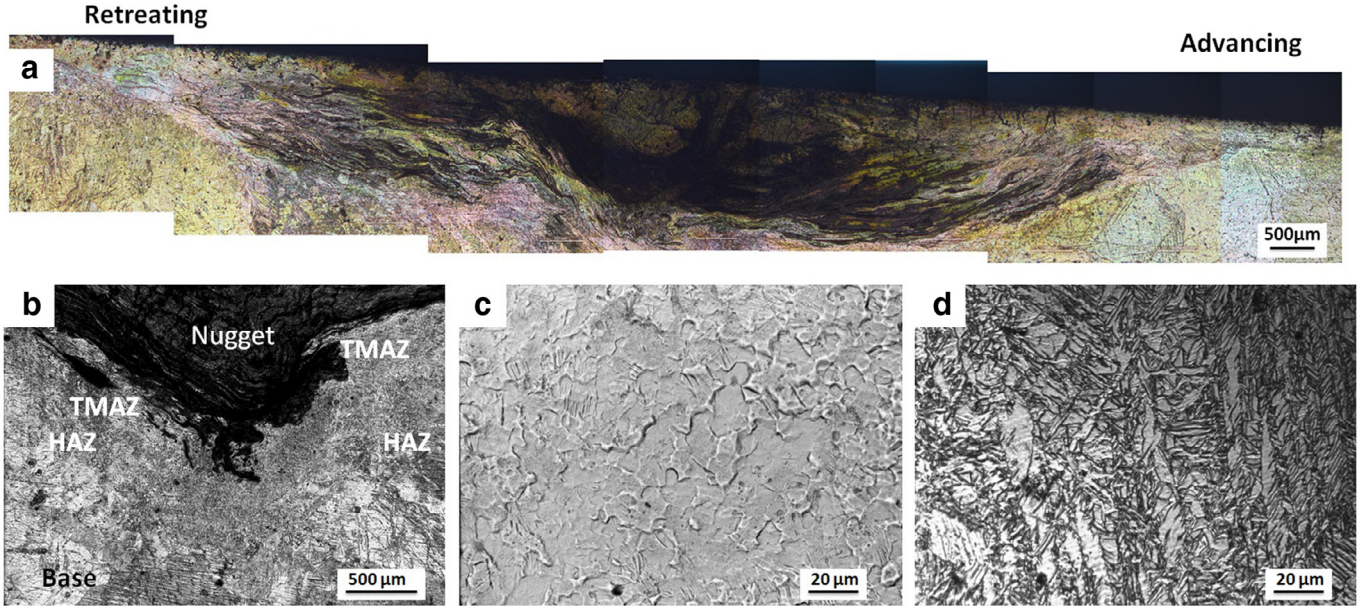

Fig. 4. Optical microscope images of MWCNT/Mg composites: (a) overall affected surface width and length observed at the cross section, (b) different zones at the cross section, (c) magnified region in TMAZ and (d) magnified region showing twins in HAZ.

with at the retreating side. It is obvious in FSP to observe such non-uniformity in the distribution of secondary phase at advancing side compared with retreating side due to the difference in the rate of material flow and the secondary phase when stirring is done by the tool pin [4].

Fig. 5a shows the XRD patterns of pure Mg, FSPed Mg and MWCNT/Mg. After FSP, preferred orientation (texture) of grains can be observed where (0002) reflection exhibits higher intensity than conventional $(10\bar{1}1)$ reflection. In principle, $(10\bar{1}1)$ reflection will be maximum in non-oriented grains of Mg. Remarkably, (0002) reflection prevails over $(10\bar{1}1)$ reflection indicating the strong basal plane texture. This preferred orientation is attributed to the complex stress states (shear and compression stress) arising from the rotation of the FSP tool pin and tool shoulder. During FSP, both heating and an intense plastic deformation can be seen on the surface, where the material shears along the surface of the pin column, generating the distribution of shear texture components of (0002) planes [22]. Furthermore, with addition of CNT, (0002) basal plane texture is still operative. However, XRD spectrum showed only peaks of Mg matrix due to the limited mass content of CNT. Nevertheless, CNT signature can be manifested from Raman measurement shown in Fig. 5b. From the Raman spectrum, the quality of the internal CNT can be established from the intensity ratio (I_D/I_G) of disordered D-band observed near 1330 cm^{-1} to the graphite G-band near 1580 cm^{-1} [23,24]. This ratio was comparatively lower in pure form of CNT than its reinforced state. In MWCNT/Mg sample, during processing, the strain gets locally accumulated in the composite, which acts mechanically on the CNT and thereby elevating the defect density. Higher value observed in MWCNT/Mg signifies the higher defect density in CNT evolved during processing. However, the similar value is reported by several researchers in which it is attributed to the damage of CNT [24–26]. Furthermore, significant shift of G-band from 1580 to 1595 cm^{-1} is observed after processing implying that lattice distortion of sp^2 bonded C-atoms occurred due to the existence of Mg atoms

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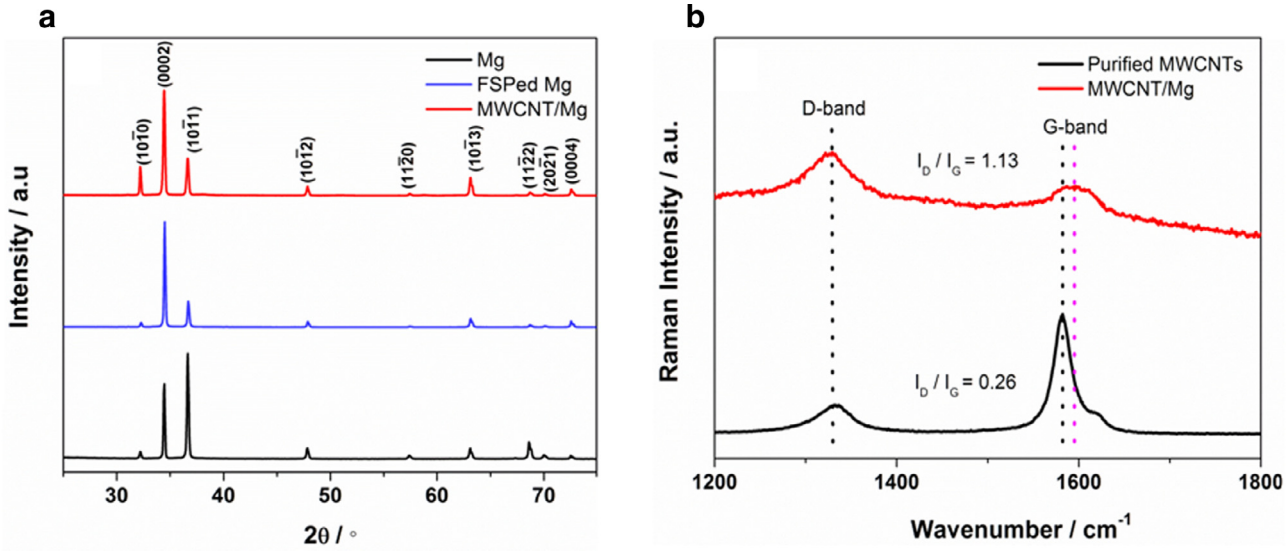

Fig. 5. (a) XRD pattern of pure Mg, FSPed Mg and MWCNT/Mg composite; (b) Raman spectra of purified MWCNT and MWCNT/Mg composite.

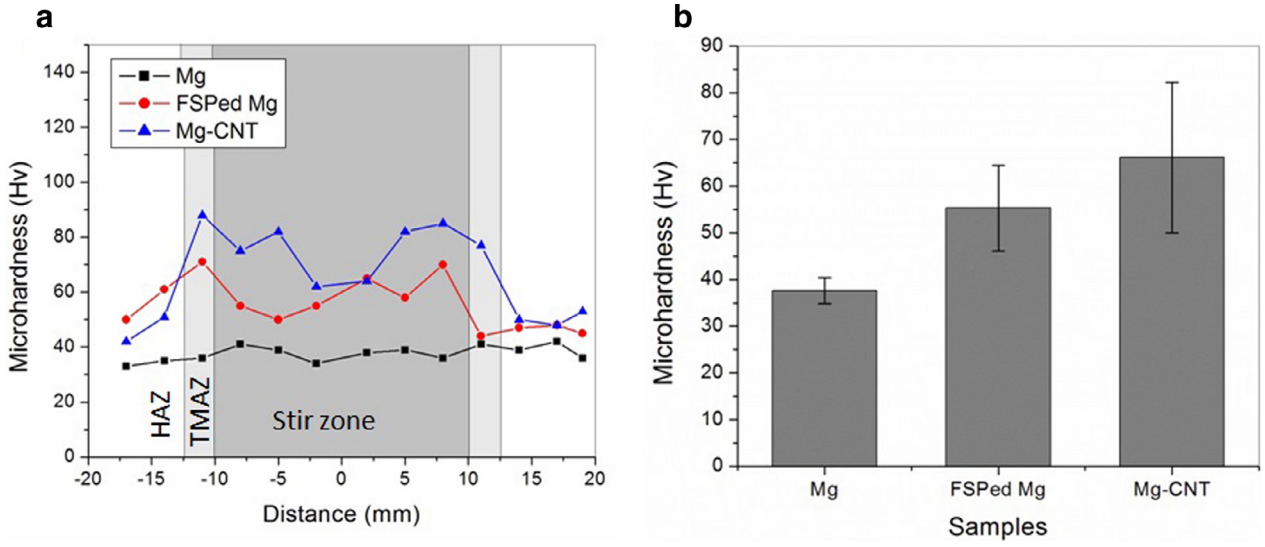

Fig. 6. Microhardness measurements of the samples: (a) hardness distribution and (b) average hardness (of 10 measurements).

in MWCNT/Mg composite. Up-shifting of G-band also attributes the transient state between crystalline and amorphous carbon [27].

Fig. 6 shows the microhardness measurements of the samples. The hardness distribution clearly indicates the increased hardness in the case of FSP Mg and MWCNT/Mg samples compared with pure Mg. The hardness data shows decreased trend when the measurements obtained at the TMAZ and HAZ. This trend supports that the microstructural variation in these three zones usually observed after FSP. It is true that the grain refinement results increase in hardness due to the grain boundary strengthening. From the results, the variation in hardness within the sample is also found to be higher for FSP Mg compared with pure Mg. Our earlier work on the grain refinement by FSP resulted two kinds of grains (coarse and fine) during processing of AZ31 Mg alloy [19]. Herein, a few regions within the stir zone have undergone more plastic deformation and resulted fine grains compared with the other regions. The level of uniformity in microstructural evolution was decreased and resulted different grain sizes within the stir zone and therefore, the variation in hardness was higher for FSP Mg compared with pure Mg. The hardness of MWCNT/Mg was observed as further increased compared with pure Mg and FSPed Mg. Interestingly, MWCNT/Mg has shown higher variation in hardness values compared with FSPed Mg. When the indent was placed on the regions where the amount of CNT was more, it is believed that higher hardness was recorded. Other regions where smaller grain size played major role have hardness close to that of FSPed Mg values. At this point, it must be emphasized here that both grain size reduction and presence of hard inclusion influence the bulk properties of the MWCNT/Mg composite. Most of

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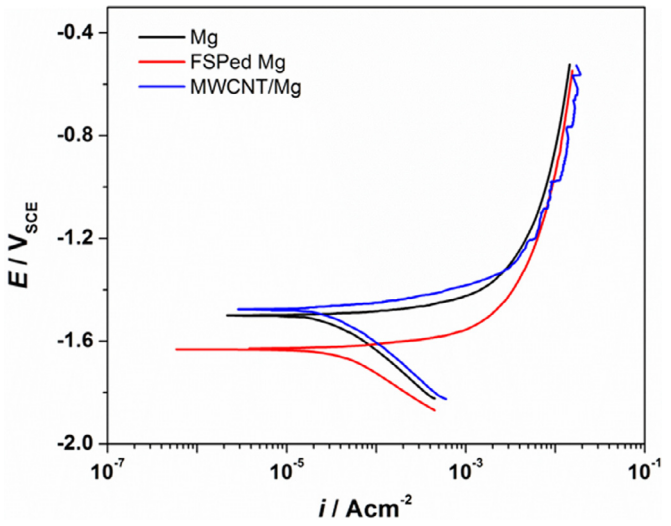

Fig. 7. Potentiodynamic polarization curves of pure Mg, FSPed Mg and MWCNT/Mg composite recorded using 3.5 wt.% NaCl at scan rate of 1 mV s^{-1}.

Table 1
Electrochemical parameters and calculated corrosion rate (mm/year) of the samples.

Sample	i_{corr} ($\mu A/cm^2$)	E_{corr} (V_{SCE})	Corrosion rate (CR) (mpy)
Mg	40.49	−1.50	36.41
FSPed Mg	35.23	−1.62	31.70
MWCNT/Mg	41.93	−1.47	37.73

the mechanical properties are structure sensitive properties which certainly influenced by the level of crystal imperfections. Usually, grain boundaries can be considered as surface imperfections. In addition, dispersing secondary phase also influence the bulk properties of the material. From the results, it is clear that the addition of CNT elevates the mechanical bulk properties of the Mg composite due to the combined effect of grain refinement and presence of CNT.

Fig. 7 shows the potentiodynamic polarization curves of the samples. The electrochemical parameters were obtained using Tafel extrapolation of the cathodic branch to the corrosion potential since the corrosion is cathodically controlled in Mg system. The corrosion potential (E_{corr}), corrosion current density (i_{corr}) and the calculated corrosion rates of the samples are listed in Table 1. From the results, it can be observed that the E_{corr} was moved to more negative values and corrosion current density i_{corr} was marginally decreased after FSP. Decrease in i_{corr} is attributed to the reduction in hydrogen evolution reaction which is clearly seen from the cathodic branch of the FSPed sample.

Reduction in cathodic kinetics can be deciphered using two crystallographic features—grain size and basal texture. In principle, smaller grain size offers more grain boundary area where the distribution of equivalent anodic current occurs simultaneously with the reduction of cathodic current due to diminution of cathodic area and results in shifting of cathodic branch to lower values of current [28]. On the other hand, more number of (0002) basal planes is exposed to electrolyte facilitating lesser dissolution due to their strong atomic coordination sites compared to other planes. Being a close packed structure, theoretical dissolution rate of (0002) planes are higher than that of other loose packed planes. In contrast, MWCNT/Mg composite showed higher i_{corr} albeit it exhibits (0002) basal texture. Addition of CNT promotes the nucleation site for galvanic corrosion to take place. Even though the basal texture predominates in MWCNT/Mg composite, CNT dispersion into matrix enhanced the galvanic intensities surpassing the texture and grain size effects. Also, from the Raman spectrum of MWCNT/Mg composite, it is clear that increase in intensity of D-band with suppression of G-band indicates that internal structure of CNT possess higher defect density. These defect areas act as active site for the higher dissolution to takes place. Formation of micro galvanic cells is the rate determining step for dissolution to take place. CNT which are nobler to the Mg matrix act as cathode and Mg matrix as anode forms galvanic couple due to their high potential difference (1.1 V_{SHE}), facilitating the corrosion to take place. Nanoscopic size of the local cathodes plays significant role in detaching the stable oxide layer and accelerating the electrochemical process. Furthermore, CNT fosters the large amount of corrosion products to be accumulated in their vicinity due to the strong galvanic corrosion at the interfaces between the Mg matrix and CNT [29]. This trend is consistent with literature reports where the corrosion resistance of Mg based composites is also decreased in the presence of CNT which initiate galvanic corrosion [17,29,30].

The major fraction of the specimen area used for corrosion tests must contain stir zone and TMAZ as the exposed area of the sample was 1 cm^2 which was collected from the centre of the processed region of the FSPed surface. But the presence of HAZ may not be seen in the specimen used for the corrosion test as the stir zone size is larger. The interfaces of stir zone, TMAZ and HAZ cannot be precisely identified and testing only such localized region requires specially designed experiments. It is worth highlighting that the addition of CNT does not always increase the performance of the composite on contrary may result in deteriorating the performance in other aspects. Hence, it can be understood that the mechanical performance of Mg can be improved to a great extent by adding CNT however care must be taken in the corrosion perspective. If the structure is made of MWCNT/Mg composite and is exposed to highly corroding environment, the possibility to corrosion initiated failure is increased due to the decreased corrosion resistance. On the other hand, studies on the effect of mechanical processing on the morphology change of embedded CNT are required. Furthermore, fine tuning of the addition of CNT into the Mg matrix to improve corrosion resistance and to investigate the CNT matrix interfaces would be the scope of the future work.

4. Conclusions

In summary, FSP has been successfully demonstrated as a promising tool to develop MWCNT/Mg composites within the solid state. Microhardness was increased for the composite

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compared with pure Mg and FSPed Mg to a great extent due to the combined effect of grain refinement and the presence of CNT. The corrosion performance of the composites was observed as degraded compared with pure Mg and grain refined FSPed Mg due to the presence of CNT which increased the galvanic corrosion. Hence, from the results it can be concluded that a special care must be taken while selecting the secondary phase particularly CNT to disperse in Mg in order to develop composites if the structure is intended to operate in corroding environment.

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