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Microstructure and mechanical properties of Al–2Mg alloy base short steel fiber reinforced composites prepared by vortex method

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Abstract Fabrication and characterization of cast Al–2Mg alloy matrix composites reinforced with short steel fibers are dealt with in the present study. Three types of steel fiber were used: uncoated, copper coated and nickel coated. All the composites were prepared by the liquid metal route using vortex methods. When tested in tension, all composites exhibited improvement in strength due to high relative strength of steel fibers. The ductility was lowered except for the composite with copper coated fibers. Copper coated fiber reinforced composites gave the highest strength. Higher strength accompanied with appreciable ductility demonstrated by composites with copper coated fibers is attributed to the solid solution and fiber strengthening as well as good bonding at the interface. Composites reinforced with uncoated and Ni coated steel fibers did not exhibit strengthening to the level exhibited with copper coated fibers because brittle intermetallic phases are formed at the interface. These phases promote initiation and facilitate propagation of cracks. The observed fracture mechanism of composites was dimple formation, fiber breakage and pullout of fibers. Fracture surface of uncoated and Ni coated composites showed extensive pull out of fibers as well as fiber breakage confirming the above inference. In case of the copper coated composites dimple formation and coalescence was more extensive. EDX analysis showed a build up Cu, Ni, and Fe at the interface.

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Introduction

Fiber reinforced metal matrix composite are widely accepted materials for their use in advanced application because they perform well at room temperature as well as in thermo structural environment. Metal matrix composites containing a variety of high strength, high modulus fibers are receiving considerable attention to meet the demand of advanced materials application. Potential applications of Al matrix composites take advantage of the excellent specific strength and specific stiffness of the aluminium specifically for large lightweight structures in the aerospace automobiles, transportation, communication and manufacturing industries [1–3]. Steel fibers are attractive as reinforcement primarily because of its high elastic modulus; high strength, low thermal expansion and good wear resistance. However it has a high density. Aluminium has good castability, low melting point, good mechanical working behavior, low density. These attractive properties are however associated with high thermal expansion and low strength. Thus a composite of aluminium with steel fiber reinforcement is expected to give in good combination of properties. However steel fiber is not wetted by aluminium [1] and is therefore difficult to fabricate such composites specific through the casting route. Fe reacts with aluminium and form intermetallic compound like Fe_2Al_5 and $FeAl_3$, at the [4, 5], damaging the mechanical properties of the theses composites. Two techniques are normally used to induce wetting in these composite system. These are coating of the fibers and the addition of specific elements to the aluminium alloy matrix.

Stainless steel wire reinforced composites prepared by squeeze casting showed tensile strength lower than that predicted by the rule of mixtures [5] indicating that the matrix fiber bonding is relatively weak probably as a result interfacial reaction. Bhagat et al. [5] identified several intermetallic compounds like $FeAl_3$ and Fe_2Al_5 at the interface in case of stainless steel wire reinforced composites. The properties of steel reinforced composites could be greatly enhanced by minimizing the volume fraction of reaction phase that formed between the steel reinforcement and Al matrix. Bhagat et al. achieved this by limiting the contact time between Al and reinforcing phase.

Coating of reinforcement in composites is important in developing high performance composites [6, 7]. Modification of the steel fiber surface is aimed to improve wetting behavior obtain better interface bonding and prevent the harmful chemical reaction between the steel fiber and aluminium. Addition of Mg to Al melt improves wettability by changing the interfacial energy possible through some interfacial reaction [8].

T. Suzuki et al. [9] examined the bending strength of electroless Cu and Ni coated carbon fiber reinforced composites prepared by the centrifugal pressure infiltration process. Bending strength of Cu coated carbon fiber reinforced composites was higher than that of Ni coated carbon fiber reinforced composites. Ni from the fiber surface comes contact with aluminium forming $NiAl_3$ at the interface and makes the composite brittle. Among these wide range of coatings used Cu and Ni appear to be most promising for an aluminium matrix.

Experimental

In the present investigation three types of short steel fibers were used as the reinforcing phase. These were used without coating as well as copper and nickel coating. A cheaper electroless method for obtaining the fibers was used. The main aim of the coating was to prevent harmful chemical reaction between the iron (in steel fiber) and aluminium melt during the mixing. Vortex method [10, 11] was used to produce the composites. The mechanical properties are correlated with microstructure of composites and the influence of coating on the fibers is analyzed in details.

The reinforcing fibers were prepared using cheaply available steel wool chemical composition as given in Table 1 and coating these with copper and nickel by an electroless plating technique. The steel fibers were dipped into an aqueous solution that contains 20% HCl for surface cleaning. After this the fibers was immersed in a solution that contain are shown in Table 2 for copper and for nickel coating. The activated steel fibers were immersed in $CuSO_4$ solution (shown in Table 2a) bath for 30 min. After that rinse the copper coated fibers with water and then dried. For nickel coating pH of the solution was maintained of the bath by addition of ammonium hydroxide. The solution was heated to 90–110 °C and then immersed steel fibers for 30 min. After the Ni coated fibers were rinsed with water and then dried.

It is observed that the Cu and Ni get deposited on the surface of the fibers. The fibers were then deoxidized in a hydrogen atmosphere at a temperature of 800 °C for 2 h. This process produces short steel fibers of about 550–850 μm length and 85–120 μm diameter with a coating of copper or nickel. The composition of steel fibers obtained is given in Table1b.

Commercial purity aluminium with composition as given in Table 3 was melted in an electric resistance furnace using a clay graphite crucible with bottom pouring arrangement. About 2 wt% commercially pure Mg was added to prepare Al–2Mg alloy. The melt was degassed with hexachloroethane tablets. After degassing the temperature was raised to 780 °C and melt was stirred with a graphite stirrer at 500–750 rpm to form a vortex. 5-wt% steel fiber preheated to 200 °C was added to the center of the vortex in a continuous stream while stirring was continued. The composite melt was bottom poured into cast iron dies preheated to a temperature of 550 °C. Plates of 8 mm thickness 65 mm width and 180 mm height were obtained. Microstructures of cast composites were examined under optical microscope to study the distribution of fibers. Samples for mechanical property measurement like hardness, micro hardness,

Table 1 Composition of fibers

Sample	C	Si	Mn	Cu	Ni	S	P	Cr	Fe
Fe	0.2	0.24	0.38			0.045	0.05	0.12	bal
FeCu	0.2	0.19	0.30	25.38		0.05	0.13	0.10	bal
FeNi	0.2	0.12	0.20		15.12	0.05	0.04	0.08	bal

Table 2 Bath composition for Cu and Ni depositions

Bath composition	Amount/Condition
Cu deposition	
$CuSO_4{\cdot}5H_2O$	62.5 g/l
PH	6.0
Temp	25 °C
Ni deposition	
$NiCl_2{\cdot}6H_2O$	45 g/l
Sodium hypophosphite	12 g/l
Ammonium chloride	50 g/l
Temp	90–110 °C
pH	8.5–10.5

Table 3 Composition of commercial aluminium

Sample	Si	Fe	Cu	Mn	Ti	Al
Al	0.09	0.168	0.04	0.03	0.01	Bal

density, and tensile strength were taken from the cast plates. Tensile samples of 25 mm gauge length, 4 mm width and 3 mm thickness were machined from the cast plate. Room temperature tensile test was carried out using a Universal Testing (Model No-AG5000G) machine at a strain rate of 2 mm/min. Hardness was measured using Brinell hardness tester with load of 500 kg. Micro hardness was measured with the help of Leco micro hardness tester with load of 0.025 kg. Fracture surface of broken tensile sample were examined using SEM.

Results and discussion

Discontinuous steel fibers were successfully incorporated into Al-2 wt% Mg alloy matrix by vortex method. The wettability is dependent on the type of interface between the liquid metal and the possible oxides that may be formed [8]. In the case of reactive metal-oxide system the contact angle is much lower indicating good wettability. Mg is one such reactive element and its addition to Al melt has been reported [12] to improve wettability of fibers by reducing the surface tension of the molten Al.

Microstructure

Microstructures of stir cast composites are shown in Fig. 1a–c. The samples are collected from top of the cast plate for optical microscopy and SEM analysis. Microscopic investigation of the casting revealed that the distribution of steel fiber was reasonably uniform with random orientation throughout the matrix. In general, it is observed that coating on the fibers improves the fiber distribution making it more uniform. As already stated, the poor wettability of steel fibers in aluminium melt is likely to give non-uniform distribution. Copper and nickel coatings on the steel fibers are expected to improve the wettability resulting in a better fiber distribution. The uncoated and the nickel-coated fibers shown in Fig. 1a, c are longer as compared to the copper coated fibers as observed in the structure. Thus, it can be concluded that the copper coated fibers are more liable to fragmentation during stirring and possibly during solidification as compared to the other fibers. Due to this shortening of the fibers contributes to greater uniform distributions in this composite. Magnesium addition to melt also serves the same purpose. The coating on the fibers prevents the contact between liquid aluminium and steel and possible formation of intermetalic compounds. The other features of microstructure are presence of porosity a characteristic of cast metal matrix composites particularly those produced by vortex method. Segregation of fibers at certain location is also noticed. These locations are invariably associated with porosity. The copper coated fibers were less segregated as compared to the nickel coated and uncoated fibers (Fig. 1a–c).

X-ray and SEM

X-ray diffractograms of fabricated steel fibers are shown in Fig. 2. In case of uncoated fibers, only Fe phase was

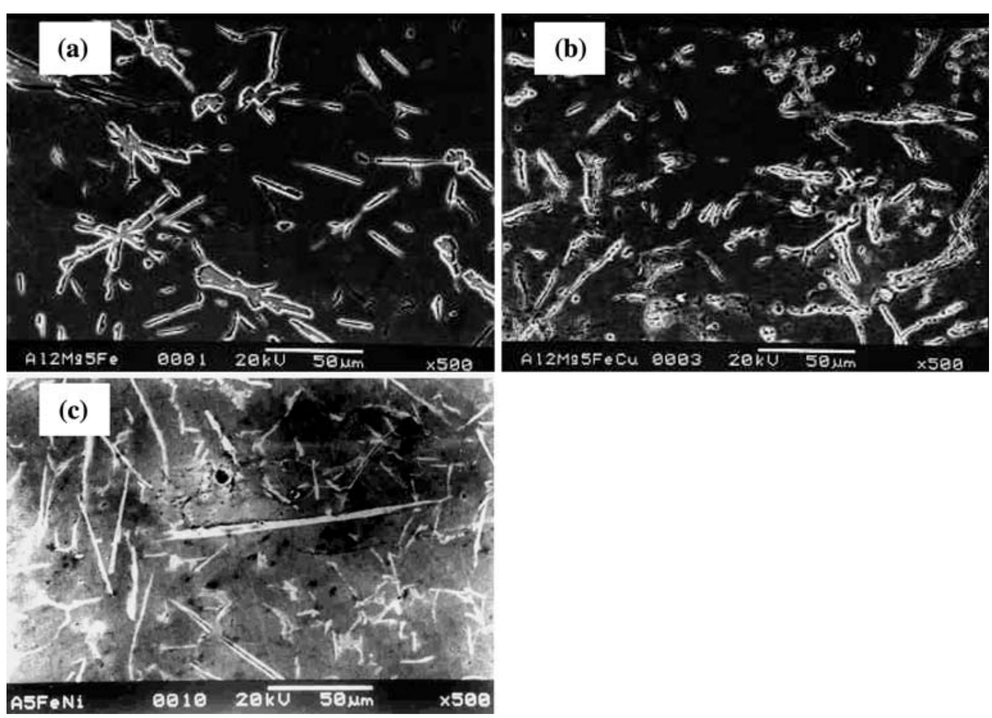

Fig. 1 Optical microstructure of copper coated fiber reinforced Al–2Mg matrix composites (**a**) Al–2Mg–5Fe, (**b**) Al–2Mg–5FeCu and (**c**) Al–2Mg–5FeNi

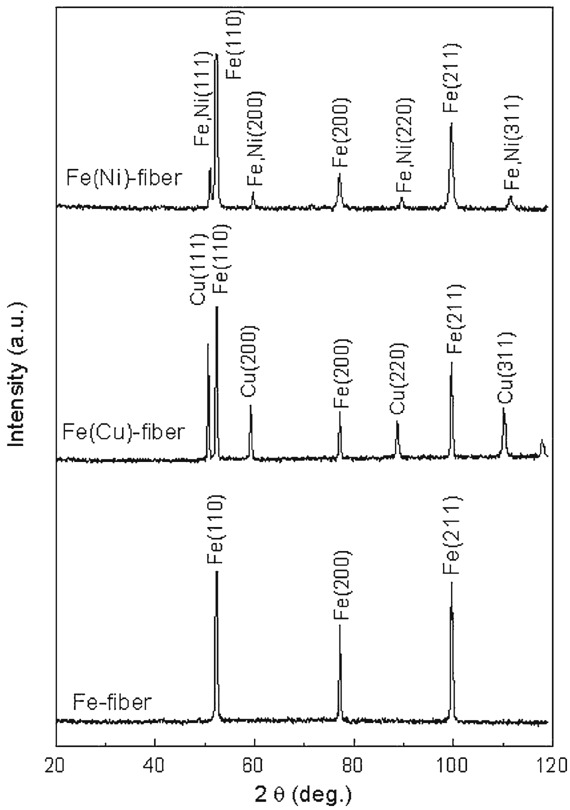

Fig. 2 X-ray diffractograms of different Fe-fibers (**a**) Uncoated (**b**) Cu-coated and (**c**) Ni-coated

identified. Presence of copper was detected in copper coated fibers. In case of nickel-coated fibers, no elemental nickel was detected. Nickel was present as compound like FeNi and $Fe_{0.64}Ni_{0.36}$ which is seen in the diffractogram (Fig. 2). However the major constituent is iron in both the cases.

The chemical analysis was done by EDX using the sample prepared for optical microscopy. The SEM micrographs for the line scanning from one fiber to another fiber are shown in Fig. 3. EDX bulk analyses give the % of elements at the interface, matrix interior and the fiber. Figure 4 shows the line scanning spectra of Al–2Mg base composites. Line scan of Mg concentration by EDX analysis confirmed the presence of magnesium through out the matrix. These traces also indicate the presence of magnesium, iron, and copper at the interface. Iron and copper content decreases whereas aluminium and magnesium increases as one moves from fiber to the matrix. The concentration of iron and copper are highest and that of aluminium, magnesium are lowest on the fiber surface.

The trace represents the variation in the effective atomic weight of all the elements present in the matrix, interface and the fiber. Solubility of copper in aluminium in solid state is 5.7%. So an appreciable quantity of copper dissolves in aluminium giving rise to solid solution

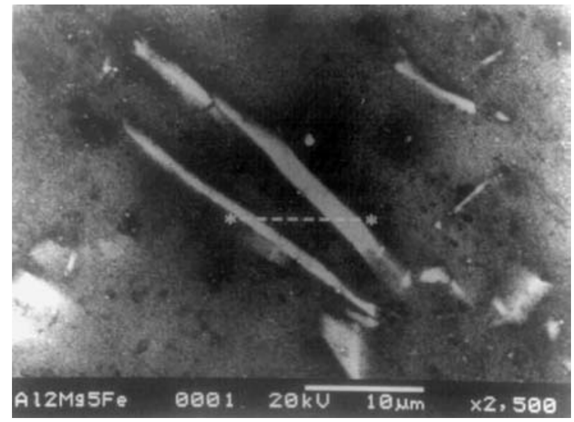

Fig. 3 SEM Photograph of Al–2Mg–5Fe composite line scanning taking between two fibers

strengthening. Thickness of interface length of copper coated fiber reinforced composites calculated from line scanning spectra is approximately 2 µm and where as it is 1.62 µm for nickel coated fiber composites.

It is clear from Fig. 4a, b that some amount of Fe, Cu and Ni are present at the interface as well as inside the matrix. These elements diffuse from the fiber surface during melting. Dissolution of copper in Al during the preparation of the composites results in a good interface bonding and improved solid solution strengthening. Good interface bonding helps in load transfer from matrix to fibers. In case of plain fibers or nickel-coated fibers, Fe and Ni on the surface react with Al and form brittle intermetallic compounds. The solid solubility of iron in aluminium is very low (0.052%) and thus most of the iron present in aluminium appears as an intermetallic phase. The solubility of nickel in aluminium does not exceed 0.04% in solid state. Over this amount it is present as an insoluble intermetallic usually in combination with iron [13]. It may be noted that the nickel-coated fibers contain Fe–Ni, $Fe_{0.64}Ni_{0.36}$ intermetallic compounds are identified by X-ray diffraction. Nickel is not present in the elemental form. These compounds further react with molten aluminium during stir casting and may form FeAlNi intermetallic compounds, as reported [14] earlier, at the interface, affecting the fiber distribution. These brittle intermetallic present at the interface act as nucleation sites for crack facilitating its propagation. X-ray studies of composites showed the presence of α-Al and α-Fe phase. However aluminide was not detected by X-ray diffraction as these are present in minor amounts. In case of copper-coated fibers copper present on the fiber surface diffuses into molten aluminium (as shown in Fig 4a) leading to better wetting, better fiber distribution and good interface bonding. The diffusivity and solubility of copper in aluminium being high there is little possibility of formation of $CuAl_2$.

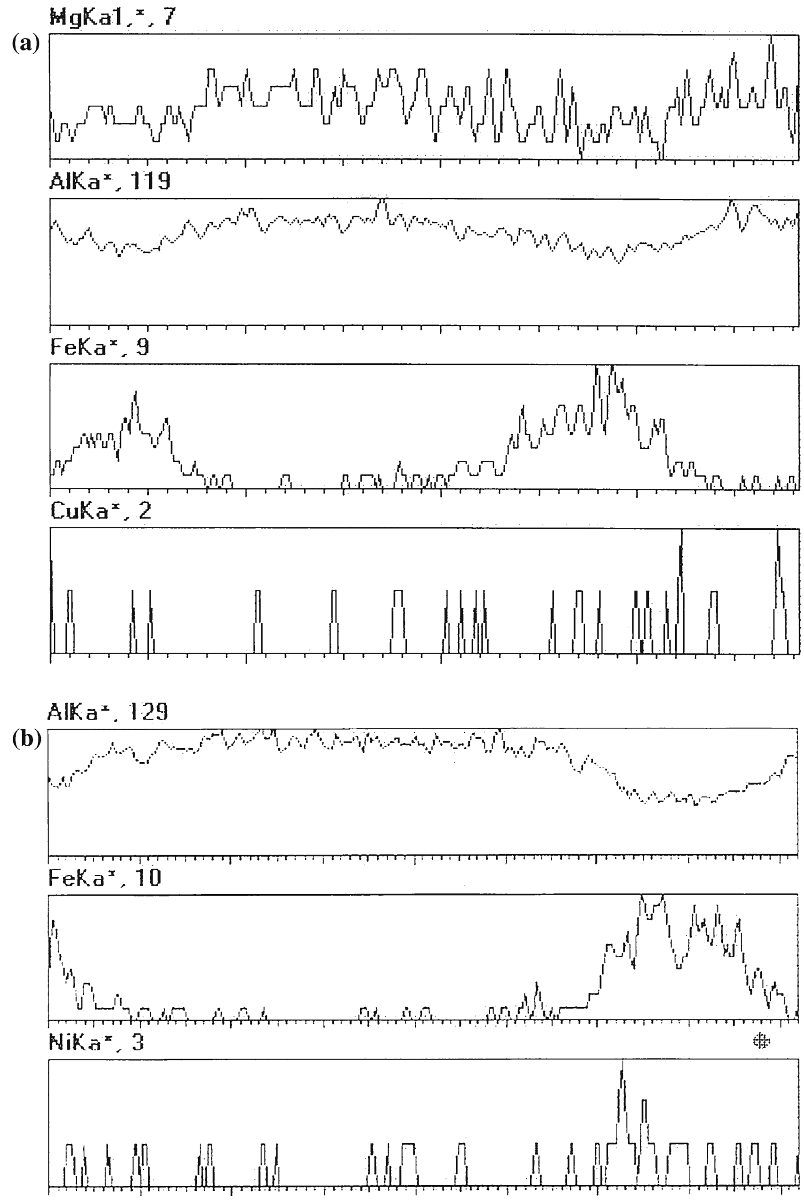

Fig. 4 Image traces of line scanning spectra of Al–2Mg base composites (**a**) Copper coated fiber and (**b**) Nickel coated fiber

Density and mechanical properties

Density and hardness of composites are shown in Table 4. As expected the density and hardness values increase with addition of fibers. In spite of rigorous degassing of the melt the casting contain porosity. Stirring of the melt to form a vortex and facilitate uniform distribution of fibers after the degassing leads to entrapment of gases and comes gas porosity in the cast plates. The porosity values tabulated in Table 4 are obtained from the theoretical density values calculated using the rule of mixtures. Lower porosity is observed in case of base alloy as compared to that in composites. Coating on steel fibers with copper gives the lower porosity level; whereas nickel coated fiber have the highest porosity. There is a significant improvement in the hardness of the composites with copper coated fibers and uncoated fibers. The addition of nickel-coated fibers also results in increase in BHN but not to that extent. The micro-hardness levels of uncoated and copper coated fibers are same where as that of the nickel-coated fibers is lower. Increase in micro hardness of the composites results the higher dislocation density in the matrix generated due to the difference in coefficient of thermal expansion of the Al matrix and steel fibers. The increase in hardness of the matrix can also be attributed to the dissolution of Cu in the matrix and the resulting solid solution hardening.

The tensile strength values of steel fiber reinforced composites are shown in Table 5. Fiber addition into Al–2Mg alloy improved mechanical properties but reduced ductility. It is seen that the ultimate tensile strength of the uncoated fiber reinforced composites is low but strength of Ni coated fiber reinforced composites was still lower.

Table 4 Density and hardness of Al–Mg base composites

Sample	Hardness (BHN)	Micro hardness (VHN)	Density (gm/cc)	Theoretical density (gm/cc)	% of porosity
Al–2Mg	39	52.00	2.6047	2.6804	2.824
Al–2Mg-5Fe	63.2	84.80	2.634	2.7717	4.960
Al–2Mg–5FeCu	66.8	85.07	2.658	2.7732	4.154
Al–2Mg–5FeNi	42	64.00	2.645	2.7725	5.681

There is a remarkable increase in strength with copper coated steel fiber reinforced composites. The variation of strength with nature of coating on the fiber can be explained by the extent and nature of interfacial reaction. It can be seen that interface reaction in the Ni coated and uncoated fiber composites are severe leading to formation of brittle intermetallic compounds and lowering the strength of composites. Previous work on steel reinforced aluminium MMC [5, 15] has shown the intermetallic phase formed to contain aluminium and iron. In case of Ni coated fiber composites the surface contains FeNi and $NiAl_3$ segregated in Al matrix [9] making the composites more brittle and lowering the strength. The coating with copper gives rise to most improvement in strength due to a clean interface, stable bonding and solid solution strengthening. Presence of Cu restricts the formation of brittle phase between Fe in the reinforcing fibers and liquid Al in Al–2Mg matrix alloy. There is a decrease in ductility associated with addition of fibers. However the drop in ductility was minimal with copper coated fibers. This also indicates good interface bonding in the case of copper coated fiber reinforced composites.

Fracture

The type of coating has not only affected the composites strength but also affected the fracture behavior of the composites. Fracture surfaces of coated and uncoated composites are shown in Fig. 5 a–d. It is observed that fracture mechanism of Al–2Mg base composites is mainly dominated by dimple formation whereas that of composites shows dimple formation and pull out of the fibers. On careful examinations, it is noticed that the pull out of fibers is more prevalent on the fracture surface of composites reinforced with uncoated fibers. Micro mechanism of fracture was given by dimples formation, fiber breakage and pullout of fibers. The micrograph in Fig. 5b shows that the failure in the uncoated steel fiber reinforced composites occurred adjacent to the fiber/matrix interface, within the reaction phase. In a previous study [16] of steel reinforced aluminium composites, it was observed that the failure occur at the fiber/matrix interface. In this case, the high volume fraction of reaction phase allows the crack to propagate readily, link up with other cracks along the fiber/matrix interface and cause failure. In effect fracture of the specimen occurs before the strength of the reinforcement is reached in the steel reinforced composites. From fractographs Fig. 5c result of the tensile testing, it can be presumed that FeNi present at the interface between steel fiber and aluminium matrix made the composites brittle and weakened the composites. Previous study [9] showed that nickel coating on the carbon fiber act with aluminium and forms brittle phase. But a careful observation of fractograph reveals the fracture surface of uncoated and Ni coated composites pull out of fibers and fiber breakages are observed more frequently. This can be attributed to a weak interface, while in the fracture surface of the Cu coated composites dimples formation and coalescence is more prevalent rather than pullout of fibers. The above facts indicate that ductile fracture occurs with the Cu coated fiber composites indicating a good bonding at interface. In uncoated and Ni coated fiber reinforced composites fracture initiated at the interface, due to presence of brittle intermetallic compound at the at the interface.

Table 5 Mechanical properties of Al–2Mg base composites

Sample code	Y.S (MPa)	U.T.S (MPa)	% Elongation
Al–2Mg	48	73	8.32
Al–2Mg–5Fe	74	112	1.65
Al–2Mg–5FeCu	122	162	3.2
Al–2Mg–5FeNi	69	105	1.26

Conclusions

Al–2Mg base steel fiber reinforced composite can be successfully obtained by vortex method. A uniform distribution of fibers with random orientation in matrices of Al–2Mg alloy can be obtained by the vortex method. Microstructure revealed the best distribution can be obtained using Cu coated steel fibers. Using coating on the fiber surface was effective in wetting the fibers and better interface bonding. The mechanical properties were better than the base metal. There is a significant improvement in strength and hardness with addition of fibers. Copper coated steel fibers improved strength from 73 MPa to 162 MPa with reasonable ductility. Ni coated fiber

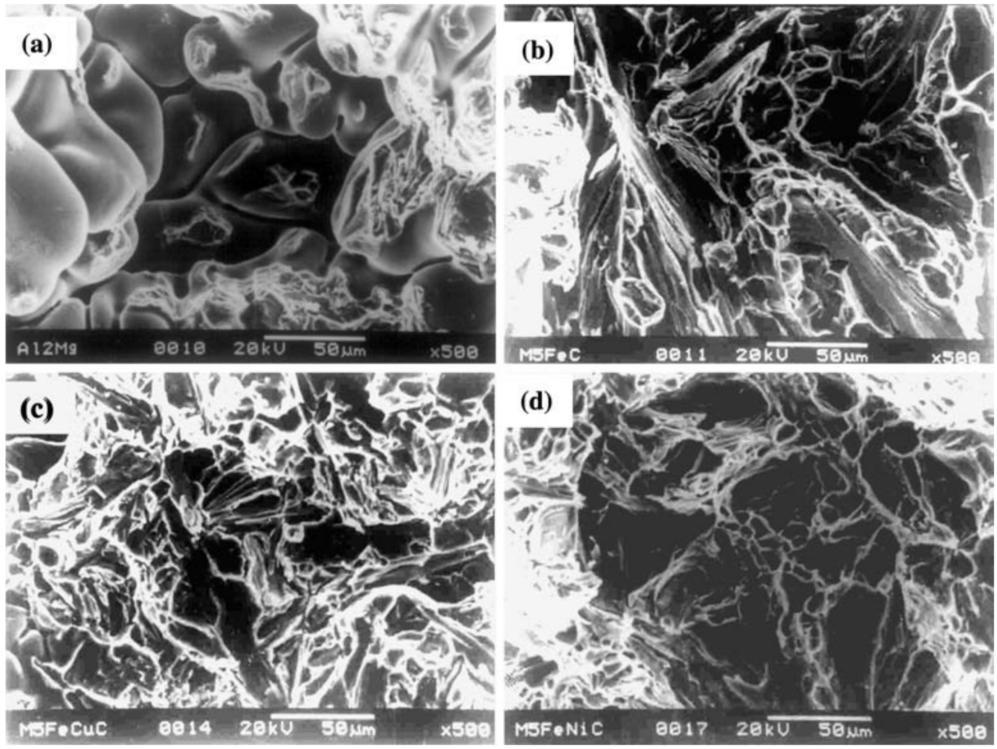

Fig. 5a-d SEM photographs of fracture surface of composites (**a**) Aluminium matrix, (**b**) Al-2Mg 5Fe composite, (**c**) Al–2Mg 5FeCu composite and (**d**) Al–2Mg 5FeNi composite

reinforced composites have lower mechanical properties due to intermetallic formation. Spot analyses indicate that Fe and Ni segregated at interface and formed some intermetallic compound like Fe_3Al_5 and $NiAl_3$, which is source of, crack initiation. In case of Cu coated fiber composites, Cu goes in to solution that restricts brittle intermetallic compound formation and formed solid solution with Al–2Mg alloy improving the mechanical properties. Fracture surface analysis showed that fiber pull out from Fe and Ni coated fiber composites was more extensive confirming brittle intermetallic compound formation at the interface.

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