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### Effect of Applied Pressure and Nickel Coating on Microstructural Development in Continuous Carbon Fiber-Reinforced Aluminum Composites Fabricated by Squeeze Casting

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# Effect of Applied Pressure and Nickel Coating on Microstructural Development in Continuous Carbon Fiber-Reinforced Aluminum Composites Fabricated by Squeeze Casting

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Continuous carbon fiber reinforced aluminum composite samples were produced by squeeze casting method, under applied pressures of 30, 50, and 70 MPa. For production of samples, nickel coated and uncoated carbon fibers with a mean volume fraction of 40% were used. After making the fiber preforms, they were preheated and then were replaced in a casting die. Molten 2024 aluminum alloy having a temperature of 750°C was poured into the die, and different amount of pressures were applied to infiltrate the melt into the carbon fiber bundles. The effect of applied pressure on infiltration mechanism and microstructure of the composite were studied, by evaluating the cross-section of the composite samples, using optical and scanning electron microscopes (SEM). The appropriate applied pressures for producing the composite samples for uncoated and nickel coated fibers were different, and the best results were achieved at 50 and 30 MPa, respectively. A specific or a certain range of pressure seems to be suitable for reaching to an appropriate microstructure in uncoated and coated samples. The results indicated that applying nickel coating on carbon fibers improves the infiltration of molten aluminum into the carbon fiber bundles and thus reduces the pressure required for infiltration significantly.

**Keywords** Carbon fiber reinforced aluminum composites; Microstructure; Nickel coating; Squeeze casting.

## INTRODUCTION

Reinforcing aluminum by carbon fibers offers the highest potential for increasing both the specific strength and the specific young's modulus, of the alloy and also decreasing its coefficient of thermal expansion [1–3]. Therefore, carbon-fiber reinforced aluminum components are of great interest for application in aerospace, automotive, and electric power cable industries. However, the poor wettability and chemical reactions between fibers and molten aluminum are main obstacles to synthesize these high performance materials and making use of their potentials [4]. One solution for these problems is to coat the carbon fibers with metal or ceramic materials which cannot only improve the wettability, but also impede interfacial reaction between carbon fibers and molten aluminum [5, 6]. For controlling harmful reactions at the interface, the contact time between the reinforcement and the molten metal, at high temperature, has to be short [7]. Since squeeze casting process is a fast filling method, it is suitable for production of these types of composites [8]. Other problems in producing these composites are the occurrence of fibers damage and their clustering under applied pressures [9]. Closely packed fibers can cause poor infiltration and

generate defects and then ends in catastrophic failure of the composite [10].

In the present study, for overcoming the problems due to harmful interfacial reactions between carbon fibers and molten aluminum occurring during processing of carbon-fiber reinforced aluminum composites, the carbon fibers were primarily coated by a nickel film prior to subjecting them to various squeeze casting pressures. Then the optimum applied pressures for processing of the composites having either coated or uncoated carbon fibers were found.

## EXPERIMENTAL PROCEDURE

### Materials

The composite samples were fabricated from 2024 aluminum alloy, containing 4.30 wt% Cu, 1.14 wt% Mg, and 0.42 wt% Si, as matrix and commercially available polyacrylonitril (PAN)-based carbon fibers as reinforcement. Carbon fibers were in the form of bundles containing 6000 single filament carbon fibers with about 5.7 μm diameter. The average volume fraction of the fibers used for producing the composite samples was about 40%.

### Coating the Fibers

The carbon fibers were coated with a film of nickel having 0.5 μm thickness, by electroless method. At first, the organic sizing, usually present on the as-received carbon fibers, was removed by heating the fibers up to 550°C for 30 minutes. Then the heated fibers were degreased with acetone and were immersed in a commercial activating solution to

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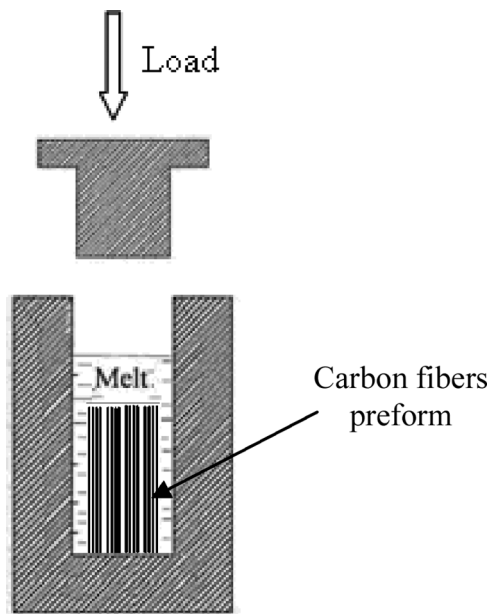


FIGURE 1.—Schematic diagram of the die apparatus and carbon fiber preform used for preparation of composite samples via squeeze casting.

achieve the required conditions for the catalytic reaction of electroless deposition. For nickel coating, commercial solution of nickel electroless made by Schlotter Company, called Slotonip 90, was used. The coating process was carried out up to a certain time to achieve an optimum  $0.5\ \mu\text{m}$  coating thickness. More details about coating process in this work and optimizing of coating thickness have been reported and discussed elsewhere [5].

#### Specimens Preparation

Continuous carbon-fiber reinforced aluminum composite samples were produced by the squeeze casting method under different applied pressures. Figure 1 shows schematically the cylindrical H13 tool steel die with an internal radius of 30 mm, external radius of 65 mm, and a height of 170 mm, used for preparing the composite samples. To prepare fiber preforms, coated and uncoated carbon fiber bundles were wound unidirectionally. Then they were preheated at  $500^\circ\text{C}$  before placing them into a preheated casting die at about  $250^\circ\text{C}$ . Molten 2024 aluminum alloy having a temperature of  $750^\circ\text{C}$  was then poured into the die, and various amount of pressures (i.e., 30, 50, and 70 MPa) were applied to infiltrate the molten aluminum into the carbon fiber bundles. When the solidification was completed, approximately after 30 s, the pressure was stopped and billets were removed from the die. The excess aluminum, around the billets, was cut away by machining, so that smooth surface composite rods were obtained.

#### Microstructural Analysis

To investigate the effects of applied pressure and nickel coating on the composite characteristics such as distributions of fibers, microstructure of the composite, and also probable casting defects, the samples were sectioned

in transverse direction and then studied in an optical microscope and in a scanning electron microscope (SEM). Compositional analysis of the selected microstructure was also carried out using an energy dispersive spectroscopy (EDS) attached to SEM.

#### RESULTS AND DISCUSSION

Figure 2 shows a typical cross-section of composite sample fabricated with uncoated carbon fibers under 50 MPa applied pressure. As shown in this figure, aluminum melt has completely filled the space between the carbon fiber bundles; however, for producing a defect free composite sample, infiltration of the melt within every single filament of the carbon fibers in each bundle is also needed.

Figure 3(a–c) shows micrographs of unidirectional Al/C composites fabricated with uncoated carbon fibers, under 30, 50, and 70 MPa applied pressures. These micrographs are focusing on the interior space of each carbon fiber bundle. Figure 3(a), taken from the sample produced by 30 MPa applied pressure for infiltration of molten aluminum into the uncoated carbon fibers, shows that incomplete infiltration has taken place. On the other hand, applying 70 MPa pressure on the melt, has caused disorder and heterogeneities in the distribution of the uncoated fibers, so that the fibers displacement created a condition that molten aluminum could not enter between all individual fibers [Fig. 3(c)]. Finally, composite sample fabricated at 50 MPa applied pressure shows a better distribution of carbon fibers within the aluminum matrix because of good infiltration characteristic [Fig. 3(b)].

Figure 4 shows a SEM micrograph taken from the cross-section of a sample fabricated with the uncoated carbon fibers under 50 MPa applied pressure. Although an ideal distribution of the fibers did not occur in the sample, this figure shows a good infiltration of aluminum within the space available between carbon fibers in the bundle has occurred, and all the spaces in the preform were filled.

Incomplete infiltration of uncoated carbon fibers with aluminum melt, for 30 MPa pressure, can be explained using the following equation, suggested by Mortensen and Cornie [11] for metal matrix composites:

$$P = \frac{\sigma_{LA} \sin(\theta - \alpha - \frac{\pi}{2})}{R \sin(\alpha) \text{tg}(\frac{\alpha}{2})} \quad (1)$$

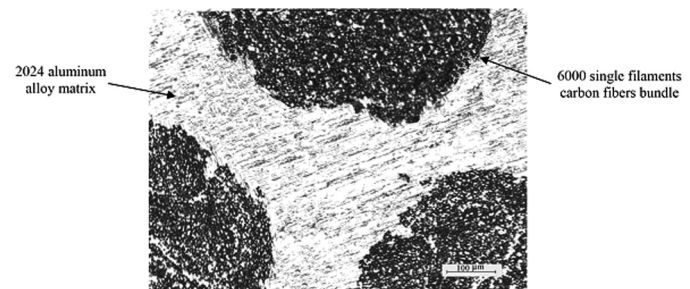


FIGURE 2.—Low magnification optical micrograph of cross-section of a continuous Al/C composite fabricated with uncoated carbon fibers under 50 MPa applied pressure.

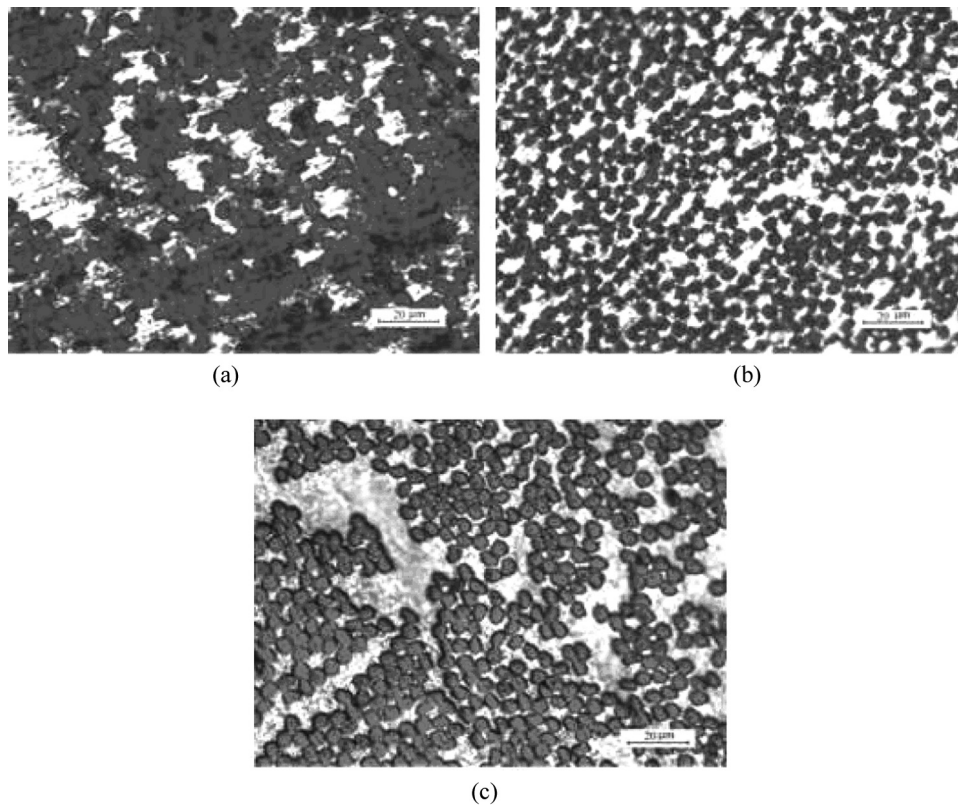


FIGURE 3.—Optical micrographs of cross-sections of continuous Al/C composites fabricated with uncoated carbon fibers under different applied pressures: (a) 30 MPa, (b) 50 MPa, and (c) 70 MPa.

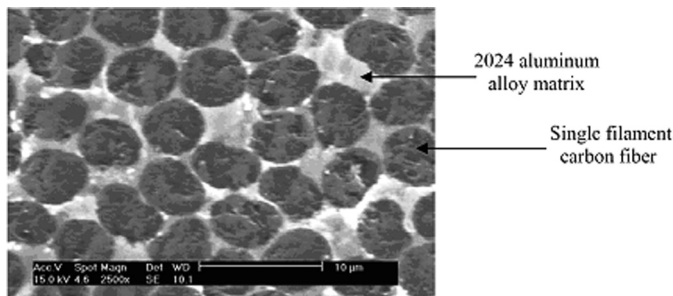


FIGURE 4.—SEM micrograph of the cross-section of unidirectional Al/C composites fabricated with uncoated carbon fibers under 50 MPa applied pressure.

This equation shows that the necessary pressure for complete infiltration, between two fibers, depends on the following:

$\sigma_{LA}$ , surface tension of the melt,  
 $\theta$ , contact angle between fibers and melt,  
 $R$ , fiber radius, and  $\alpha$ , which is an angle between contact points of two fibers and the point where the melt has stopped moving.

The later is an indication of the ability of melt infiltration between two fibers. Figure 5 shows the relationship between these parameters schematically.

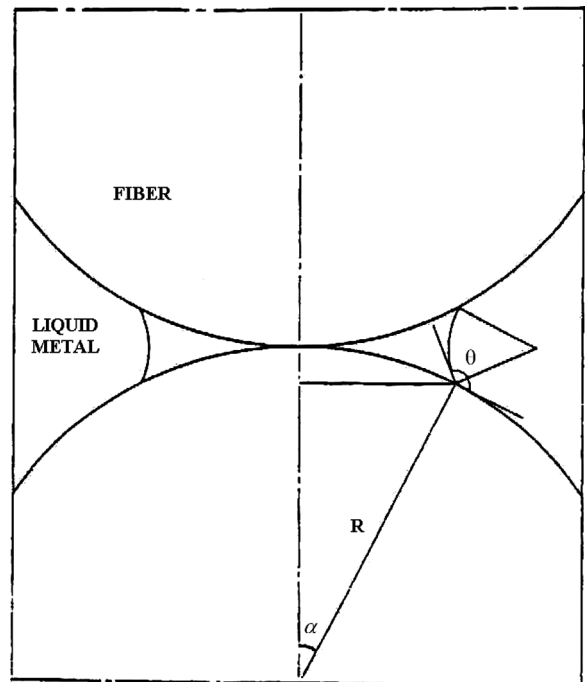


FIGURE 5.—Schematic illustration of infiltration between two fibers which are in contact with each others [11].

The contact angle between the carbon fiber and aluminum melt is  $140^\circ$  according to Ref. [12], which means that the

wettability between these two materials is quite poor. On the other hand, since the diameter of carbon fibers,  $R$ , is very small ( $5.7 \times 10^{-6}$  m), it causes a rise in the amount of pressure. In addition, because the fibers within a bundle are very close to each other, i.e., almost in contact with each other, this can cause the value of wettability angle  $\alpha$  to increase. Therefore, considering the above argument and using Eq. (1), one can deduce that a relatively high pressure is necessary to complete infiltration of uncoated carbon fibers with molten aluminum. Therefore, a 30 MPa applied pressure seems to be insufficient to complete infiltration of uncoated carbon fibers.

On that other hand, it seems that the flow of infiltrated melt has not been regular in Fig. 3(c). Interestingly, there is some similarity between Figs. 3(a) and (c). In both cases, clustering and heterogeneities in distributions of carbon fibers can be observed, but the noticeable difference in these figures is the presence of alloy matrix between some of close fibers in the bundle in Fig. 3(c). The presence of alloy matrix within some of the fibers and heterogeneities in distributions of carbon fibers observed in Fig. 3(c) is not due to lack of enough pressure, but it may occurred due to other phenomena acting on the fibers, not letting them to reach a regular distribution. For example, this can be due to the turbulent melt flow which might have been occurred as the applied pressure increased to 70 MPa. It is not clear, at this stage, what the possible threshold pressure for starting the turbulent flow of the melt is. However, based on the obtained results, the best pressure for avoiding turbulence and getting acceptable results during infiltration at the uncoated carbon fibers is about 50 MPa.

Figure 6 shows a SEM micrograph of unidirectional Al/C composites fabricated with nickel coated carbon fibers under 30 MPa applied pressure. This figure indicates that, in the case of nickel coated carbon fibers, 30 MPa applied pressure has caused complete infiltration of aluminum melt into the fiber bundles and has also led to acceptable distribution of the carbon fibers in the composite matrix. It is worth mentioning that the result of 30 MPa pressure, on nickel

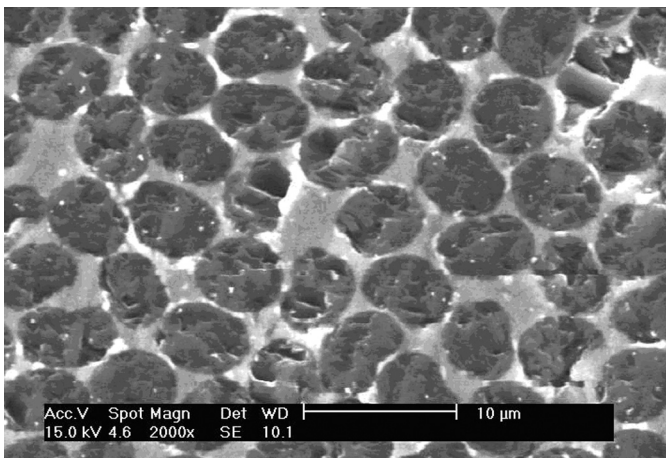


FIGURE 6.—SEM micrograph of the cross-section of unidirectional Al/C composites fabricated with nickel-coated carbon fibers under 30 MPa applied pressure.

TABLE 1.—Effects of applied pressure and coating of the fibers on the composites quality.

Carbon fibers condition	Applied Pressure (MPa)		
	30	50	70
Uncoated carbon fibers	Unsatisfactory	Good	Unsatisfactory
Ni-coated carbon fibers	Good	Unsatisfactory	Unsatisfactory

coated fibers, was very similar to the uncoated fibers when a pressure of 50 MPa was used (Table 1).

Micrographs presented in Fig. 7 show the SEM image taken from the cross-section of Al/C composite, fabricated with nickel-coated carbon fibers under 30 MPa applied pressure, and the corresponding Al, Ni, and C EDS maps. The presence of nickel-coated layer around the carbon fibers and good infiltration of the molten aluminum between the fibers can be observed in this figure.

Some researchers have reported the effect of nickel coating on improving the wettability between coated carbon fibers and molten aluminum [12, 13]. It has also been reported [12] that nickel coating can reduce the contact angle from  $140^\circ$  to less than  $90^\circ$  [12]. The change of contact angle plus a minor change in  $R$  and  $\alpha$  can reduce the required pressure for filtration, on basis of Eq. (1). Considering the above argument and the result presented in Table 1, it seems the microstructures of unidirectional Al/C composite fabricated with nickel-coated carbon fibers, using 50 and 70 MPa pressures, had the similar effects such as those shown in Fig. 3(c), which have unacceptable amounts of heterogeneities in fibers distribution. Therefore, in this case, the optimum pressure seems to be 30 MPa, and using applied pressures such as 50 and 70 MPa is not suitable for production of a sound composite from coated carbon fibers and molten aluminum.

## CONCLUSIONS

To sum up, the following conclusions can be drawn from the results obtained in this research:

- Continuous carbon fiber reinforced aluminum composites with uncoated and nickel-coated carbon fibers can be produced by squeeze casting method, using appropriate applied pressures.
- Using nickel-coated carbon fibers can reduce the required pressure for production of the composite significantly. This is due to improvement of the wettability of carbon fibers with liquid aluminum during processing.
- Results of this research showed that the appropriate pressures for manufacturing carbon fiber reinforced aluminum composites, by squeeze casting process, in uncoated and Ni-coated condition are 50 and 30 MPa, respectively.
- There are optimum pressures ranges for production of both uncoated and coated composites, outside which irregularities in the fibers distribution may occur and lead to an unsatisfactory result.

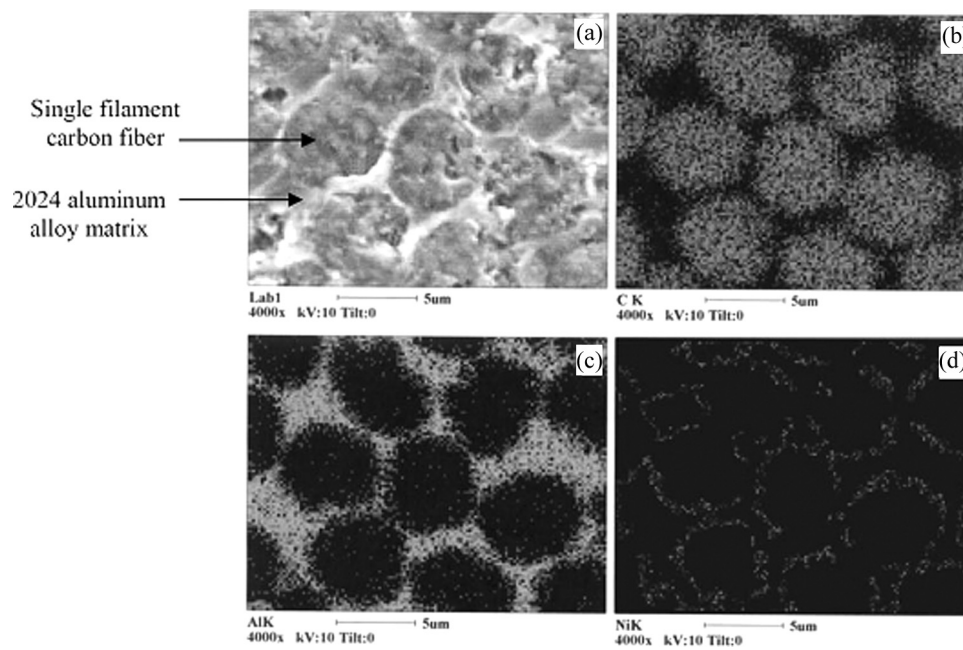


FIGURE 7.—EDS maps of the cross-section of unidirectional Al/C composites fabricated with nickel-coated carbon fibers under 30MPa applied pressure: (a) SEM micrograph of the scanned area, (b) C distribution map, (c) Al distribution map, and (d) Ni distribution map.

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