# THE STUDY OF ELECTROLESS COATING OF NICKEL ON CARBON FIBERS

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***Abstract:*** *The continuity and thickness of the coating layer, are the most important factors in wetting properties and strength of carbon fibers. These factors are crucial in the quality of metal matrix composites made with carbon fibers. In this research the Polyacrylonitrail base carbon fibers have been nickel coated with 0.2, 0.5, 0.8 and 11 μ m thickness, by the electroless method. The effect of the thickness of nickel coating on surface condition and also the tensile strength of the carbon fibers has been investigated. The study of surface condition of the coated carbon fibers by SEM showed that the nickel coating at the thickness of about 0.5 μm has the best continuity on the carbon fibers. The results of tensile tests of carbon fibers coated with different thickness of nickel showed that increasing the thickness of coating layer decreases the overall strength of fibers.*

***Keywords:*** *Carbon fibers, Nickel coating, Surface condition, Tensile strength.*

## 1. INTRODUCTION

Carbon fiber reinforced metal matrix composites are suitable substitutes for conventional metals used at different industries, especially aerospace industry, due to their high special strength, stiffness and good dimensional stability. Weak wettability and destructive chemical reaction between the carbon fibers and aluminium matrix are problems that always have been mentioned in fabrication of this composites by many workers [1,2,3].

To solve these problems many researchers have used metals like copper, silver, nickel, tantalum and cobalt and ceramics like titanium boride and born carbide to coat carbon fibers [4]. Nickel is used in many cases to improve wettability and also prevent destructive reaction between carbon fibers and metallic matrix. It has been reported that nickel coating not only improves the wettability but also decreases the temperature and pressure which is necessary for infiltration of liquid metal in between carbon fibers [5].

The first attempts for coating carbon fibers with nickel, has been performed by Sara [6]. He nickel coated, Ryon based carbon fibers, using electroplating method. Lamotte[7] and Rye[8] have also used nickel coated carbon fibers in their research in order to fabricate the aluminum-carbon composites.

Although the coating of carbon fibers with nickel seems to be a relatively simple, there are some limitations, which may cause serious problems in the procedure of production of MMC. It has been reported that, if the thickness of the coated layer becomes less than about 0.4 μm, the infiltration of molten metal will be imperfect [5].

Different processes and methods have been introduced and used for coating of carbon fibers. However, any method that is used has to solve the problem of continuity of coating on the surface of all existence fibers in a bandle. Abraham et al. [9] in their research work coated carbon fibers with nickel by different methods like cementation, electroplating and electroless process. They suggested that electroless process is easier and has lower probability of mechanical damage to fibers. In addition it is easier to control the thickness of metal deposite on fibers [10]. The thickness of coating layer and its morphology has an important role on the mechanical properties of the carbon fibers. Carbon fibers when imposed to these surface treatments, lose their tensile strength to some extend. The decrease in the strength of the fibers is usually because of a) the lake of continuity in

the coating, which may act like a crack or notch and b) the overall increase of the fiber cross-section [9].

In this research carbon fibers have been nickel coated using electroless method and then the continuity of the coating on the carbon fibers bandles and also the effect of coating thickness on tensile strength of fibers have been investigated.

## 2. EXPERIMENTAL PROCEDURE

The fibers used in this work were commercially available Polyacrylonitrail (PAN) base, with diameter of about 5.7 $\mu$m under the trade name of TC-42 from Tairyfil.

To determine the thermal stability of carbon fibers, they were subjected to simultaneous thermal analysis (STA) in the first stage. The diagram of thermal analysis of the carbon fibers is shown in Fig.1. Then the organic sizing present on the as received carbon fibers, was removed by heating the fibers at 550 ºC for 10 minutes. Then the heated fibers were degreased with acetone and after washing with distilled water they were immerged in commercial activating solution. The solution included 0.15 g/lit $SnCl_2$ and 0.3 g/lit $PdCl_2$. To remove the excessive tin from the surface of the fibers, the samples were immersed in commercial HCl 10% for 5 minutes and then washed with distilled water. After these steps, the surface of fibers were activated to achieve the required preparation for the catalytic reactions of electroless deposition.

For nickel coating, commercial solution of nickel electroless made by Schlotter Company named Slotonip90, was used. This solution contains $Ni^+$ ion, reduction elements and also complexeant, buffer, accelerator and stabilizer elements. The coating process was carried at the temperature $90\pm2^o$ C and the solution pH was fixed at $4.7\pm0.1$ using Ammoniac. The rate of nickel deposition from the solution was measured to be approximately $13\pm1$ $\mu$m/h. To achieve different coating thicknesses, the coating process was carried at different times. Carbon fibers with coating thickness of 0.2, 0.5, 0.8 and 1.1 $\mu$m, were obtained. To make sure of proper coating of internal fibers within the bandle and also to eliminate

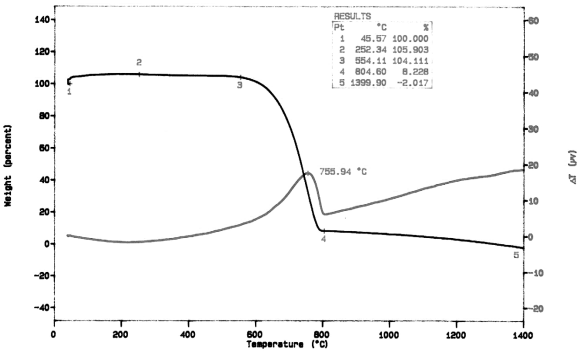


**Fig. 1** Simultaneous thermal analysis diagram of TC-42 carbon fiber in air.

the gas bubble from the surface of fibers, the solution was mixed with a mechanical pump.

In order to calculate the coating thickness, the weight of the carbon fibers were measured before and after electroless deposition using analytical balance of 0.0001g accuracy. From the weight of the coating,$\Delta G$, the coating thickness, Äã, was calculated usig the formula (1)

$$\Delta\gamma = \left( \sqrt{1 + \frac{\rho_{Ni}}{\rho_c} \frac{\Delta G}{G}} - 1 \right) \gamma \qquad (1)$$

where $\rho_{Ni}$ and $\rho_c$ represent the density of nickel and carbon fiber respectively, G, the weight of the fibers prior to coating, and، ã, the diameter of the uncoated fibers.

In order to evaluate the coating morphology on the fibers, surface of the coated fibers were studied by SEM after the coating process.

Evaluation of the effect of nickel coating on the strength of the fibers in as-received and after coating was carried out in different thickness of coating. Tensile tests were performed using single fiber test (SFT) method according to ASTM D3379-75. Based on the introduced procedure of the test, a randomly selected carbon fiber was taken from the coated bandle. It was glued on to a paper frame using an epoxy adhesive and the sides of the frame were carefully cut after it was gripped in the tensile testing machine. A Fafograph micro tensile testing machine with a 10 N-load cell was used for the tensile test of the carbon fibers. Fifty tests were carried out for each experiment with an effective gauge length of 10 mm and a crosshead rate of 1.0 mm/min. The procedure of the tensile test for carbon fibers is

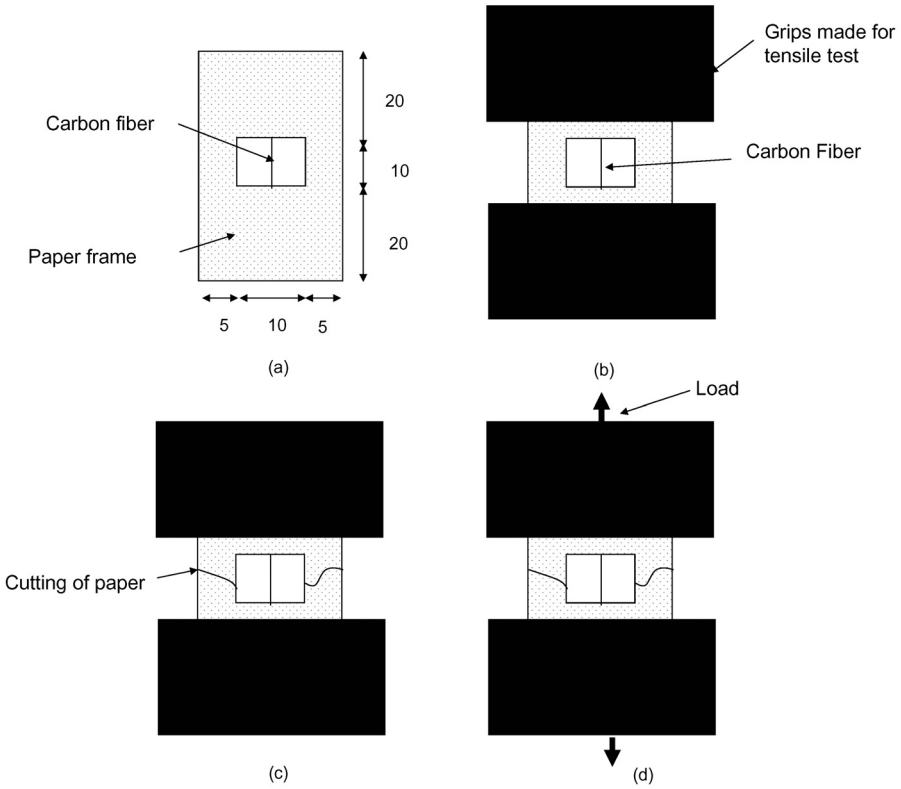


**Fig.2** The procedure of the tensile test of carbon fiber. (a) A single fiber glued onto a paper frame of a single fiber; (b) gripping in the tensile machine of paper frame; (c) cutting of paper frame; (d) tensile test.

shown in Fig.2.

## 3. RESULTS AND DISCUSSION

### 3.1. Surface condition of nickel coated carbon fibers

Typical cross-section of nickel coated carbon fibers is shown in Fig.3 and the EDX (Energy Dispersive X-ray analysis) mapping of this section is shown in Fig.4. Formation of nickel coating on the carbon fibers is quite clear in the Fig. 4. SEM micrographs of the longitudinal section of nickel coated carbon are shown in Fig.5 to Fig.9. These figures show fibers that have been coated with different thickness.

Figurers 5 and 6 show two positions of fibers coated with thickness of 0.2 $\mu$m. It seems that nickel coating had not been formed on fibers uniformly and some parts on the surface of the fibers are free from coating or possibly with low thickness. Figure 7 shows SEM micrograph of some nickel coated carbon fibers with 0.5 $\mu$m thickness. The thickness of coating seems to be nice and uniform in this picture.

Fig.8 shows fibers with thickness of coating about 0.8 $\mu$m. Although the coating of fibers seems to be uniform to some extent, there are indications of some new phases which have started to grow on the surface of nickel coated fibers. In the Fig.9 in which the thickness of coating has increased to 1.1 $\mu$m, there are more indications of growth of new phases. By increasing the thickness of coating from 0.8 $\mu$m to 1.1 $\mu$m, nonuniform coating has started to form on the surface of the fibers due to local growth of nickel coating on already coated surface.

It seems that, at first, nickel deposits at energetically favored sites on the surface of carbon fibers. In the thickness of coating of order of 0.2 $\mu$m discontinuity of coating is quite clear. Later by continuing the process, coating covers remained surface of the fibers. Ultimately at the thickness of around 0.5 $\mu$m and possibly less than 0.8 $\mu$m the entire surface of the fiber will be coated uniformly. Continuing the deposition and at

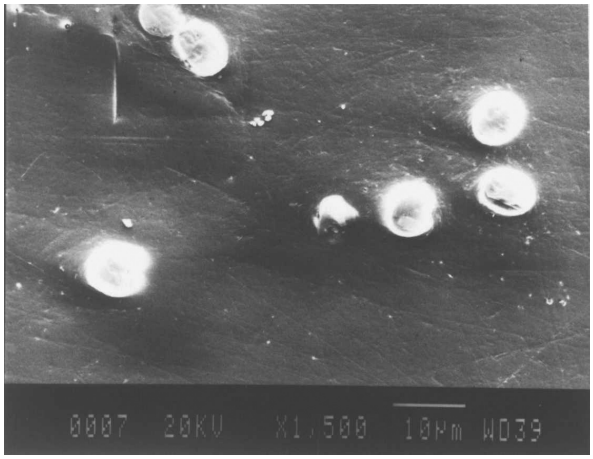


**Fig. 3** The SEM micrograph of the cross-section of nickel coated carbon fibers.

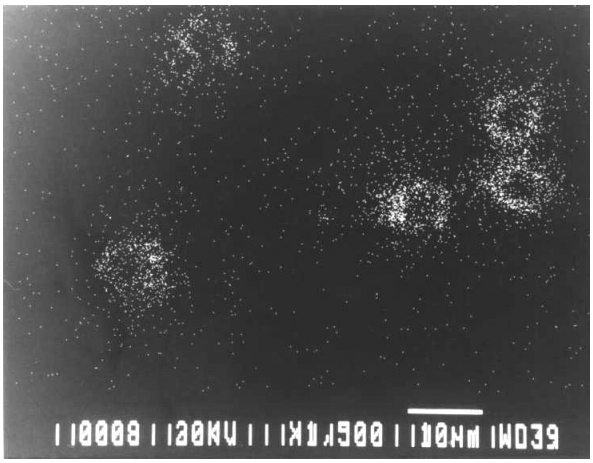

**Fig. 4** EDX mapping of the same area in Fig. 3

**Table 1:** Properties of TC-42 carbon fiber.

| | |
|---|---|
| Filaments per Tow | 6000 |
| Unit Weight (g/m) | 0.28 |
| Diameter (μm) | 5.7 |
| Density (g/cm$^3$) | 1.81 |
| Sizing Content (%) | 1.2 |
| Tensile Strength (MPa) | 4680 |
| Tensile Modulus (GPa) | 290 |

thickness more than 0.8 $\mu$m, nickel coating starts to growth denderitically on the surface of the fibers that have been already coated.

### 3.2. Tensile strength of nickel coated carbon fibers

In Fig.10, changes of ultimate tensile strength of nickel coated carbon fibers as a function of coating thickness is illustrated. The empirical values driven from tensile tests of carbon fiber coated at different thickness, is compared with the theoretically calculated values. The theoretical tensile strength of nickel coated carbon fibers has been calculated by the rule of mixture (ROM).

$$\sigma_C = \sigma_f \ V_f + \sigma_{Ni} V_{Ni} \qquad (2)$$

In this equation, $\sigma_c$is the ultimate tensile strength of nickel coated carbon fibers, $\sigma_f$ is the ultimate tensile strength of uncoated carbon fibers and $\sigma_{Ni}$ is the tensile strength of nickel. $V_f$ and $V_{Ni}$ are the volume fraction of carbon fibers and nickel coating respectively. The amount of $\sigma_f$, has been considered 4577 MPa from tensile test of uncoated carbon fiber (a little less than the amount presented at Table 1) and the amount of $\sigma_{Ni}$ has been considered 345 MPa.

According to Fig.10, the tensile strength of carbon coated carbon fibers has decreased from 3456 MPa in the coating thickness of 0.2 $\mu$m to 2194 MPa in the coating thickness of 1.1 $\mu$m. The rate of decrease in the range of uncoated fibers to 0.2 $\mu$m thickness of coating is more than other parts of graph. Theoretically calculated results show the same trend as experimental in the range of 0.2 to 1.1 $\mu$m.

The main reason of decrease in tensile strength of carbon fibers, is the increment of cross-section of the coated fibers as a result of coating by nickel. Nickel tensile strength is much less than carbon fibers, therefore the average tensile strength of two materials together would be less than carbon fibers alone. This conclusion could be driven from both theoretical and experimental curves, while both are lowering as a result of increment of coating thickness although with different rates.

According to Fig.10, the difference between theoretical calculated value and empirical results gained for carbon fiber tensile strength has increased. At the thickness of coating of 0.2 $\mu$m, the strength of nickel coated carbon fibers is 81% of the theoretical value and at thickness of 1.1 $\mu$m is only 66% of the theoretical calculated value. It means that the difference between the theoretical calculated strength and the empirical results

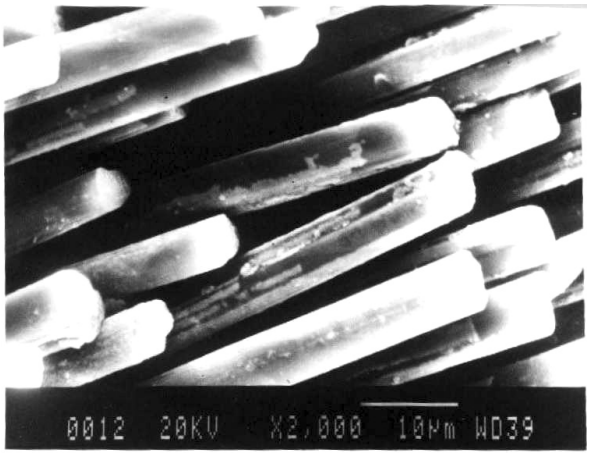


**Fig. 5** SEM micrograph of carbon fibers coated with 0.2 m thickness of nickel.

**Fig. 6** The longitudinal SEM micrograph of carbon fibers coated with 0.2 m thickness of nickel.

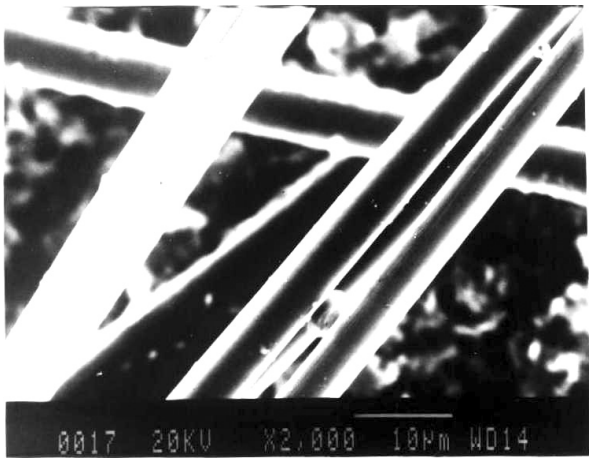


**Fig. 7** The longitudinal SEM micrograph of carbon fibers coated with 0.5 m thickness of nickel.

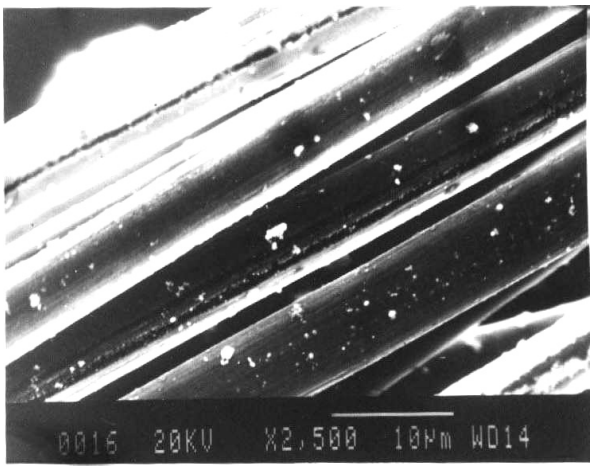


**Fig. 8** The longitudinal SEM micrograph of carbon fibers coated with 0.8 m thickness of nickel.


**Fig. 9** The longitudinal SEM micrograph of carbon fibers coated with 1.1 m thickness of nickel

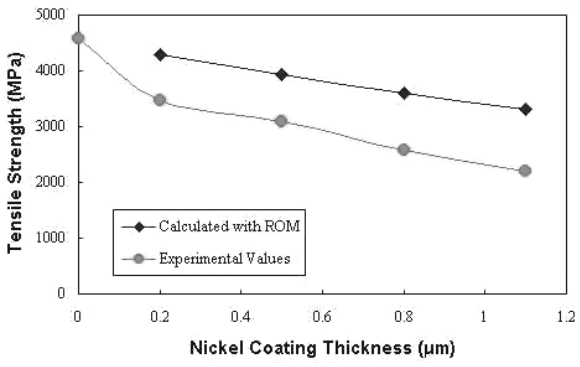


**Fig. 10** Tensile strength of the nickel coated carbon fibers as a function of thickness of nickel.

gained from carbon fiber tensile tests, has increased from 19% to 34% as a result of increase in coating thickness from 0.2 $\mu$m to 1.1 $\mu$m. This difference suggests that formation of nonuniform coating on the surface of fibers might causes stress concentration and reduction of overall strength.

## 4. CONCLUSION

1- Electroless method could be applicable for uniformly nickel coating of the carbon fibers.
2- Minimum thickness required to form a uniform coating of nickel on surface of carbon fibers is approximately 0.5 $\mu$m. Coating thickness less than 0.5 $\mu$m, could results the formation of nonuniform coating on surface of the fibers and coating of carbon fibers by nickel at higher thickness also will result in dendritic growth of coating and so nonuniformity on surface of the fibers.
3- Nickel coating will decrease the tensile strength of the carbon fibers. This decrease, is proportional with nickel coated layer thickness.
4- If the coating is nonuniform the chance of stress concentration increases and therefore the strength decreases.

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