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# Removing nickel from nickel-coated carbon fibers

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**Abstract.** Conductive fibers/yarns are one of the most important materials for smart textiles because of their electrically conductive functionality combined with flexibility and light weight. They can be applied in many fields such as the medical sector, electronics, sensors and even as thermoelectric generators. Temperature sensors, for example, can be made using the thermocouple or thermopile principle which usually uses two different metal wires that can produce a temperature-dependent voltage. However, if metal wires are inserted into a textile structure, they will decrease the flexibility properties of the textile product. Nickel-coated Carbon Fiber (NiCF), a conductive textile yarn, has a potential use as a textile-based thermopile if we can create an alternating region of carbon and nickel along the fiber which in turn it can be used for substituting the metallic thermopile. The idea was to remove nickel from NiCF in order to obtain a yarn that contains alternating zones of carbon and nickel. Due to no literature reporting on how to remove nickel from NiCF, in this paper we investigated some chemicals to remove nickel from NiCF.

## 1. Introduction

Smart textiles have become an important sector in the textile field due to its numerous potential applications for wearable technologies. Conductive fibers/yarns are one of the most important materials in this area because of their electrically conductive functionality, flexibility and light weight that can be applied in many fields such as the medical sector, electronics, sensors and even thermoelectric generators. As an example, temperature sensors can be made by using the thermocouple principle whereby the combination of two different metal wires produces a temperature-dependent voltage.

The use of metallic wires for thermocouple or thermopile in textiles has been reported by many authors such as follows. Jones used a metallic Type T thermocouple inserted in woven and knitted cotton fabrics and studied the possibility to make a textile-based temperature sensing device [1]. A heat flux sensor based on the thermopile principle and embedded into a textile structure has been reported in various scientific publications. In those works, metal wires made of constantan (Cn) with copper (Cu) coating via electrochemical deposition were inserted into a woven fabric and then etched in such a way that Cn-Cu junctions were obtained [2–4]. Since the flexibility of the textile is an important concern, researchers also investigated the thermocouple principle applied on textile for some applications using conductive polymers [5,6].



Nickel-coated carbon fiber (NiCF) is one of conductive fibers available in the market. It has a number of applications such as electrostatic dissipation [7] and electromagnetic interference (EMI) shielding [8,9].

In order to obtain a yarn that contains alternating regions of carbon and nickel, one of the ways is to start from a nickel-coated carbon fiber which is then alternately covered with something that is resistant to the etching chemicals and then etched the nickel chemically. In this paper, we investigated several chemicals to remove nickel from NiCF since to the best of our knowledge there is no literature reporting on this subject.

## 2. Experimental

In the experiment, we used nickel-coated carbon fiber (Tenax<sup>®</sup>-J HTS40 A23 12K 1420tex MC) which was purchased from Toho Tenax Europe GmbH, Germany. In this paper the Tenax<sup>®</sup>-J will then be called NiCF. The number of NiCF filaments was 12000 filaments and the linear mass density was 1420 tex.

Chemicals used in this experiment were ammonium persulfate (Fluka), hydrochloric acid/HCl (Sigma Aldrich) and hydrogen peroxide/H<sub>2</sub>O<sub>2</sub> (Chem Lab). They were all analytical reagents. The removing process of nickel in this experiment is also called the stripping process. Ammonium persulfate is typically used to dissolve a number of metals such as copper, cobalt, iron, zinc, magnesium and nickel [10]. Acid (sulfuric acid) and hydrogen peroxide solution can be used to remove copper in the manufacturing process of PCBs (printed circuit boards) [11], but in this work we used hydrochloric acid (HCl) and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) solution to remove nickel from NiCF.

Removing nickel was carried out in a glass beaker. NiCF sample was dipped in the chemical solution as mentioned in Table 1 (section Result and discussion) below. After the dipping process, the samples were rinsed under running water and put between blotting paper to remove excess water several times and finally air dried at room temperature for 24 hours.

Microscopic images of the fibers were taken to observe the fiber surface. Images were taken with an Olympus BX51 microscope equipped with Cell<sup>^</sup>D software.

The linear electrical resistance was measured based on the four wire method. Each sample was placed in a clamping device (Burster Type 2381, Germany). The voltage was measured after a DC current was introduced to the sample at different length of measurement from 5 cm up to 20 cm with intervals of 5 cm. The resistance in each length was calculated from the following equation:

$$R = \frac{V}{I} \quad (1)$$

Where  $R$  is the resistance in ohm ( $\Omega$ ),  $V$  is the voltage in volt (V) and  $I$  is DC current introduced to the fiber in ampere (A). The linear resistance was taken from the slope of the linear graph of resistance versus distance.

## 3. Results and discussion

In this work, we aim at removing nickel from NiCF by using chemicals which are commonly used in PCB manufacturing processes. Table 1 shows the chemicals, the conditions and the visual remarks during this nickel removing process.

**Table 1.** Chemicals, conditions and results of the nickel removal process of NiCF

Chemicals	Temperature	Dipping time	Visual remarks
220 g/L ammonium persulfate	40°C	30 min	-
250 g/L ammonium persulfate	40°C	30 min	-
350 g/L ammonium persulfate	40°C	30 min	-
500 g/L ammonium persulfate	40°C	30 min	-
37% HCl and 3% H <sub>2</sub> O <sub>2</sub> (1:1)	RT	30 min	+
37% HCl and 6% H <sub>2</sub> O <sub>2</sub> (1:1)	RT	30 min	+++
37% HCl and 10% H <sub>2</sub> O <sub>2</sub> (1:1)	RT	30 min	++++

(-) No sign of nickel removal from NiCF, no colour change

(+) A sign of nickel removal with very little colour change

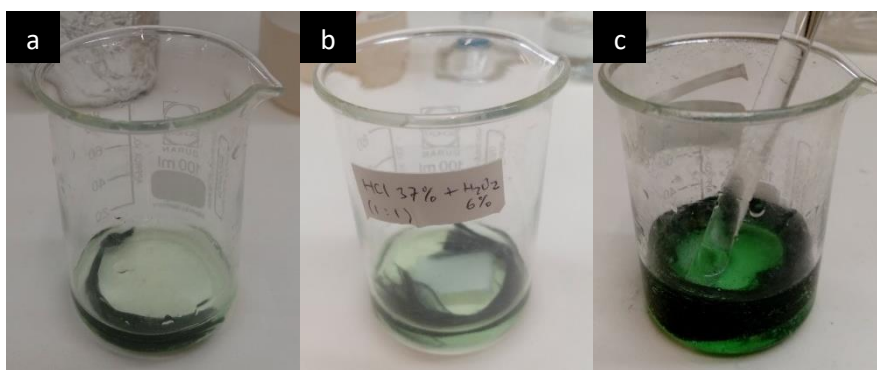
(+++ ) A sign of nickel removal with significant colour change

(++++ ) A sign of nickel removal with very clear colour change

### Visual observation

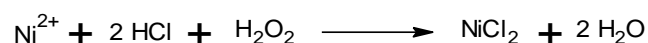
Carbon fibers are normally black, while nickel-coated carbon fibers are brown in colour due to the nickel layer on the surface. So, logically if the nickel is completely removed from the NiCF it will leave a black colour from the carbon fibers remaining. In this experiment colour change on NiCF filaments dipped in 220 – 500 g/L ammonium persulfate solutions at 40°C for 30 minutes were not observed and visually there was no colour change in the ammonium persulfate stripping bath. This means that ammonium persulfate cannot dissolve nickel from NiCF.

In contrast, when using 37% HCl and 3-10% H<sub>2</sub>O<sub>2</sub> (1:1) as stripping chemicals for the NiCF, different intensities of colour change from colourless to green in the stripping baths were observed. The 37% HCl and 10% H<sub>2</sub>O<sub>2</sub> (1:1) stripping solution shows the highest intensity, indicating that a huge amount of nickel was dissolved into the solution. This phenomenon can be seen in Figure 1.



**Figure 1.** NiCF filaments after 30 minutes dipping in: (a) 37% HCl + 3% H<sub>2</sub>O<sub>2</sub> (1:1); (b) 37% HCl + 6% H<sub>2</sub>O<sub>2</sub> (1:1); (c) 37% HCl + 10% H<sub>2</sub>O<sub>2</sub> (1:1).

Colour changing from colourless to green was attributed to nickel chloride. The following is the possible reaction of stripping nickel with HCl and H<sub>2</sub>O<sub>2</sub>.



Similarly, Figure 2 shows that after stripping with 37% HCl and 3-10% H<sub>2</sub>O<sub>2</sub> (1:1) the filaments are turning to black. The higher the concentration of H<sub>2</sub>O<sub>2</sub>, the darker the colour of the NiCF filaments. A drastic colour change of the NiCF filament was observed when dipped in 37% HCl and 6% H<sub>2</sub>O<sub>2</sub> (1:1) (Figure 2.c). It also confirms that nickel was oxidized and dissolved by hydrochloric acid and hydrogen peroxide.



**Figure 2.** NiCF filaments: (a) before stripping; after stripping in (b) 37% HCl +3% H<sub>2</sub>O<sub>2</sub> (1:1); (c) 37% HCl +6% H<sub>2</sub>O<sub>2</sub> (1:1); (d) 37% HCl +10% H<sub>2</sub>O<sub>2</sub> (1:1). Rulers show main unit in cm.

From the microscope images in Figure 2, it is seen that the diameter of the NiCF filament becomes smaller after stripping in hydrochloric acid and hydrogen peroxide. The higher the concentration of hydrogen peroxide, the smaller the diameter of the NiCF filament. These images also confirm that when the nickel layer becomes thinner, the colour of NiCF filament becomes more black. However, even in the concentration of 10% hydrogen peroxide, a trace of nickel is still seen on the surface of the NiCF filament indicating that nickel was not completely removed (Figure 2.d).

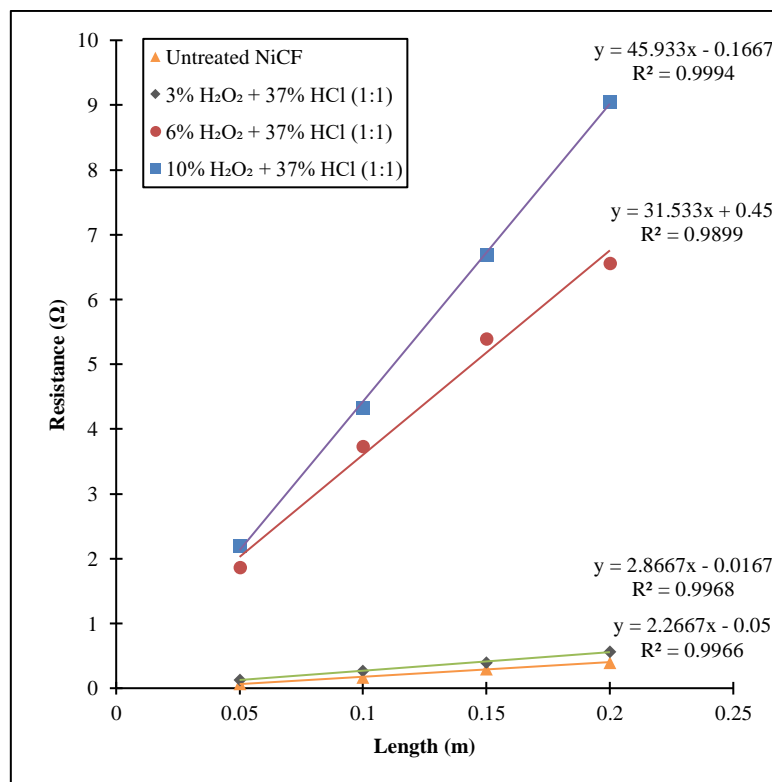
### *Linear electrical resistance*

The linear electrical resistance was measured to see the NiCF's electrical characteristics after the stripping process. The higher the concentration of hydrogen peroxide, the higher the linear electrical resistance of the NiCF filament become, as can be seen in Table 2 and Figure 3. This phenomenon is related to the decreasing of nickel layer thickness on the filaments that causing the linear electrical resistance to be higher. It agrees with the work of Pierozynski that the resistance of nickel-coated carbon fiber is getting lower as the weight percent of the nickel deposited on the fiber is higher [7]. In other words, nickel can increase the conductivity of the carbon fiber.

Similar to the colour change, an extreme difference in linear electrical resistance can be seen on the NiCF treated in 37% HCl and 6% H<sub>2</sub>O<sub>2</sub> (1:1) solution (31.53 Ω/m) compare to the untreated (2.27 Ω/m) as well as 37% HCl and 3% H<sub>2</sub>O<sub>2</sub> (1:1) treated (2.87 Ω/m) sample. This implies that nickel was dissolved in 37% HCl and 6% H<sub>2</sub>O<sub>2</sub> (1:1) solution so fast that a lot of nickel was removed from the surface of the carbon fiber. The sample treated in 37% HCl and 10% H<sub>2</sub>O<sub>2</sub> (1:1) has the highest linear electrical resistance (45.93 Ω/m).

**Table 2.** Linear electrical resistance of the samples

Samples	Linear electrical resistance ( $\Omega/m$ )
Untreated NiCF	2.27
37% HCl and 3% H <sub>2</sub> O <sub>2</sub>	2.87
37% HCl and 6% H <sub>2</sub> O <sub>2</sub>	31.53
37% HCl and 10% H <sub>2</sub> O <sub>2</sub>	45.93

**Figure 3.** Linear electrical resistance of NiCF filaments

#### 4. Conclusion

In summary, we successfully removed nickel from nickel-coated carbon fibres (NiCF) with chemicals. In this study, the 37% HCl and 10% H<sub>2</sub>O<sub>2</sub> (1:1) solution could dissolve much nickel from the NiCF filaments, that can be seen from the colour change of the filament and of the stripping solution. The decrease in fiber diameter and the increase in linear electrical resistance of the NiCFs confirm that nickel was removed from the NiCF filaments. In contrast, ammonium persulfate could not remove nickel from NiCF. In further studies, the 37% HCl and 10% H<sub>2</sub>O<sub>2</sub> (1:1) solution will be applied to selectively strip the NiCF filaments and make a thermopile.

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