Microstructural Characterization of Electroformed Nickel and Its Composites

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Abstract

In the present work the nickel metal matrix was developed by electrochemical forming technique, were the nickel matrix is built around the reinforced carbon fiber in various thicknesses and fiber volume fractions. The metallographic analysis indicate that the experimental depositing condition have a dominating effect on the observed composite microstructure. The grain size of the deposited nickel is found (from the microstructure) to vary inversely with the amount of the depositing electrical current density. Also the temperature of the solution seem to reflect quite well on the microstructure of the metal matrix, were relatively high and low solution temperature produced rather refined grain structure, while an intermediate temperature of (50ºC) is found to deposited courser grain size.

Secondary thermal and mechanical treatments are found to modify the microstructure in a way that higher annealing temperatures tend to enhanced the grain growth process and tend to reduced apparent porosity at (650ºC), similarly the forging of the electroformed composites made at different temperature tend to reduced the internal porosity, by a notable ratio, depending on the temperature of the forging process.

Keywords: Metal matrix composites: Electroforming: Nickel-Carbon fiber composites

خصاص التركيب الدقيق للنيكل المشكل كهربائيا مع متراكباته

الخلاصه

في هذا العمل تم تطوير وسط النيكل المعدني باستخدام تقنية التشكيل الكهروكيمياوي, اذ تم بناء وسط النيكل حول الياف الكاربون التي استخدمت كمواد تدعيم, باسماك مختلفه و بكسور حجميه مختلفه. اظهرت التحليلات المجهريه ان لشروط الترسيب التجريبيه تاثير بارز على التركيب الدقيق للمتراكبات. فقد وجد ان الحجم الحبيبي للنيكل المرسب (من التركيب الدقيق) تتغير عكسيا مع مقدار كثافة التيار الكهربائي للترسيب. كذالك فان لدرجة حرارة محلول الترسيب تاثير ينعكس بصور واضحه على التركيب الدقيق للاساس المعدني (matrix), اذ ان الترسيب عند درجات حراره المحلول المرتفعه و المنخفضه نسبيا ينتج عنها تركيب حبيبي ناعم, بينما الترسيب عند درجة حراره متوسطة (50°C) ينتج عنه حجوم حبيبيه كبيره.

كما وجد ان للمعاملات الثرمو ميكانيكيه اثر في تغير التركيب الدقيق بطريقه اسهمت في خفض نسبه المسامات عند درجة حرارة (650°C), و بالمثل فان عملية الطرق التي اجريت عند درجات حراريه مختلفه على المتراكبات المشكله كهربائيا تميل الى خفض نسبه المسامات بنسب ملحوضه اعتمادا على درجة حرارة عملية الطرق.

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1. Introduction

The use of a modified nickel-electroplating process for electroforming metal matrix composites, is rather unique and may be considered as new process for the development of such composites which is otherwise impossible to be developed using traditionally known techniques [1,2]. Such products may be indispensable for a number of industrial applications, like textile, communication, aerospace, building products, electronics, automotive, photo-copying and other indusries. Electroforming technique has a number of application and rather popular as in the case of nickel matrix since nickel is considered to be strong, tough, and corrosion/erosion resistant [3,4,5]. In this process the mechanical nature of the deposited nickel or nickel composite, may be controlled over a wide range, by adjusting the electroplating and alloying parameters [5,6]. By using a coprecipitation process or by incorporating particulates [7] and fibers [8,9,10] within the bulk.

Several investigators [1,11,12,13] have found that the structure of electroformed metal matrix is affected by the depositing conditions (pH, agitation, additives, current density and solution temperature)[14]. However no one has investigated the possibility of producing such composites. The use of such a contemporary technique for developing metal matrix composites may be considered as rather advantageous when compared with classical metallurgical processes, since it dose not involve the use of high temperatures. For instance, a copper matrix may be reinforced with carbon steel fiber [10], or a steel mesh [15], and tungsten fibers, and similarly the same process may be used for nickel and nickel alloy matrices [16].

In the present work, it is aimed at developing a nickel matrix composites reinforced with carbon fibers. It is also aimed at investigating the general microstructure of the developed composites. And the way it is affected by varying the electroplating parameters, like current density, solution temperature and the degree of mechanical agitation. Moreover, to study the effect of thermomechanical treatment on the final microstructure of the resulting composites.

2. Experimental techniques

Plain nickel sheets (149*50*3mm) were deposited in a sulfate nickel watt's bath (Table (1)), using a copper strip as a substrate. The copper substrate is normally removed from the electrodeposited plain nickel by dipping both of them in solution containing (10:1) g/L chromic acid and sulfuric acid respectively. This solution would dissolve the copper substrate living the deposited nickel free. The carbon fiber reinforced nickel composites were formed by depositing nickel ions on the carbon fiber stretched-fixed from both ends inside a hollow polymeric frame. Proper attention was taken to ensure good electrical contact between the carbon fibers and the cathode, using a metallic strip inside the plastic frame dipped in the bath. The whole process is performed in a standard electroplating chemical cell as shone in fig(1).

All nickel matrix composites produced in this manner will subjected to a secondary thermomechanical treatment. The strip sample was annealed at temperature (450,550,650°C) for a period of (1 h), most of the annealed samples were forged with hot using a hydraulic press. Some of these hot forged strips underwent in multiple past hot forging process, to investigate the reflection of such treatment on the microstructure of the specimen. In each forging event, a force of (10 ton) was applied to the hot specimen for a period of (1 min), some preparation was taken to insure the thermal insulation of the specimen during the forging process.

The microstructure of the composites was investigated using an optical microscope connected to a digital camera. The lateral cross sections which were investigated microscopically, where those of the composites mounted in a resin and prepared using standard polishing technique.

3. Results and Discussion

3.1 Microstructure of Electro-formed Plain Nickel

Fig(2) exhibits microphotographs showing the grain size of plain nickel deposited at various solution temperatures (bath temp.) while the electric current density passing through the bath was fixed at the value of (0.05 Amp/cm^2). The columnar grain structure is shown to be in uniaxial alignment parallel to the direction of depositing current. It may be noticed that a solution temperature at of (50°C) is resulted in a grain size larger than those deposited at (40 and 60°C). This may be due to the fact that such deposition processes normally liberates hydrogen gas. It is also know the final grain size depend on the process of hydrogen liberation in addition to the fact that some of the liberated hydrogen may remain in the bulk as an inclusion. However, higher solution temperature is found to liberate less hydrogen gas [11], thus leading to less hydrogen inclusion in the bulk which in turn reduces the small grain size. However, at lower solution temperature (40°C) the hydrogen inclusion may inhibit the grain growth process [17], thus, the hydrogen here acts as grain refiner (fig(2a)). At solution temperature of (50°C), this inhibition effect becomes smaller and less effective leading to relatively coarse grain structure as shown in fig(2b). At temperatures as high as (60°C) a further decrease in hydrogen liberation brings about a higher nickel deposition rates [11], as this shows the increased deposition rate leads to a higher nucleation rates, resulting in smaller grains (Fig(2c)).

Fig(3) shows the influence of deposition current density on the final microstructure of deposited nickel, with the solution temperature being kept constant, it is notable that increasing current densities is resulting finer grain structure. One may conclude from this figure that higher deposition current densities would enhanced the deposition rate, resulting in a finer grain stricture, which may have resulted from the increased rates of nucleation.

3.2Microstructure of Ni-C Composite

Fig(4) shows a cross section of the fiber reinforced nickel composites, where three main regions are recognizable. The first is the black circular spots representing the reinforced carbon fiber. The second

is the triangular black spots representing the vacancies or the voids which are not filed with deposited nickel. The redially aligned bright grains represent the deposited nickel accumulated on the carbon fiber from all direction, thus containing the fiber in a cylindrical manner. The void regions are those regions formed because deposited nickel ions were unable to reach. This radial deposition process may be understood much better by studying fig(5) and fig(6), where it is clearly illustrated how the nickel is radially accumulated around individual carbon fibers. The radius of this nickel cylindrical sheath gets bigger and bigger until these nickel cylinders surrounding each carbon fiber would overlap with each other, forming the bulk of the nickel matrix. This may also explain how the voids are formed when a number of nickel cylinders overlap, thus shielding any void present between them. As the deposition process begins, the total surface area of the fiber is supposed to be at its minimum, hence the deposition current density is at its maximum value, after this process is carried out the outside surface area of the carbon fiber gets continuously larger and larger, hence the deposition current density drops down continuously with increasing surface area [18]. Accordingly, accumulated of the nickel will have a continuously variable grain size. Those grains directly attach to the carbon fibers are expected to be finer or much smaller than those located at the circumference of the final nickel cylinder, as illustrated clearly in fig(7).

3.3 The Effect of Secondary Thermal and Mechanical Treatment on the Microstructure

A selected group of nickel–carbon fiber composite was annealed and forged in the aim of eliminating the residual porosities remnant in the bulk of the composite. Fig(8) shows the preforged specimen heat treated at (450,550,650 °C). A comparative study of these microphotographs exhibit the grain growth process initiated by the heat treatment. The grain structure of the composite annealed at (650 °C) is clearly much coarser than those of specimen annealed at (450 and 550°C).

Fig(9) exhibits the microstructure of similar specimens but forged with different number of passes. By comparing the two sets of microstructure fig(8) and fig(9) we may notice that there is a little difference between the forged and preforged composite which have been annealed at (450°C). However, there is a difference between those that are annealed at (550,650°C), where the grain structures seem to get coarser and coarser upon forging. This is specially noticeable in the case of specimen annealed at (650°C) there the grain look much coarser and there is a sizable number of crack formation initiated by the forging process. This would indicate how the fine grains structure would resist as compared to that of the coarse grain structure.

4 Conclusions

The deposition parameters which are the electrical current density and solution temperature exhibited a strong and direct relation to the final microstructure of the composites. Uniform and continuous depositing of nickel was given to carbon fiber

irrespective to depositing condition except in the grain structure.

The thermomechanical treatments seem to enhance the microstructure of nickel matrix by reducing the porosity. Further forging steps seem to damage the microstructure and initiate cracks within the matrix, and higher annealing temperatures may have started a recrystallization process associated with the initiation microcracks.

5 References

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Table (1) Composition and operation conditions for Nickel Watt's bath

Electrolyte composition g/liter	
Watt's nickel	
$NiSO_4.6H_2O$	330
$NiCl.6H_2O$	45
H_3BO_3	30
H_2O_2	0.5 per 24h
Operation Condition	
Temperature	40-60°C
Agitation	mechanical
Cathode current density	0.025-0.075 A/dm^2
Anode	soluble nickel
pH	4

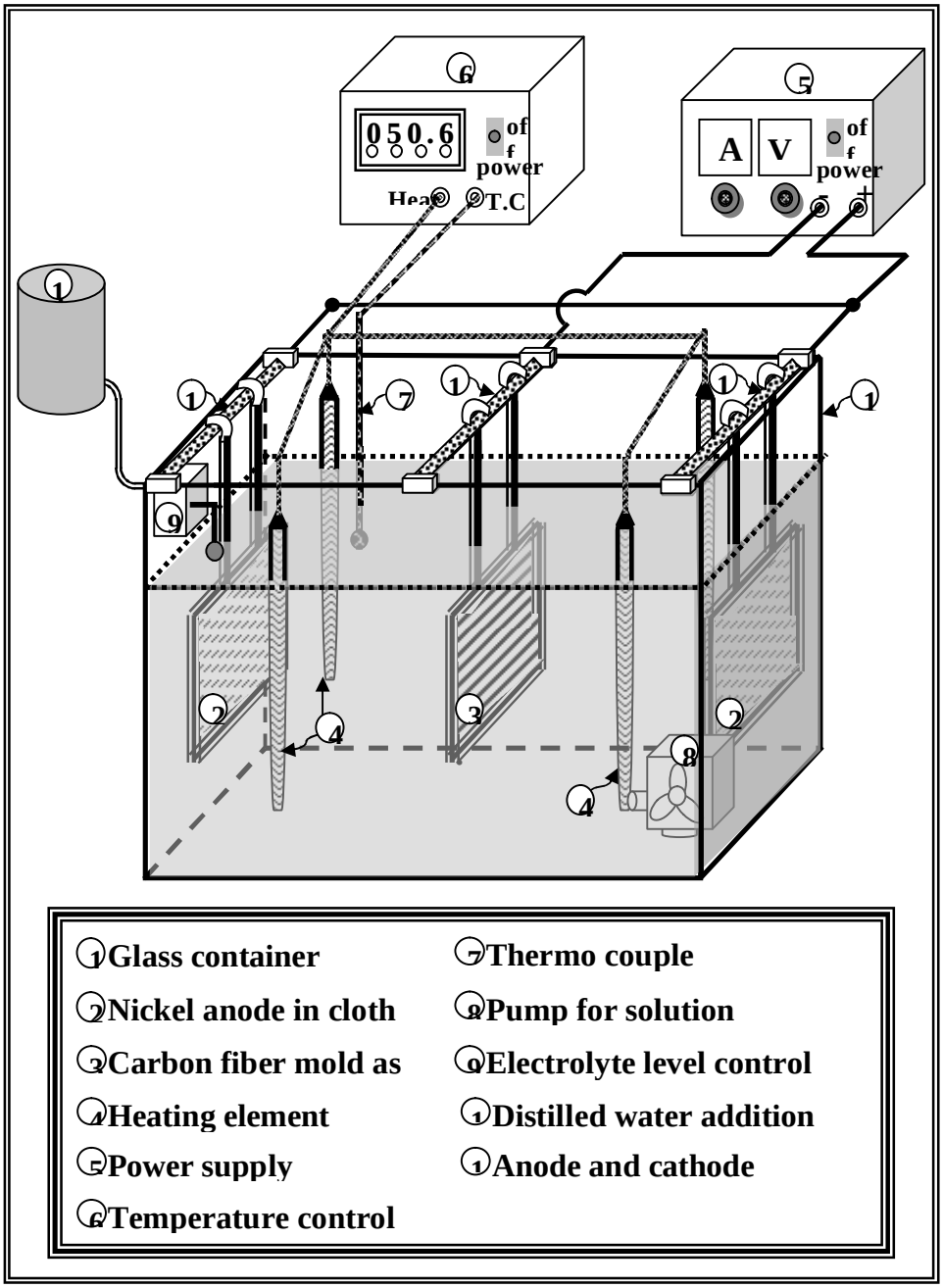


Figure (1) Electroforming cell

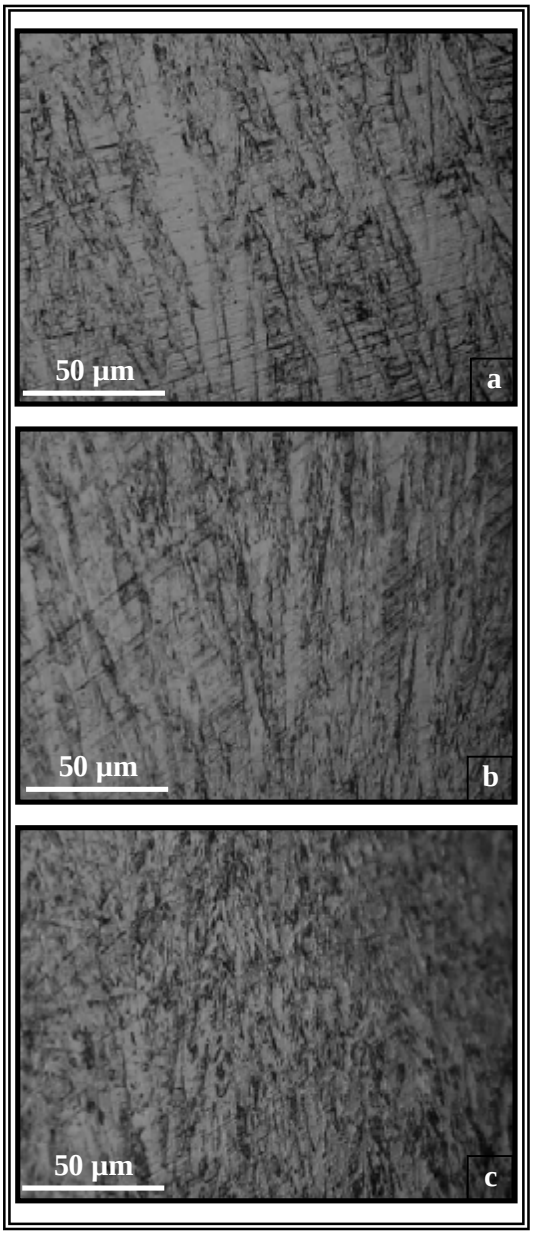


Figure (3) The microstructure of electroformed nickel at temperature of value (50°C) and deposited current densities (a) 0.025 Amp/cm^2, (b) 0.05 Amp/cm^2 (c) 0.075 Amp/cm^2

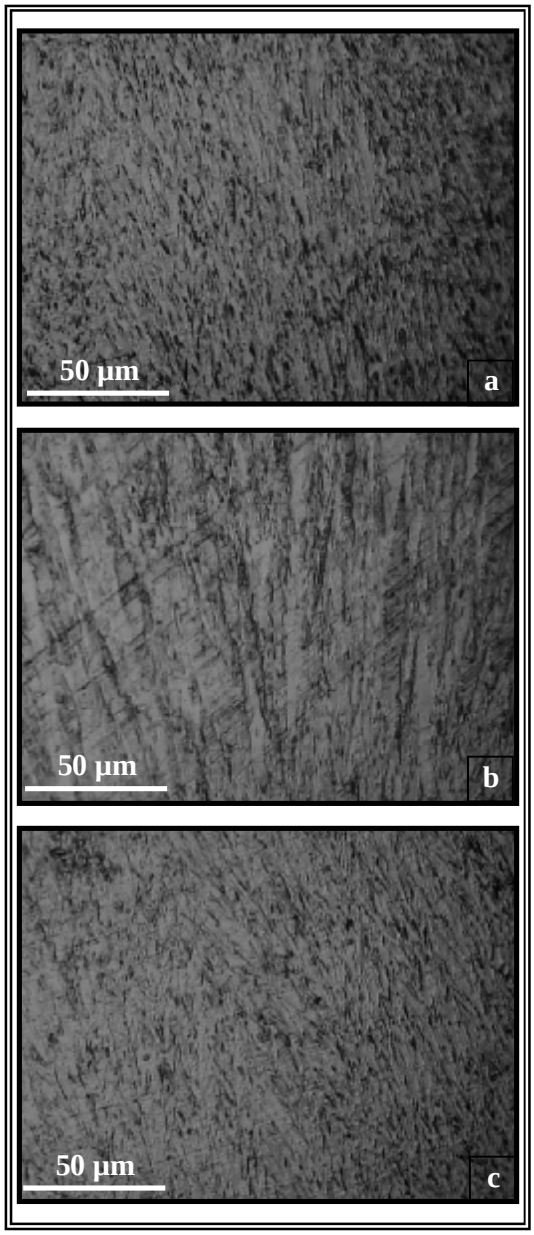


Figure (2) The microstructure of electroformed nickel at current density of value (0.05 Amp/cm2) and solution temperature (a) 40°C, (b) 50°C (c) 60°C

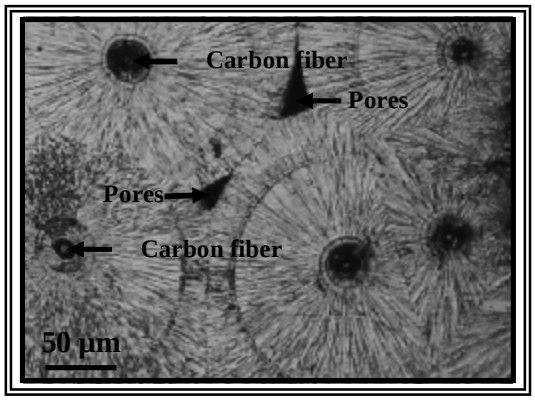


Figure (4) Ni-C composite cross section shows the three regions

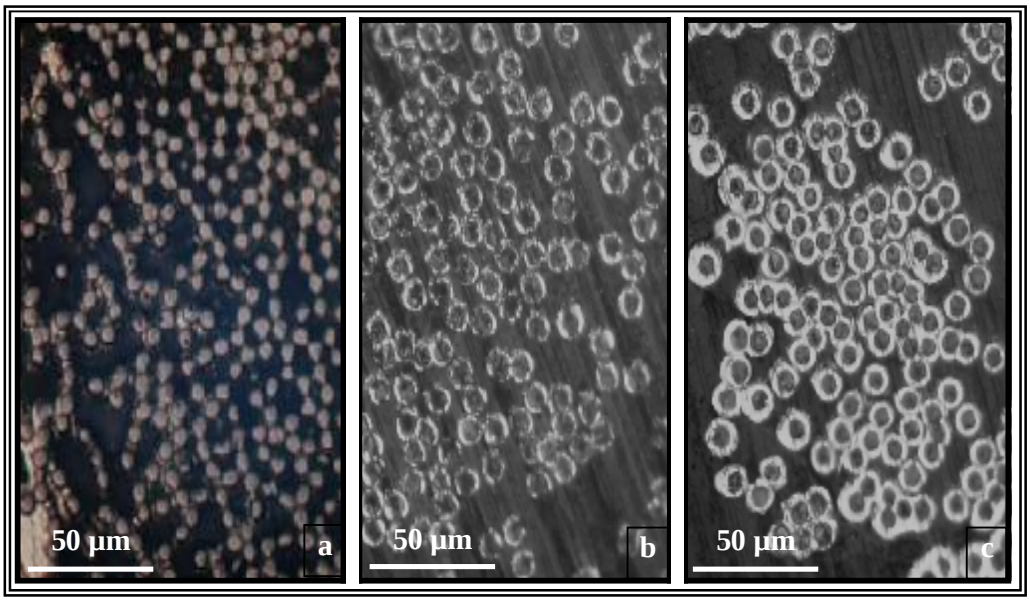


Figure (5) Cross-section of depositing nickel on individual carbon fibers for(a) 0 min (b) 15min (c) 30min

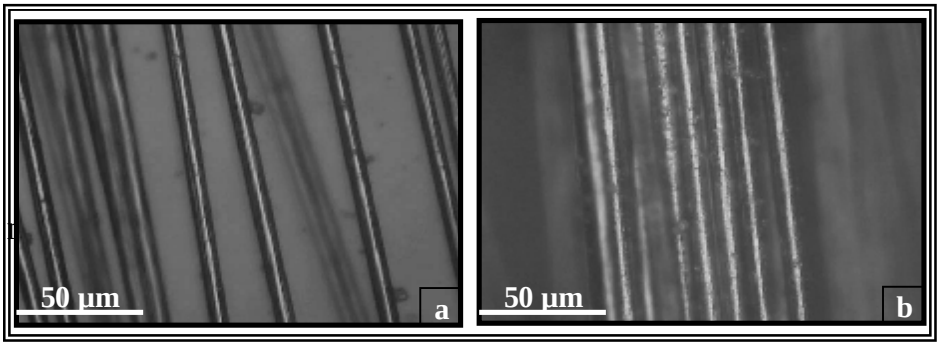


Figure (6) shows the comparison between the outer surface of the carbon fiber before and after deposition.

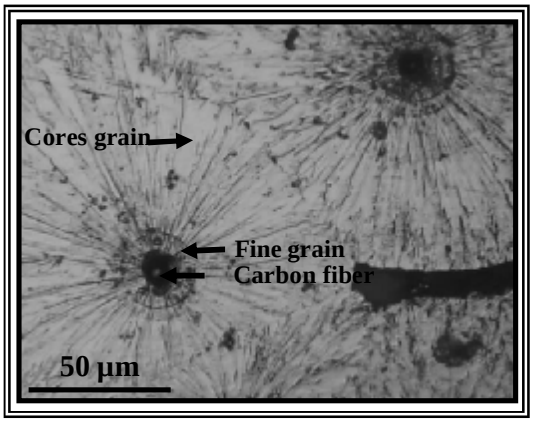


Figure (7) Fine columnar grains surrounding the carbon fiber with radial direction in Ni-C composite cross section.

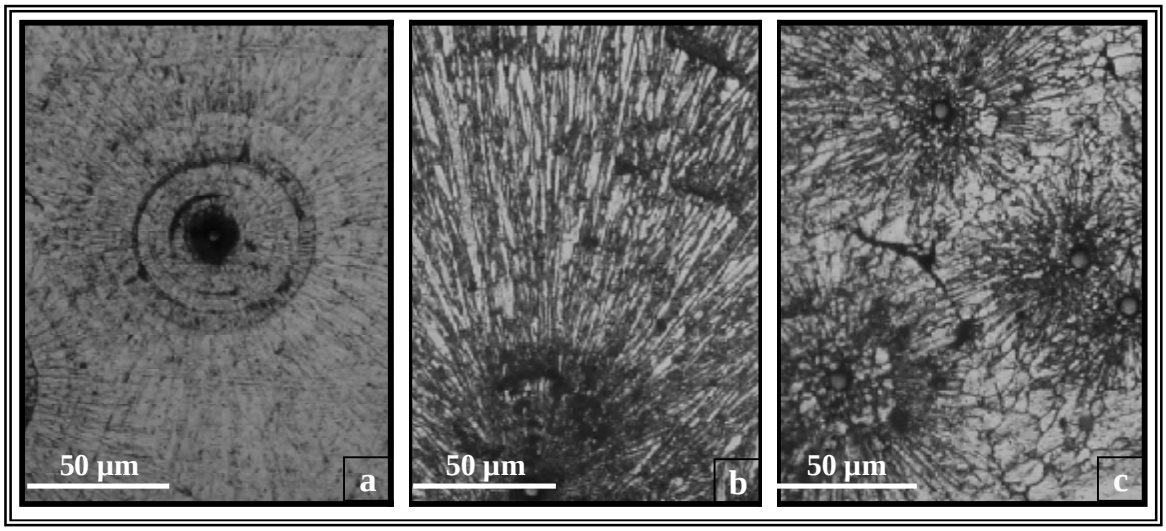
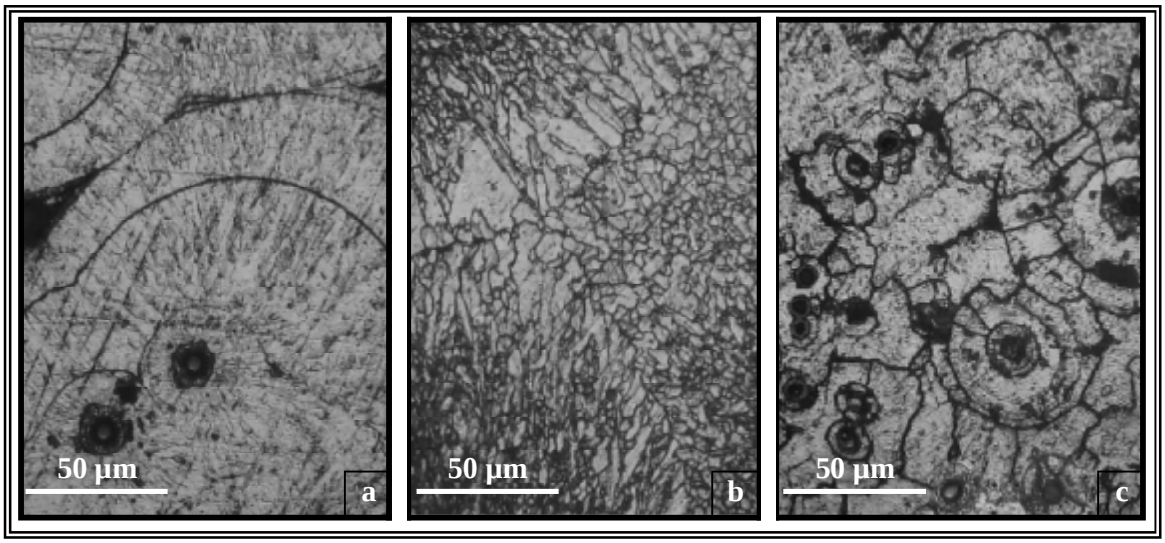


Figure (8) The microstructure of Ni-C composite preforging at heat treatment temperature (a) 450°C (b) 550°C (c) 650°C.



Figure(9) The microstructure of Ni-C composite with three forging passes at heat treatment temperature (a) 450°C (b) 550°C (c) 650°C.