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# THE ELECTROLESS NICKEL PLATING OF SILICON CARBIDE AND ALUMINIUM POWDER MIXTURES

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## ABSTRACT

In this study, silicon carbide, which have been produced in laboratory and aluminum powders were tried to coat with nickel by using electroless nickel plating methods.

Aluminium particles are not as well covered as the SiC. All the nickel lines were very broad which reflects the small crystallite size but the calculated lattice parameter shows quite good agreement with the literature value of 0.35238 nm.
If this work succeeds, this method can be used for preparing ceramic-metal matrix composites for the future works.

The results were obtained by the x-ray diffractometer analysis and scanning electron microscope analysis techniques.

**Key Words :** Electroless Nickel Plating, Silicon Carbide

# SİLİSYUM KARBÜR VE ALÜMİNYUM TOZLARININ ELEKTRİK AKIMSIZ NİKEL KAPLANMASI

## ÖZET

Bu çalısmada, elektrik akımsiz nikel kaplama metodu kullanılarak, labaratuvarda uretilmis silisyum karbür tozları ile aluminyum tozlarının nikel kaplanması denenmistir.

Alüminyum tozlarının silisyum karbür tozlarına nazaran tam kaplanamadığı görülmüştür. X-ışını analizinde nikelin açık bir şekilde elde edildiği ve hatta hesaplamalar sonucu nikelin latis parametresi 0.3523 nm olduğu ki bu

rakamın literatüre çok yakın olması bu çalışmanın amacına ulaşacağına işaret olmuştur.

Sayet bu calısma basarılı olursa; ilerdeki calismalarda elektrik akımsız nikel kaplama metodu seramik-metal kompozitlerin hazırlanmasinda kullanılabilir. Sonuclar x-ışınları analizleri ve taramalı elektron mikroskobu kullanılarak elde edilmistir.

**Anahtar Kelimeler:** Elektrik Akimsiz Nikel Kaplama, Silisyum Karbur

## 1. INTRODUCTION

Electroless nickel is not a new product having been used in several industries since 1950. It has been used for many different applications, since its development in the 1960's. With the introduction of functional, consistent electroless nickel coatings, and with the increased importance being given to equipment costs and reliability, this process is now being considered for increasingly uses. Electroless nickel coatings are today one of the fastest growing segments of the metal finishing industry and it seems useful to expand this technology to coat ceramic powders.

Electroless nickel plating, in contrast to conventional plating, does not use electric current to produce a deposit but is a chemical reduction. The electroless process, also called autocatalytic deposition, removes one of the major drawbacks of electroplating, namely the difficulty or in some cases, impossibility, of uniformly plating irregularly shaped parts or components. An electroless plating solution produces a deposit wherever it contacts a properly prepared surface, without the need for conforming anodes and complicated fixturing. Since the chemically reduced bath maintains a uniform deposition rate, the plater has precise control over deposit thickness simply by controlling immersion time. This uniformity and control open up new choices for the design engineer in selecting plated finishes for his products.

Brenner and Riddell [1] in 1946 did the seminal work to allow the technological development of a non-electroplating method to be developed from their work on electroplating of Ni-W alloys from citrate baths. In the duration of 25 years, this engaging coating method has gained increasing significance and ever wider areas of use. It complements in an important way the method of galvanic nickel plating by producing coating with mechanical properties between those of electro-chemical nickel and hard-

chrome coatings. The different chemical processes used in electroless nickel plating are in the present day used for repair purposes as well as protection against corrosion and wear of new parts of machines. They offer special advantages for coating inner surfaces of vessels, pipes, bored holes, etc, where galvanic processes can only be used at great cost or where they fail entirely. Chemical plating has its place in the metallisation of different insulators (plastics, glass, ceramics) as well as of semi-conductors (Germanium, Silicon, etc...). In summary advantages of electroless nickel plating are:

- Superior chemical and atmospheric corrosion resistance,
- Good wear resistance,
- Freedom from porosity,
- Controlled hardness, heat treatable to 65-69 Rockwell C,
- Infinite throw, eliminating auxiliary anodes,
- Overall uniformity of deposit with no edge build-up,
- A solderable, hard coating for light metals,
- Natural lubricity, providing excellent release properties,
- Self polishing effect in molding operations,
- Anti-galling characteristics for mating metallic surfaces,
- A sound base coating for subsequent finishing operations, and
- Selective plating salvages worn or mix-machined parts.

In previous work at Bradford [2], the electroless deposition of nickel on ceramic powders was developed and from a knowledge of the Ni-Al phase diagram it was noted that a potential existed for a transient liquid phase to be present during the time the system was moving towards equilibrium. This was convenient because the method used to produce sub-micron sized AlN particles, namely reactive arc melting, always contained some un-reacted aluminium. A successful method was developed that required only low temperatures, 650-1000C, for very short sintering times to obtain specimens >98 % theoretical density.

The literature on metal matrix-ceramic composites, particularly when SiC is the ceramic, is clear in demonstrating that the major problem is reaction at the interface to form silicides and carbides [3-5]. These brittle phases, in this most important region of a composite, can lead to poor mechanical properties [6,7]. To overcome this, such techniques as coating the SiC with carbon are employed and this increases process difficulty and cost etc... Thus the short reaction times and relatively low temperatures involved for the AlN-Al-Ni work, determined the starting point for this research.

## 2. MATERIALS AND METHODS

In this work, two different kinds of silicon carbide (SiC) powders were used. The first SiC powder with a mean particle size of 7 μm and a purity of 99.98 % was supplied by Good fellow Ltd., U.K. The second powder was made in laboratories on the way described as follows: 99.99 % silicon in lump form (Si) and carbon graphite (C) also lump, were crushed and pressed into a pellet about 4 grams, in weight. The pellet was melted on the cooled copper hearth of an arc furnace of special design [8].

In this work, aluminium powder with a mean particle size of 25 μm supplied by B.D.H. Ltd. U.K. was used.

In most of experiments, nickel was produced in situ by the electroless deposition methods using nickel chloride and hydrazine hydrate or sodium hypophosphite baths. During the electroless nickel deposition 35vol % ammonia solution was added constantly to keep the pH at 10. When the temperature reached 95 °C the solution gave pure nickel. All salts and reagents used were either Analar or general-purpose grade and were supplied by B.D.H. Ltd. U.K. The experimental procedure of electroless nickel plating carried out is described as follows:

Preparation of an electroless nickel-plating bath is usually based on one or two solutions. These are mixed with deionised water to give the correct dilution. The bath is raised to the correct temperature and pH is measured, being adjusted as required with sulphuric acid, caustic soda or ammonia and is then ready to use. In order to ensure optimum quality of an electroless nickel deposit, the following parameters of a deposition process must be monitored and, where possible, optimised:

- Bath temperature,
- pH,
- Nickel content,
- Reducing agent concentration,
- Stabiliser concentration and
- Metallic impurities.

In the literature, hundreds of nickel plating baths can be found. In some part of this study, three kinds of electroless nickel plating bath were used. Two of them are sodium hypophosphite baths and one of them is hydrazine bath. Hypophosphite-reduced electroless nickel plating solutions can be seen in

Table1. The composition of the solution for hydrazine electroless nickel plating is illustrated in Table 2.

Table 1. Hypophosphite-reduced electroless nickel plating solutions

| Composition | Bath 1 | Bath 2 |
|---|---|---|
| Nickel chloride* | 45 | 30 |
| Sodium hypophosphite* | 11 | 10 |
| Ammonium chloride* | 50 | - |
| Sodium hydoxy-acetate* | - | 10 |
| PH | 8.5-10 | 4-6 |
| Temperature (°C) | 90-95 | 88-95 |

*gr/lt

Table 2. Chemical composition for hydrazine electroless nickel plating.

| Chemicals | Conditions |
|---|---|
| Nickel Chloride, $NiCl_2.6H_2O$ | 100 g/l |
| Hydrazine hydrate, $N_2H_4.H_2O$, (20 % solution) | 80 ml/l |
| pH value (adjusted with 35 Vol ammonia solution) | 10 |
| Temperature | 95 °C |

SiC powder was mechanically mixed with Al powder in a drum mixer for 48 hours, composition was set to obtain a variety of volume fraction of ceramic by weighing the powders prior to mixing. Then, the electroless nickel-plating method, which already described earlier, was employed to plate the SiC+Al powder mixture so that a fine and homogenous distribution of nickel is deposited over the SiC+Al powder mixture. Powders prepared by this way were then washed with distilled water and acetone and dried in an oven at 100 °C for 24 hours. The powder was analysed by X-Ray and SEM.

## 3. RESULTS AND DISCUSSIONS

For producing SiC, using a tungsten electrode and argon gas of 99.99 % purity flowing through the heating chamber at pressure above 1 atm. and a flow rate of 40 ml per minute were used. This process produces an arc cast head consisting of β-SiC plus Si, which has to be removed from the cool areas of the furnace. The quality of this powder in terms of particle size and SiC content depended on the voltage at which the arc was operated. Optimum conditions were found to be 90-110 volt. βSiC + Si powder had a

mean particle size of approximately 25-50 μm produced by using 110 volt; while the βSiC +Si powder by 90 volt had a mean particle size of approximately 100-125μm and contained less βSiC content.

By comparing x-ray powder films of the mixture with prepared standards, the relative percentage of the two components was found. The standard silicon carbide powder was analaysed by X-Ray using a Hagg-Guinier powder camera for ½ hours using Cu $K_{\alpha 1}$ radiation. The x-ray analysis data of βSiC + Si powder which was produced by using 110 V is illustrated in Table 3. The SEM picture of this sample is illustrated in Fig.1 In addition, x-ray data of standard powder can be found in Table 4.

Most of the experimental work was done using a large volume fraction of SiC, which was kept constant throughout the series. Much of the SiC was obtained from the arc melting procedure which allready described in earlier chapter. The consistency of the nickel plating procedure led to conclusion that a constant percentage of nickel chloride was converted into a nickel layer on the aluminium and silicon carbide powders, which became black in appearance. Fig 2. shows that the nickel-coated particles are about 4 μm in diameter. However, the Ni appears to be agglomerated unevenly over SiC and Al powder particles. Extremely broad x-ray diffraction lines for the Ni point to the fact that the particles are themselves agglomerates that the crystal size is below 0.4 μm.

Aluminium particles are not as well covered as the SiC (see A in Fig 2). X-ray data are given in Table 5; as expected only three phases are existing. All the nickel lines were very broad which reflects the small crystallite size but the calculated lattice parameter shows quite good agreement with the literature value of 0.35238 nm[8].

## 4. CONCLUSION

1. Using Hydrazine hydrate and $NiCl_2.6H_2O$ plating bath is very useful for nickel plating of SiC and Al powder mixtures.
2. If we could solve the wetting problem of ceramic this method would be alternative for the preparing of SiC+AlNi such a composite.

Table 3. XRD analysis data for powders using a 110 volt arc furnace

| Observed Intensity | $Sin^2\theta$ Observed | *$Sin^2\theta$ Calculated | Plane hkl | Phases |
|---|---|---|---|---|
| Strong | 0.0602 | 0.0602 | 111 | Si |
| V.Strong | 0.0933 | 0.0936 | 111 | β-SiC |
| Strong | 0.1248 | 0.1248 | 200 | β-SiC |
| Strong | 0.2212 | 0.2212 | 311 | Si |
| V.Strong | 0.2497 | 0.2496 | 220 | β-SiC |
| Medium | 0.3215 | 0.3215 | 400 | Si |
| V.Strong | 0.3432 | 0.3432 | 311 | β-SiC |
| Medium | 0.3739 | 0.3744 | 222 | β-SiC |
| Medium | 0.3817 | 0.3816 | 331 | β-SiC |
| Strong | 0.4817 | 0.4817 | 422 | Si |

* Calculated: β-SiC, a = 4.364 °Si, a = 5.440 °A

Table 4. XRD data of standard silicon carbide powder

| Observed Intensity | Observed $Sin^2\theta$ | Calculated* $Sin^2\theta$ | Plane hkl | Phase |
|---|---|---|---|---|
| Strong | 0.0856 | 0.0856 | - | α-iv SiC |
| V.Strong | 0.0936 | 0.0936 | (111) | β-SiC |
| Strong | 0.1062 | 0.1062 | - | α-SiC |
| Medium | 0.1247 | 0.1248 | (200) | β-SiC |
| V.Strong | 0.2496 | 0.2496 | (220) | β-SiC |
| Strong | 0.2933 | 0.2933 | - | α-SiC |
| V.Strong | 0.3432 | 0.3432 | (311) | β-SiC |
| Strong | 0.4982 | 0.4982 | (400) | β-SiC |

* Calculated β-SiC, a = 0.4358 nm

Table 5. XRD data of the Ni plated mixture of SiC and Al powders

| Observed Intensity | Observed $Sin^2\theta$ | *Calculated $Sin^2\theta$ | Plane hkl | Phase |
|---|---|---|---|---|
| Weak | 0.0933 | 0.0936 | (111) | β-SiC |
| Strong | 0.1084 | 0.1085 | (111) | Al |
| V.B.V.Strong** | 0.1432 | 0.1431 | (111) | Ni |
| V.B.V.Strong | 0.1909 | 0.1908 | (200) | Ni |
| Weak | 0.2497 | 0.2496 | (220) | β-SiC |
| Medium | 0.2892 | 0.2894 | (220) | Al |
| Weak | 0.3431 | 0.3432 | (230) | β-SiC |
| V.B.V.Strong | 0.3817 | 0.3816 | (220) | Ni |

**Very broad very strong *β-SiC$a_o$=0.4361nm Ni$a_o$= 0.3523nm Al$a_o$= 0.4051nm

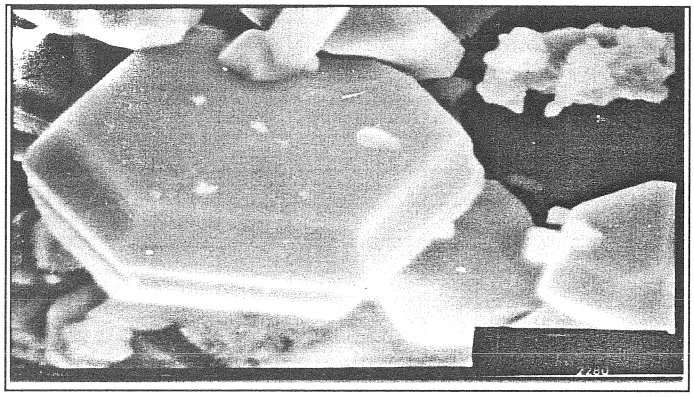

Figure 1 SEM picture of SiC powder (X10000)

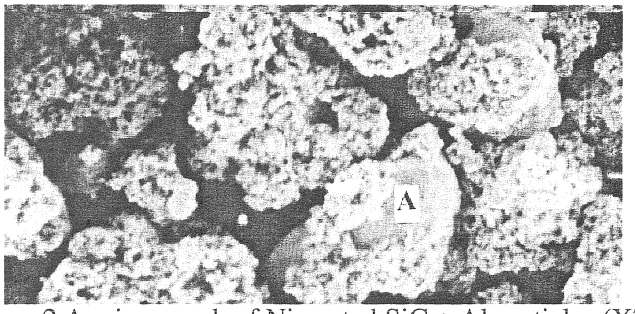


Figure 2 A micrograph of Ni coated SiC + Al particles (X700)

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