Effect of Heat Treatment on Coated Ceramics for Composite Formation by Laser Processing

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ABSTRACT

Metal matrix composites are popular now-a-days because of their superior mechanical properties such as high mechanical strength; wear resistance, excellent thermal conductivity and ability to retain strength at high temperature. However, there still exist challenges in producing high quality metal matrix composites by different laser additive manufacturing processes and one such important process is direct metal laser sintering. To obtain the highly densified products by direct metal laser sintering the critical factor is attaining excellent bonding between the reinforcement and matrix. Further to achieve more improvement in uniform dispersion and mechanical properties, there is a need to concentrate on reducing the interfacial reaction between the matrix and reinforcement. The above mentioned critical issues associated with metal matrix composites are addressed in the recent times by improving the wettability of the reinforcement. Metallic coating of the ceramic reinforcements are accepted universally to address the above issues. Electroless plating technique can be used to coat metallic coatings on ceramic reinforcements. Several parameters influence the process of coating, on the ceramic particles to get uniform and adherent deposition of metallic particles including pre-treatment of ceramic powder. With reference to the above facts, this paper focuses on the methodology for achieving a thin uniform Nickel-Phosphide coating on Siliconcarbide (SiC) particles, which is the most commonly used ceramic reinforcement material to develop metal matrix composites by direct metal laser sintering process and also about the importance of heat treatment of ceramic powders after coating. Scanning Electron Micrograph (SEM), Energy Dispersive Spectroscopy (EDS), and X-ray Diffraction (XRD) studies have been carried out on the coated ceramic particles. These studies clearly reveal the uniform coating of Nickel-Phosphide on ceramic particles and also reveal that the coating on the surface of the SiC is in amorphous before heat treatment and reaches crystalline state after heat treatment indicating the presence of stable Ni and Ni_3P phases.

Key words: *Metal matrix composites; Additive manufacturing process; Direct metal laser sintering; Electroless plating; SiC; Heat treatment; Nickel-Phosphide coating; SEM; EDAX; XRD studies.*

1 INTRODUCTION

Properties of the metal matrix composites strongly depend on the interfacial phenomena between the metal matrix and the ceramic reinforcement [12].The direct metal laser sintering process used here for fabrication of metal matrix composites is based on liquid phase sintering (LPS) mechanism involving a partial melting of the powder [10,15]. Here the multi-component powder mixture is generally composed of the high melting point metallic/ceramic component acting as reinforcement and low melting point metallic component acting as a binder [8, 9]. Here the densification of solid/liquid system and the wetting characteristics of the metal/ceramic inter-phase decide the final success of the laser sintered part. The wettability of reinforcement by liquid metal is the key factor to achieve high interface bonding strength [3]. Hence the coating on metal/ceramic plays an important role in both wetting and densification. Silicon carbide used here possesses good thermal conductivity, electrical conductivity, chemical stability, and high mechanical strength, and has been used to prepare metal matrix composites to enhance their mechanical properties and to resist high-temperature [14]. It has been reported that the coating on these ceramic particles not only improves the green density and sintering activity, but also enhances phase uniformity and mechanical properties of the sintered body [16]. Even though, different coating methods such as co-precipitation, sol-gel and electroless plating are used for coating powders, the electroless plating is considered as one of the most effective coating techniques to coat the surface of different ceramic powders [6]. This method of coating is also used to coat material surfaces such as plastic, alloy and powders [13]. Thin metallic films on plastics, metals and ceramics is obtained by dipping the substrate in electrolyte solution by electroless plating method. In this method conducting and non-conducting materials are deposited with metals and alloys by controlled autocatalytic electrochemical reactions. This autocatalytic metal plating represents an easy and effective method, since variety of metals can be used and a thick coating can be formed [11]. Brenner and Ridell invented electroless nickel coating in 1940s [1]. It is called as electroless plating due to the absence of external electrodes. Here substrate acts as cathode and metal is supplied by metallic salt and electrons are supplied by the reducing agent. This plating process offers more number of advantages compared to the conventional electroplating. The important features of the electroless plating are its excellent coating power, ability to coat uniformly reaching down deep bores, corners and edges in a short period of time for whatever the complexities involved in geometry of the surface to be plated. High corrosion, High hardness especially when deposited as a nickel-phosphorous and heat treated and wear resistance of nickel coatings have already been reported [5] and as a result of which it finds wide range of applications in aerospace, automotive, electronics and computer related industries.

Recent literature reports about metallic coating use to overcome the challenges posed during the development of metal matrix composites. Metal matrix composites normally have two distinct phases, the matrix and reinforcement with different physical and mechanical properties. The thermal mismatch exists because of the large difference in the coefficient of thermal expansion [3] of the matrix and reinforcement, which are commonly ceramic in nature, leading to thermal stresses. De-cohesion effect occurs because of the residual stresses. Applications of metal matrix composites are limited due to decrease in strength and wear resistance of developed metal matrix composites because of de-bonding of the ceramic reinforcements in metal matrix composites. Poor wettability of the ceramic reinforcement is the main reason for the de-bonding phenomenon [15]. There are reports about nickel coated ceramic powders perform better with respect to wettability when compared with uncoated ceramic reinforcements.

There are reports about a uniform and continuous nickel-phosphide coating on both aluminiumoxide and SiC powders [11], where experiments have been performed on infiltration of nickel coated SiC powders with molten aluminum and reported improved adhesion and wettability of reinforcements in metal matrix composites. Some researchers concentrated on coating of nickel on nano SiC powders and have reported that pre-treatment of powders have greater impact on the rate of nickel plating [4]. Experiments are performed with respect to coating on nano particles of SiC and reports about greater effect of coating on the dielectric behavior of the coated SiC [17]. There are reports about the importance of the pH range and holding time for better coating of Nickel-Phosphide on ceramic powders by the recent research on coating. With the vast literature review very less information is available about the heat treatment effects on the ceramic powders. In light of the above, the present investigation is aimed at characterization of coated and uncoated ceramic powders and the importance of heat treatment on the coated ceramic powders.

2 EXPERIMENTAL DETAILS

Silicon carbide was chosen as reinforcement for preparing composites. Silicon carbide (SiC) is a ceramic compound of silicon and carbon. The choice of SiC as reinforcement in matrix is because of its high hardness and wear resistance, low density, high mechanical strength, and ability to retain strength at high temperature. Carborundum is the other chemical name for silicon carbide (SiC). The hard bonded ceramic SiC particles are used in car brakes, and car clutches. Hence, SiC particles are widely used as reinforcement in composite matrix phase of metal, plastic, and ceramics [2]. SiC light black in color, procured from Grindwell Norton, Bangalore was chosen as reinforcement to prepare the composites. The density of silicon carbide used in the present work was 3.2 g / cm^3.

The SiC powders were characterized for size, morphology and composition. Particle size analysis was carried out using laser based particle size analyzer of make: HRLD, USA and model: 8000A. A known quantity of SiC powders was mixed with an ultra clean hydraulic oil of NAS class ‘0’ cleanliness. The powder-oil mixture was thoroughly mixed with mechanical agitator and then passed through particle size counter. The average particle size of the SiC was found. The morphology and the elemental analysis were found using scanning electron microscope (SEM) of make: Carl Zeiss and model: Neon 40 crossbeam. The X-ray diffraction (XRD) study was done using X-ray diffractometer of make: Bruker and model: XRDD8ADVANCE.

Nickel-phosphide coating was done on SiC particles by electroless coating process. The electroless nickel bath composition was prepared and the coating is done as per the literature reported [7].The bath temperature was maintained at 85^0 C and the coating duration of 30 minutes was adopted. The operating pH range is between 4 to 8 and the preferred pH range for maximum deposition of Ni-P coating is between 4.3 and 4.9. Magnetic stirrer with heater of make: Remi and model: 2MLH, electronic balance with least count of 1 mg of make: Sartorius and model: BSA223S, and pH meter of make: Equip-tronics and model: 6Q-610 were used for coating purpose with Nickel Chloride, hexahydrate ($NiCl_2.6H_2O$), 30 g/L, Sodium succinate, hexahydrate ($Na_2C_4H_4O_4.6H_2O$) 10 g/L, Sodium Hypophosphite, monohydrate (NaH_2PO_2,H_2O) 20 g/L, Glycine (H_2NCH_2COOH)10 g/L, and Lead nitrate ($Pb(NO_3)_2$) 2 mg/L as chemicals for preparing the electroless nickel bath solution.

3. RESULTS AND DISCUSSION

3.1 Particle Size, Morphology and Scanning Electron Microgrphs (SEM) and XRD Studies on Silicon carbide (SiC) Powders

The SEM of uncoated silicon carbide is shown in Fig.1. It clearly shows that the particles are bright and smooth with irregular shape. The average particle size of silicon carbide is around 28μm.

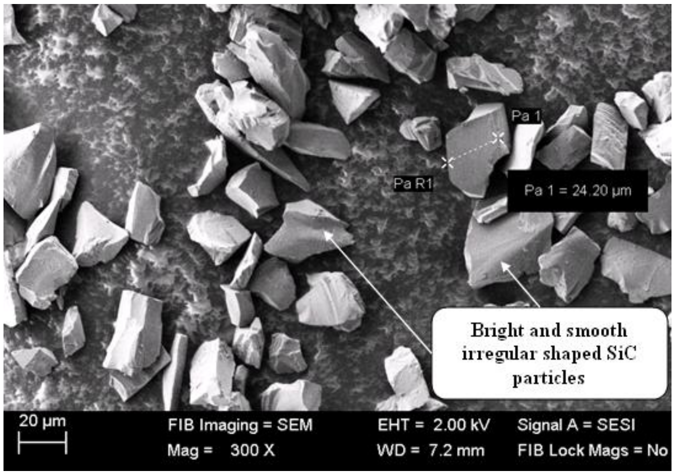

Fig. 1 SEM Showing the Shape and Size of SiC Powder

The XRD patterns of the procured SiC particles are shown in Fig.2. The X-ray diffraction patterns of the powders clearly shows only the diffraction peaks of SiC.

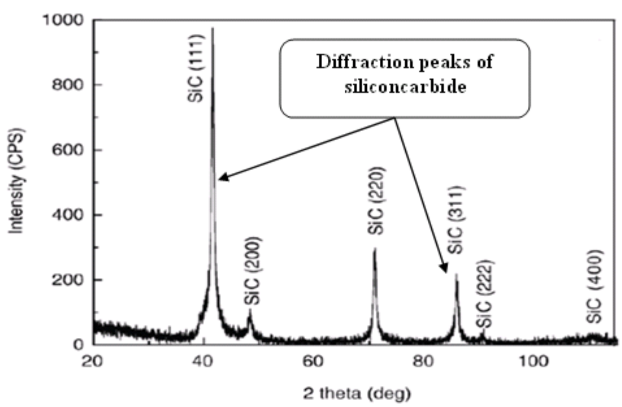

Fig. 2 X-ray Diffraction (XRD) pattern of SiC Powder

3.2 Nickel-Phosphide Coating on Silicon carbide (SiC)

The surface of the SiC powders are at first coated with palladium. The SEM of the palladium coated SiC is shown in Fig.3. The rough surfaces are observed indicating the presence of palladium coating on SiC particles.

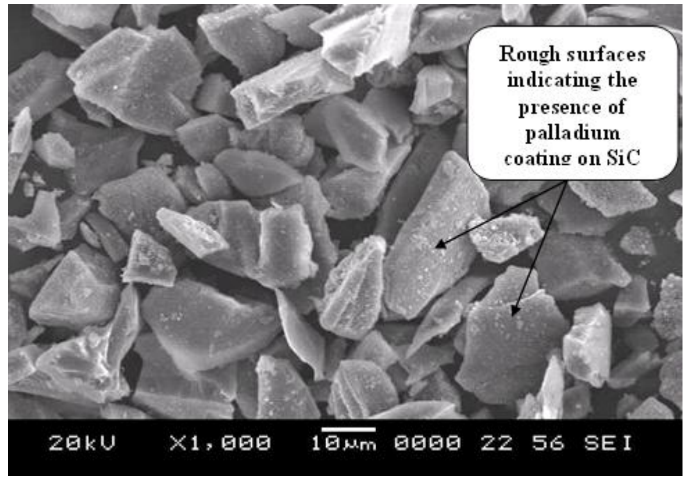

Fig.3 SEM of Palladium Coated SiC Powder

The palladium coated SiC powder is now coated with Nickel-Phosphide. The SEM of the Nickel-Phosphide coated SiC powder is shown in Fig. 4.

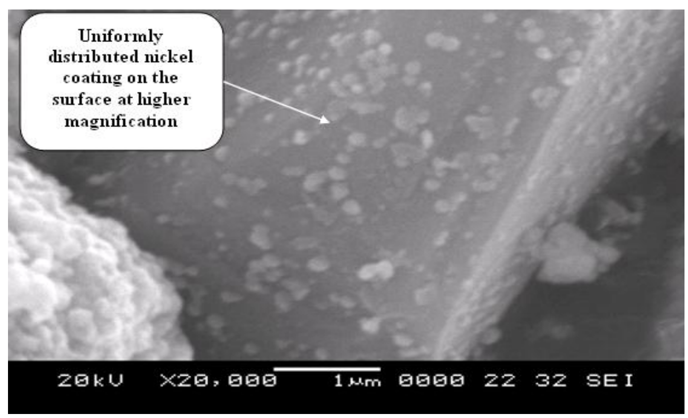

Fig.4 SEM of Nickel-Phosphide Coated SiC Powder

The coated powder shows a uniformly distributed nickel coating on the surface, and at high magnification (X 20,000), a continuous nickel coating is observed over the whole surface; the coating is present on all the regions of the particles regardless of size and shape. The EDS analysis of nickel coated SiC shown in Fig.5 indicates the presence of both the nickel and phosphorus.The presence of nickel on the surfaces of the ceramics helps in considerable improvement in wettability of the cermic reinforcement with the molten matrix.This factor results in better interfacial bond strength and also minimizes the formation of interfacial complex products such as carbides and silicides.

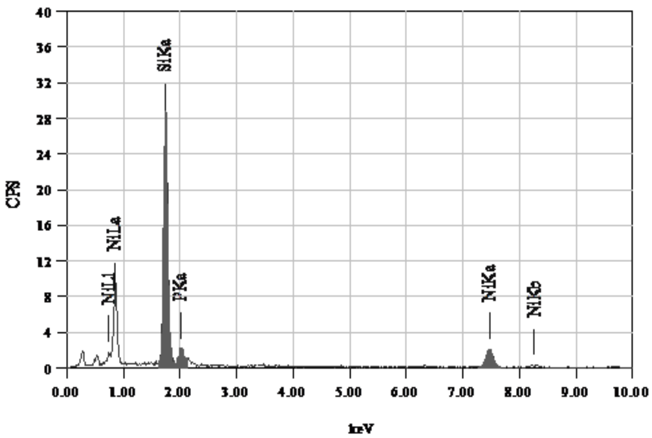

Fig.5 EDS of Nickel-Phosphide Coated SiC Powder

3.3 Effect of heat treatment on coated SiC powders

Literature suggests that coating results in increase in the specific surface area. This is because of the fine nickel particles of about 1 μm found on the outer layer of the deposit and the presence of micro voids between the fine trapped particles. The literature also suggests that electroless nickel deposits, which have not been heat-treated, can have structures ranging from extremely small crystallites to those that are fully amorphous. So the XRD studies have been made to check this suggestion given in the literature. The XRD studies in the Fig.6 indicate a pronounced broad band in the region corresponding to the Ni (111), signifying that this particular deposits contain the metal in amorphous state. Now this coated powder upon heat treatment at 400^0C, transformed into a mixture of crystalline Ni and Ni_3P. XRD studies shown in Fig.7 on the heat treated powder indicates sharp peaks corresponding to a fully crystallized structure. Therefore it is clear that amorphous character of coated nickel powder becomes crystalline after heat treatment, showing deposits containing the stable Ni and Ni_3P phases.

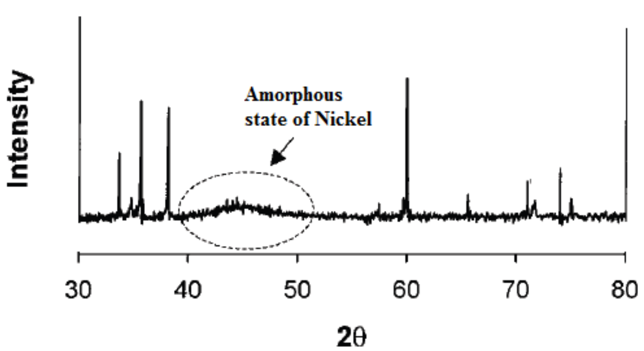

Fig.6 XRD Pattern of Ni-P Coated SiC before Heat-treatment

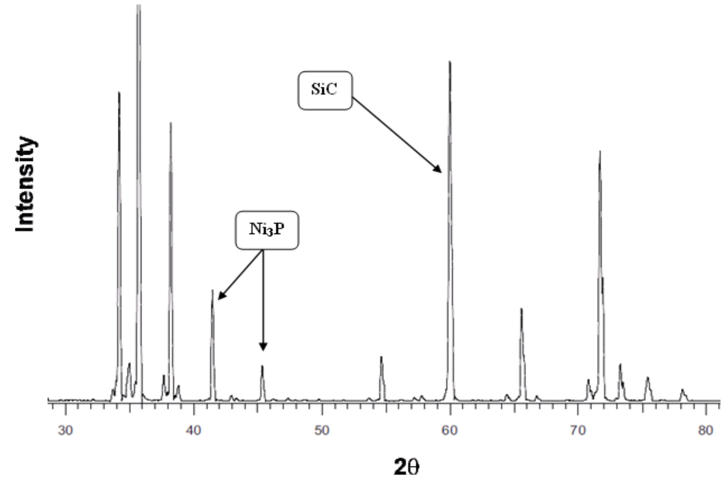

Fig.7 XRD Pattern of Coated SiC after Heat-treatment

4. CONCLUSIONS

The following conclusions can be drawn from this work…

- Particle size analysis and scanning electron micrograph studies on the procured SiC particles reveal that the average size of the SiC particles is 28 μm with irregular shape.
- SEM studies on palladium coated SiC powder indicate the presence of palladium on the surface of SiC particles.
- SEM, EDS and XRD studies on Nickel-Phosphide coated SiC shows a uniform coating of Nickel-Phosphide and the elemental analysis by EDS indicate the presence of Nickel (Ni) and Phosphorus (P) on the surface of SiC.
- XRD studies also reveals that the coating on the surface of the SiC is in amorphous before heat treatment and reaches crystalline state after heat treatment indicating the presence of stable Ni and Ni_3P phases.
- Presence of stable Ni and Ni_3P phases helps in better wetting of reinforcement with matrix, bond strength and flowability during blending of reinforcement and matrix powders during composites formation.

Thus, with the above conclusions we can consider the metallic coated SiC particles as a potential reinforcement for high temperature materials in fabrication of metal matrix composites by direct metal laser sintering process.

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