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# High-resolution TEM characterization of SiC nanowires as reinforcements in a nanocrystalline Mg-matrix

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ABSTRACT

SiC nanowires have been used as potential reinforcements in a nanocrystalline magnesium-based matrix composite processed by cryomilling and spark plasma sintering. The morphology and crystallinity of SiC nanowires were studied by scanning and transmission electron microscopies. Our results indicate highly faceted sidewalls and bamboo-like SiC nanowires that contain a high density of twins and stacking faults. In addition to the common cubic 3C crystal structure, we also found the hexagonal 2H polytype. We suggest that the particular morphology of the SiC nanowires enhances their interlocking with the Mg matrix. A detailed analysis of the interface between a SiC nanowire and the nanocrystalline magnesium alloy was performed using high-resolution transmission electron microscopy and energy dispersive spectroscopy techniques. We demonstrate the absence of interfacial reaction at the SiC nanowires/Mg-matrix interfaces, which is critical for the development of high-performance composites.



## 1. Introduction

SiC nanowires have received a lot of attention due to their outstanding properties [1–3] that make them excellent candidates as reinforcements in a variety of composites including polymers [4,5] and metal-matrix composites [6,7], for advanced technological applications.

The interface between the matrix and the reinforcements plays a key role in the development of high-performance composites. For instance, the characteristics of the interface may influence the crack resistance and load transfer during deformation, which affect directly on the mechanical properties of the composite. Therefore, the interfaces should be properly tailored in order to meet the property-performance requirements. To this end, the presence of voids at the interface and/or interfacial reactions that may result in an insufficient interfacial bonding should be minimized. This is the main requirement for any kind of reinforced composites. In particular, for SiC reinforcements in Mg-matrix composites this is a challenging task due to the high reactivity of Mg, which may lead to the formation of several reaction products, such as oxides [8,9], $Mg_2Si$ [10,11] and Al–C–O ternary compounds [11]. It has been previously reported that the presence of second phases may cause embrittlement at the interfaces [12]. In fact, a weak interfacial bonding will result in a detrimental effect on the mechanical behavior of the composite.

Here, we report transmission electron microscopy (TEM) studies of SiC nanowires used as potential reinforcements of a Mg-based composite processed by cryomilling and spark plasma sintering (SPS). We confirm that the high density of structural defects causes the zigzag appearance of the contoured surface of SiC nanowires. We believe that this particular morphology will better promote their interlocking with the Mg matrix, making them more suitable to be used as reinforcements in composite materials. A well-bonded inter-

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face between the SiC nanowires and the matrix is observed with no presence of second phases.

## 2. Experimental Procedure

Commercially available gas-atomized Mg and Mg–Al alloy ($Mg_{50}Al_{50}$) powders were blended to formulate a final composition of $Mg_{70}Al_{30}$ (wt.%). Twinned SiC nanowires provided by Prof. Tu at University of California Los Angeles were prepared via the preparation of a carbonaceous silica xerogel with the subsequent carbothermal reduction of the xerogel via the vapor–liquid–solid (VLS) process [13]. The details of the experimental procedure of the SiC nanowires are presented in Ref. [14]. In order to prepare the Mg-based composite, 500 g of the blended Mg-alloy powders along with 0.14 wt.% of SiC nanowires were cryogenically-milled for 8 h under liquid nitrogen atmosphere in a Union Process, Szegvari mill (Akron, Ohio) extensively modified at California Nanotechnologies for light alloy processing. Hardened stainless steel balls (6 mm diameter) were used as milling media at a particular ball-to-charge ratio for a given material. A 30:1 ball to powder mass ratio is common for milling metallic powders. Powder consolidation was conducted using a spark-plasma-sintering (SPS) system (Syntex Inc., Dr. Sinter Lab TM, model SPS-515S, Kanagawa, Japan). The cryomilled sample was then placed in a graphite die lined with graphite foil to prepare the bulk material. The sintering process was performed in vacuum and under a maximum uniaxial pressure of 70 MPa. A K-type thermocouple inserted into the outer wall of the die was used to control the ramp rate (75 °C/min) and hold temperature (for 6 min at 400 °C) as a pulsed electric current is applied through graphite punches. The SPS-consolidated specimen resulted in a disk with 25.4 mm and 6 mm of diameter and thickness, respectively.

The morphology of the SiC nanowires before and after being embedded into the Mg matrix was investigated using scanning and transmission electron microscopies. A FEI Nova 230 Variable Pressure SEM (VP-SEM) at 10 kV accelerating voltage and a FEI Titan 300 kV scanning transmission electron microscope (STEM) equipped with an Oxford Instruments EDS system were used for this purpose. A FEI Nova 600 Nanolab DualBeam focused ion beam (FIB)–SEM was used to prepare a thin film TEM sample from the nanocomposite material.

In order to investigate local variations in structure and/or composition at the SiC Nws/Mg-matrix interface, we employed fast Fourier transform (FFT) of lattice resolved TEM images acquired from several regions across the interface using Image J [15].

## 3. Results and Discussion

Fig. 1 shows the representative SEM images of the as-grown twinned SiC nanowires. All the nanowires studied (with a length of tens to hundreds of micrometers [14] and diameters between 200 and 400 nm), exhibit hexagonal cross-sections as shown by the slightly tilted nanowire in Fig. 1a. In addition, they present highly faceted sidewalls that lead to a remarkable zigzag surface morphology (Fig. 1b).

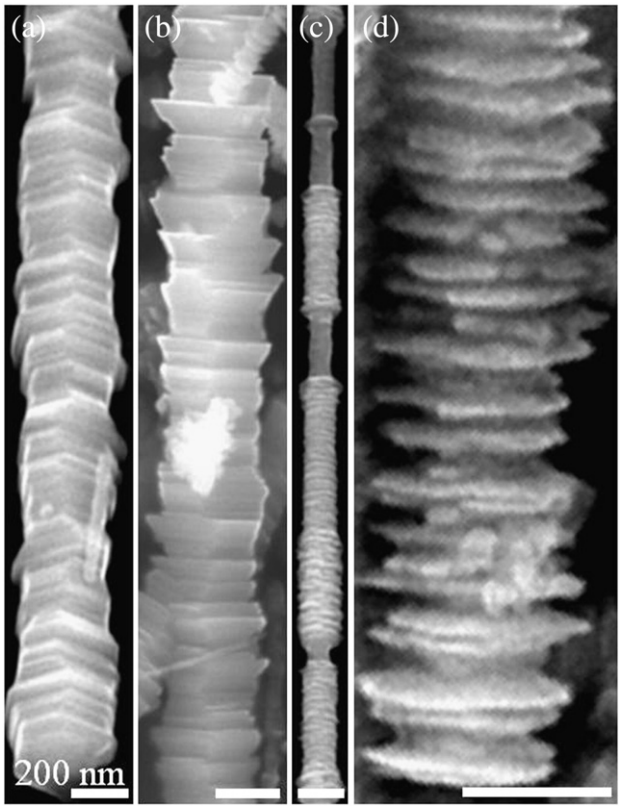


**Fig. 1 – Scanning electron microscopy (SEM) images showing the morphology of the as-received SiC nanowires. Scale-bar is 200 nm.**

Fig. 1c is a SEM image of a typical bamboo-like SiC nanowire showing the characteristic "stem-and-node" structure [16,17]. SEM image at higher magnification (Fig. 1d) displays the particular shape of the "nodes" of a bamboo-like SiC nanowire.

A detailed TEM analysis of the as-received SiC nanowires was performed in order to better understand their morphology and crystallinity. Fig. 2a is a bright-field TEM image of a bamboo-like SiC nanowire showing the characteristic stem-and-node structure [16,17]. The nodes are longer and thicker than the stems as can be clearly seen at higher magnification in Fig. 2b. A stem with a diameter of ~50 nm bounded by two nodes of ~165 nm in diameter presents a smooth surface. We observe a typical contrast variation along the growth direction within the nodes, which is due to a high density of structural defects, such as stacking faults (SFs) and twins as confirmed by the high-resolution TEM (HRTEM) image (Fig. 2c). SFs are highlighted by white arrows and twins by black solid lines. (111) twin boundaries (TB) are also marked by black dashed lines. The selective area electron diffraction (SAED) pattern from the nodes acquired along the [011] zone axis is shown as inset. It is interesting to observe the characteristic streaks along the [111] (the growth direction) as a consequence of the structural defects parallel to this direction. From the diffraction data, we identify the typical cubic 3C-SiC crystal structure [18]. Interestingly, we also observe some defects (highlighted by a white arrow) in the stems that are not parallel to the growth direction (Fig. 2d). Fig. 2e is a HRTEM