Microstructural characterization of Al-MWCNT composites produced by mechanical milling and hot extrusion

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Abstract

Aluminum-based nanocomposites were produced using multiwalled carbon nanotubes (MWCNTs) dispersion by mechanical milling (MM) followed by sintering and hot extrusion. MWCNTs showed stability after MM and thermo-mechanical processes. Excellent adhesion of nanotubes to Al-matrix was observed. Formation of an amorphous interface between nanotubes and aluminum matrix in nanocomposites was observed by TEM. This amorphous layer is probably responsible of the excellent adhesion of MWCNTs to the aluminum matrix.

Keywords: Metal matrix composites, Powder metallurgy, Sintering, SEM, TEM.

Introduction

Major interest in composite materials started in the middle of the last century, mainly to meet the materials demanded by the aeronautic industry where lower weight and higher strength materials are desired. Since then, important progress in the development of composite materials has been achieved. In the field of composite materials, aluminum-based composites are an important area due to their low density and good workability; such properties are attractive for diverse industrial applications. The excellent mechanical properties, concomitant with their chemical stability [1,2], suggest that carbon nanotubes (CNTs) might be suitable as a novel reinforcement material for aluminum-based metal matrix composites. Aluminum matrix composites reinforced by multi-walled carbon nanotubes (MWCNTs) dispersion is an emerging area that is calling the attention of several research groups in the scientific community [3–6]. Several works have been reported in this area, and one of the main problems faced is the wetting of nanotubes by aluminum matrix [7,8].

The aim of this work deals with the microstructural characterization of Al-MWCNTs nanocomposites prepared through mechanical milling, sintering and hot extrusion. Morphological stability and adhesion of CNTs are presented and briefly discussed from a microstructural point of view.

Experimental

Al powder (99.9% purity, −325 mesh in size) and MWCNTs were used to produce Al-based nanocomposites. The MWCNTs used in this work were produced by the spray pyrolysis method [9]. Different nanocomposite compositions were studied, namely through MWCNTs additions of 0.0, 0.25, 0.50, 0.75, 1.0, 1.25, 1.50, 1.75 and 2.0 wt.%. Pure Al was also investigated for comparison purposes. Each mixture (80 g) was mechanically milled during 5 h in a high energy mill (Simoloyer). Milling time was chosen from previous results [10–12]. Argon was used as the inert milling atmosphere. All milling runs were performed with 0.5 ml of methanol as a process control agent (PCA). The obtained products were compacted under ~60 tons during 2 min. Compacted samples were pressure-less sintered during 3 h at 823K under vacuum with a heating rate of 50 K/min. Sintered products were held for 0.5 h at 773K and hot extruded into a rod of 10mm in diameter by using indirect extrusion and an extrusion ratio of 16. The pure Al (reference sample) was not milled; it was only consolidated, sintered and extruded under the same conditions. The characterization of MWCNTs was performed by scanning electron microscopy (SEM) in a JSM-7401F instrument operated at 3–5 kV. Also, microstructural observations of Al-based nanocomposites were performed in a SEM model JSM-5800LV operated at 20 kV, and by transmission electron microscopy (TEM) in a CM200 instrument operated at 200 kV. For TEM observations, a foil was prepared from the specimen at 2.0 wt.% of MWCNTs in order to observe the dispersion of MWCNTs into the Al-matrix. All samples were prepared according to the optimal conditions reported for pure aluminum by Ünlü [13].

Results and discussion

Figs. 1(a) and 1(b) show SEM micrographs of the as-prepared MWCNTs. From these figures, a diameter of ∼40–100nm and a length of ∼10μm can be observed. High homogeneity in diameter along CNTs is also observed.

To facilitate the observation of MWCNTs within the Al-matrix, an Al–2 wt.% MWCNTs nanocomposite was produced. Fig. 2 shows a bright field TEM image of this material. This figure shows that the MWCNTs are dispersed quite homogeneously in the composite (black lines in Fig. 2 indicated by arrows).

Fig. 3 shows a conventional bright field TEM image (low magnifications) of the Al–1.0 wt.% MWCNTs composite. The contrast corresponds to bent contours, grain boundaries, dislocations and MWCNTs dispersed into aluminum matrix.

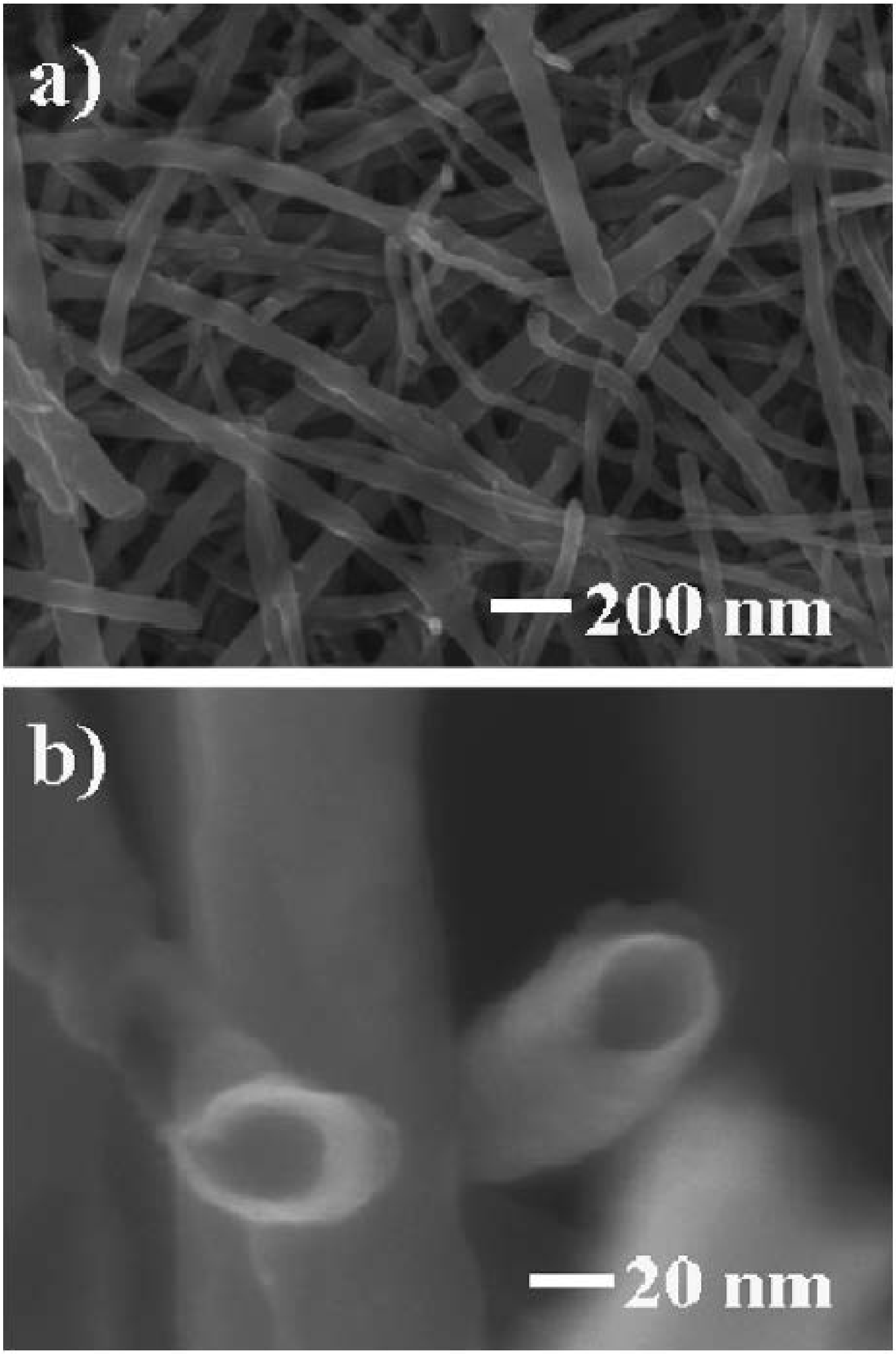

Fig. 1. Secondary electron SEM images from CNTs at (a) 40 and (b) 400 kX.

Fig. 4 shows SEM micrographs from a fractured surface in an aluminum-based nanocomposite after tensile test. Fig. 4(a) shows the presence of ductile fracture in composites. In our previous works, it has been reported homogeneous and random MWCNTs dispersion in the aluminum matrix [10]. Additionally, it has been reported an important increment on the mechanical properties evaluated by tension, compression and hardness tests [10–12]. From these results, it is evident that by MWCNTs dispersion is possible to increase the mechanical properties keeping almost invariable the intrinsic properties, like ductility.

Fig. 4b (higher magnifications) shows a MWCNT emerging from the broken surface after tensile test. Notice that the nanotube kept the morphology after all the long experimental sequence (mechanical milling-sintering-hot extrusion). This figure shows the internal and external diameters in the emerging nanotube. SEM characterization shows that nanotubes are chemically and physically stable. This figure shows the adhesion ofMWCNTsto the aluminum matrix, which indicates the existence of strong interface strength between aluminum matrix and the nanotubes. From this figure it can be assumed that the MM, sintering and thermo-mechanical treatments have a positive effect on the wetting of MWCNTs by Almatrix. Additionally, damage at the ends of the nanotubes is not completely evident, which means that MWCNTs are stronger than the amorphous interface and, under uniaxial loads (tensile test), exists a sliding in the amorphous layer before nanotubes break. This characteristic makes CNTs excellent candidates for reinforcing aluminum alloys.

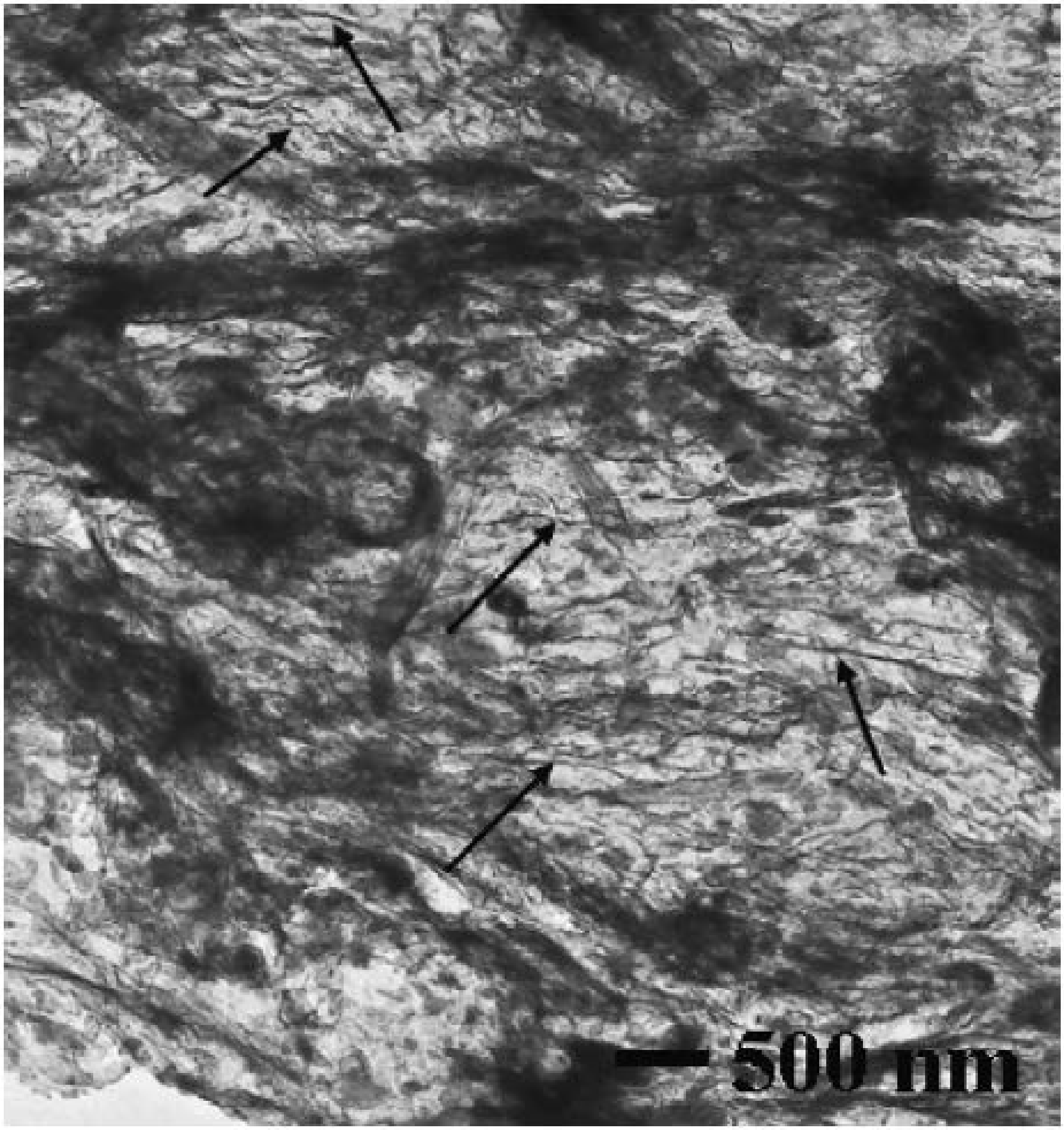

Fig. 2. Bright field TEM micrograph (low magnifications) of Al-MWCNTs composite containing 1.0 wt.% of MWCNTs, annealed for 3 h at 823 K in vacuum. The arrows indicate some MWCNTs.

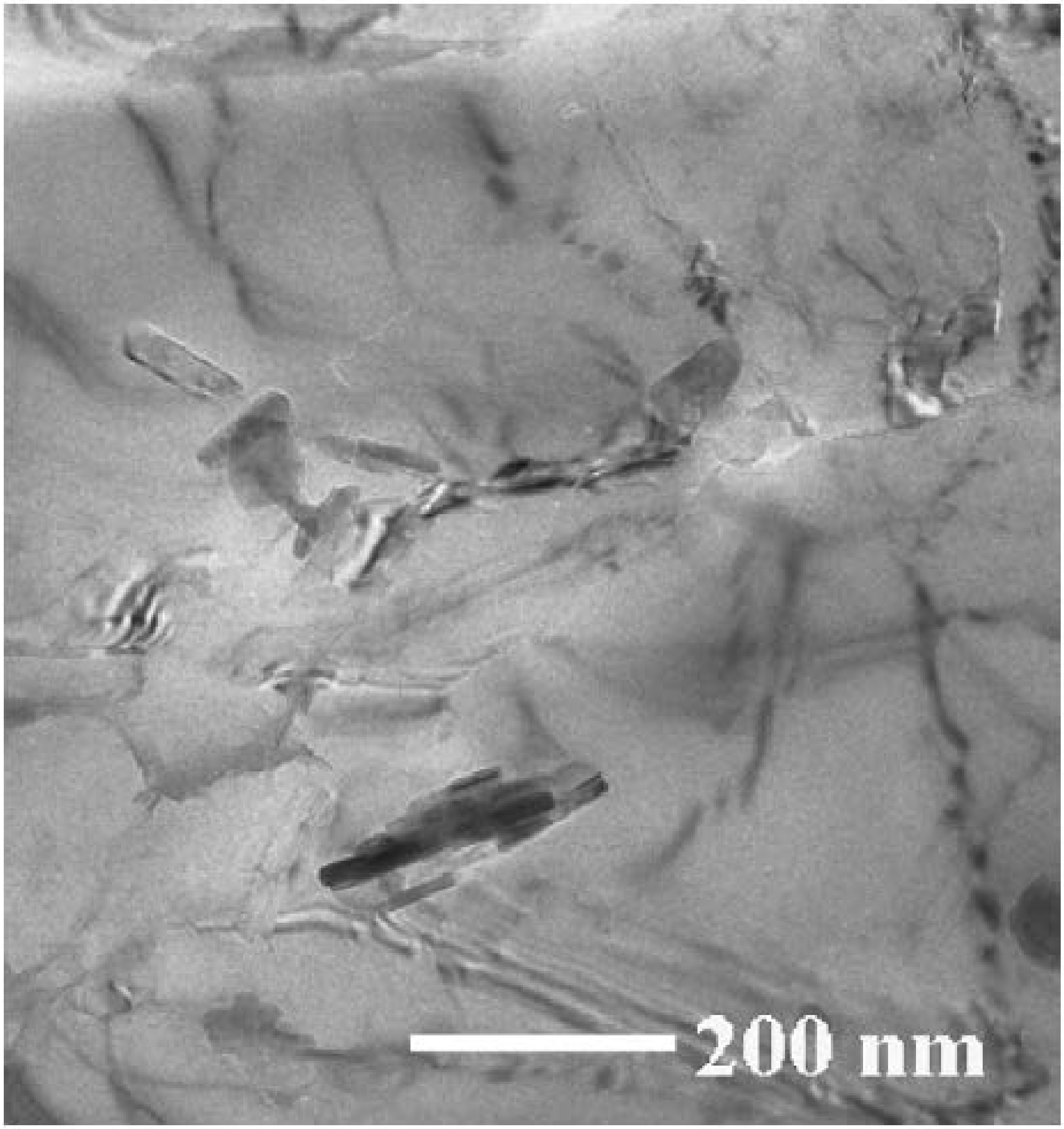

Fig. 3. Bright field TEM image of an Al-MWCNTs composite containing 1.0 wt.% of MWCNTs.

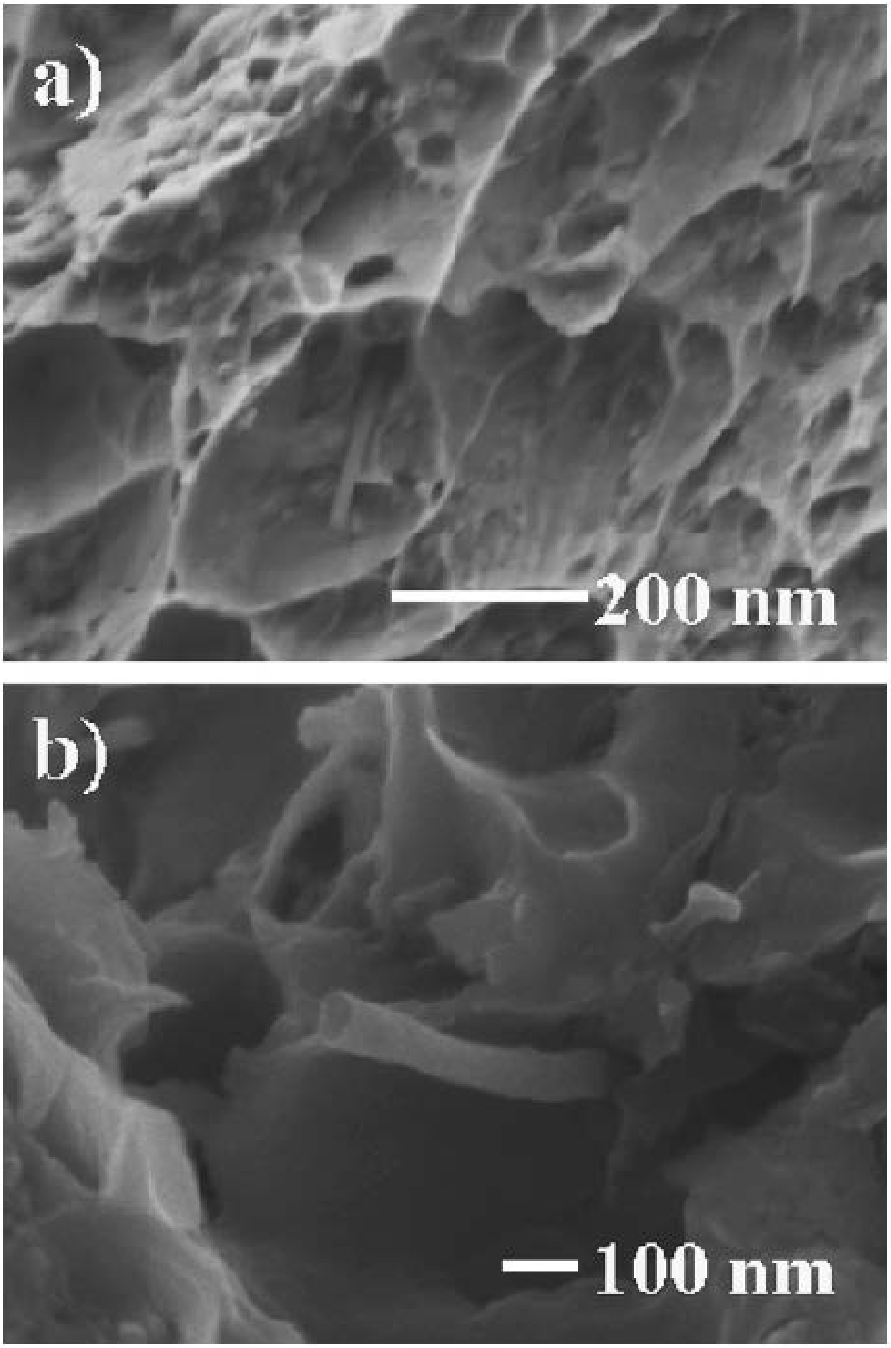

Fig. 4. Secondary electron SEM images on the broken surface showing CNTs outstanding from the broken Al-matrix surface. (a) Ductile fracture and (b) a CNT emerging from the broken surface.

A bright field TEM image of Al–2 wt.% MWCNTs composite is presented in Fig. 5; the inset shows the respective selected area electron diffraction pattern. The figure shows a MWCNTs section where continuous and well-defined lattice fringes of the CNTs walls can be seen. Furthermore, some regions of the observed MWCNTs suggest some amorphization of the outershells (black arrow in Fig. 5). Additionally, showed by a dashed black arrow, the lost of periodicity in the outer walls in MWCNTs is presented, this is probably the beginning of an amorphous layer formation. The formation of this amorphous layer could be responsible for the excellent adhesion of MWCNTs to Almatrix; in addition, this amorphous layer could favor the load transfer from aluminum matrix to MWCNTs during tensile or compression test, resulting in an increment in the resistance of the composite.

These observations confirm experimentally the expected chemical and mechanical stability of the MWCNTs when processed by MM in an Al-matrix, at least for the milling times used in this work (5 h). According to the theory of short fiber reinforced composites, the even distribution of MWCNTs in the matrix, effectively inhibits matrix deformation and produces a strengthening effect, resulting in an increment on mechanical properties, as it has been reported before [10–12].

Conclusions

Multiwalled carbon nanotubes were dispersed homogeneously into aluminum matrix by mechanical milling. Nanotubes showed high stability even after thermo-mechanical treatment for consolidation. Nanotubes present high adhesion to aluminum matrix, meaning that Al can wet CNTs. Formation of an amorphous layer was observed during characterization, this interface could be responsible for the increment in the wetting of nanotubes by Almatrix.

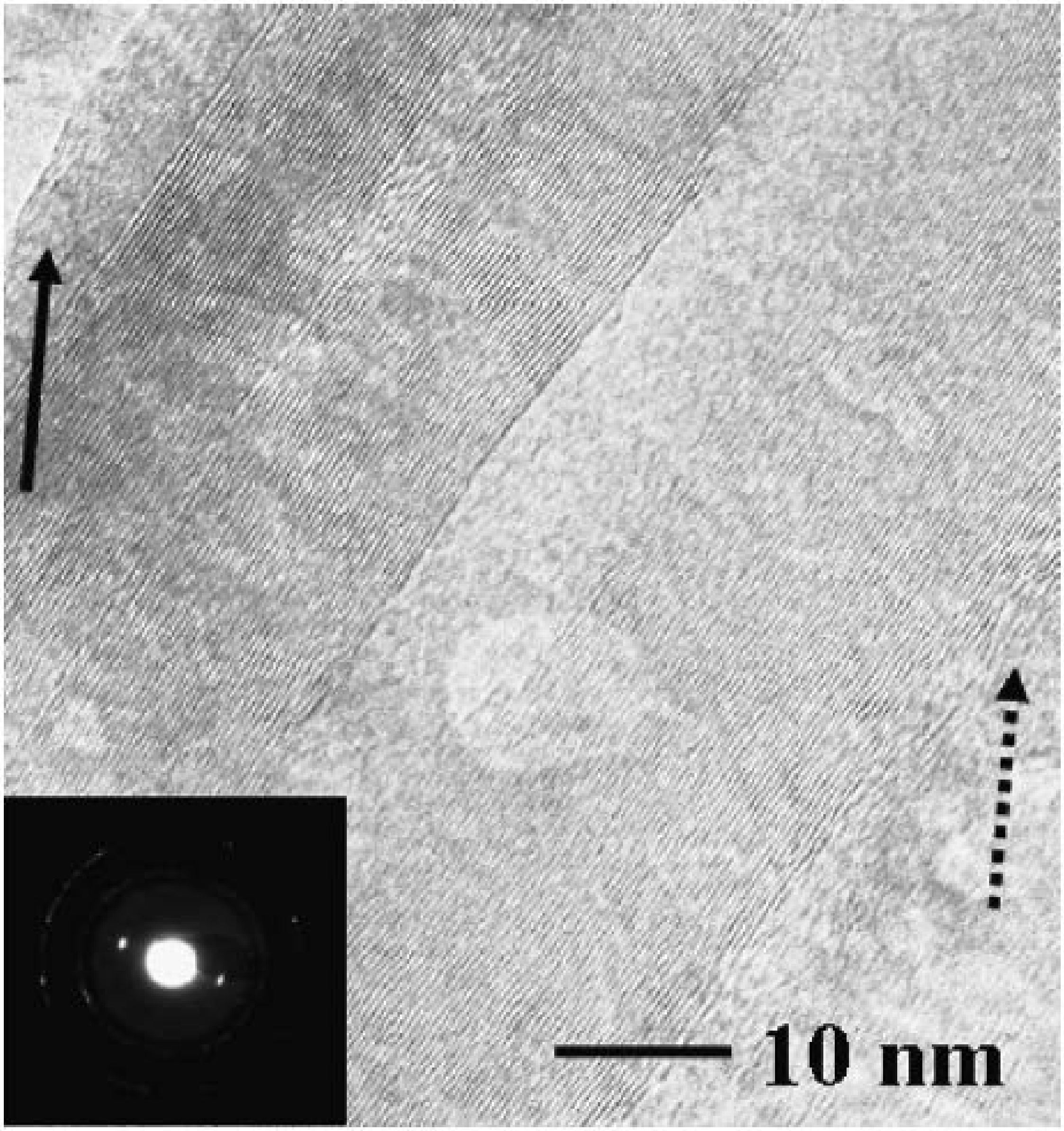

Fig. 5. HRTEM micrograph of the Al-MWCNTs composite showing the interface between MWCNTs and Al-matrix; the inset shows the corresponding diffraction pattern. The arrows show the transition layer between the MWCNTs core and the Al-matrix.

Acknowledgments

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