

TiNi Reinforced Magnesium Composites by Powder Metallurgy

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Abstract

Rod shaped Mg-TiNi composite samples were manufactured by powder metallurgical route in which the samples were heated and deformed simultaneously using rotary hot swaging technique. Firstly, encapsulated argon filled copper tubes which contained compacts of pure magnesium and pre-alloyed TiNi alloy powder mixtures were deformed about 45% in two steps at 450°C. Pre/post annealing heat treatments were applied at 450°C for 20 mins between the stages of coaxial deformation to enhance the sintering degree and to homogenize the heavily deformed composite structures. Next, copper peeled and machined samples were compression tested under quasi-static conditions to investigate the mechanical properties, i.e. yield and peak strength, and ductility. Transmission and Scanning Electron Microscopy studies were carried out to examine the Mg-TiNi interface and fracture surfaces of the compression tested composites, respectively.

Introduction

Magnesium and magnesium alloys are preferred especially in weight critical applications such as automotive and aerospace industry where high specific strength is needed. An increase in the usage of magnesium and its alloys is anticipated since energy is saved as a result of reduced fuel consumption due to relatively low weight of magnesium and magnesium alloy based parts [1, 2]. Accordingly, aluminum and aluminum alloy parts used in various components of automobile body and its engine have been replaced by magnesium and magnesium alloys, i.e. AZ91, AM50/60. In addition, some of magnesium alloys such as WE43 are being considered as attractive lightweight materials to manufacture ultra lightweight armored ground vehicles.

Some of the critical material properties of parts designed for many structural applications in automotive and aerospace industry are specified as density, mechanical properties, i.e. strength and ductility, corrosion and wear resistance. For high temperature applications, on the other hand, the material must carry various types of loads without excessive deformation, which necessitates high creep resistance. For further improvement of mechanical properties of magnesium and its alloys, deformation grain refinement and alloying are used. However, the former is difficult because Mg has poor formability at room temperature due to its HCP crystal structure which is deformed only by basal slip of $(0001)\langle 11\text{-}20 \rangle$ [3]. With respect to alloying recently high strength and ultra high strength magnesium alloys are manufactured by adding rare earth elements such as Y and Re [4, 5]. However, aging heat treatments conducted for increasing the strength of alloyed magnesium [6] may sometimes degrade both creep and corrosion resistance of the alloy. For example $\beta\text{-Mg}_{17}\text{Al}_{12}$ phases, formed during aging of Mg-Al-Zn alloys are reported to reduce the creep resistance as they precipitate along the grain boundaries [7].

The magnesium based parts with higher yield strength and wear resistance compared to their counterparts produced by alloying can be obtained only by the production of magnesium composites. Magnesium composites are manufactured by solid state, i.e. powder metallurgy, or liquid state processing techniques by introducing ceramic or metallic reinforcements in fiber, whisker or particulate form. In liquid processing technique, some problems such as inhomogeneous distribution of reinforcements, formation of pores and inclusions, limited wettability of reinforcement by liquid matrix phase, formation of secondary brittle phases in the matrix/reinforcement interface may arise and degrade the mechanical properties of composites. Because of that, the use of powder metallurgy in composite production has become essential mainly due to relatively low temperatures used, feasibility of adjusting the composite's average grain size by controlling the prior powder particle size, homogenous distribution of reinforcement particles and possibility of utilizing relatively higher amounts of reinforcement, i.e. 50 vol.%.

Although low density ceramic reinforcements such as SiC, TiC, Al_2O_3 , B_4C , Y_2O_3 and TiB_2 provide desired high yield strength, the ductility of magnesium composites decrease due to formation of brittle phases in the interface region or due to pores caused by poor wettability of reinforcements in liquid processing techniques. The observed decrease in ductility of such composites is attributed to the elastic moduli and coefficient of thermal expansion differences between matrix and ceramic reinforcements [8]. In addition, differences in the crystal structures of magnesium and ceramic reinforcement particles prevents load transfer to reinforcement particles and ceramic reinforcement fractures prior to matrix phase. Because of this, the metallic reinforcements in particulate or fiber form have been used to eliminate such disadvantageous effects of ceramic reinforcements. Metallic micrometer sized reinforcements such as copper and nickel [9, 10] have been utilized in manufacturing of magnesium composites. Moreover, some studies were also carried out to investigate the effect of carbon nano-tube addition on mechanical properties of magnesium composites [11]. However, either the required ductility level cannot be achieved or the specific weights of the composite parts increase due to high density of metallic reinforcements. A recent study carried out by Hassan and Gupta [1] has shown that notable ductility levels is achieved in addition to increased yield and tensile strength by using titanium as a reinforcement particle.

Magnesium matrix composites are produced using either conventional compaction and sintering technique or extrusion process [12, 13], which make use of powders or pre-sintered compacts of composites. Conventional techniques are not preferred since they are not effective in obtaining fully dense samples. As these techniques cannot break the oxide layers effectively available diffusion paths for sintering are limited. Because of that, simultaneous application of stress and temperature is an effective way in magnesium composite production by powder metallurgy. Rotary swaging with the characteristics of multidirectional forging and high-frequency

stroking, which results in rod-shaped components, seems to be an effective way of magnesium composite production [14].

This study investigates the powder metallurgy production of TiNi alloy powders added magnesium composites rods via rotary hot swaging. The influence of processing conditions on microstructure has been presented by considering particle boundaries and interfacial regions between matrix and TiNi particles. In addition, the effect of microstructure and TiNi content on mechanical properties of Mg-TiNi composites have also been discussed using compression stress-strain curves and the analysis at the fractured surfaces of samples.

Experimental

Materials

During the composite production, elemental magnesium and pre-alloyed TiNi powders were utilized. Irregular shaped magnesium powders (99.8 %purity) with an average powder particle size of 40 μm were supplied from Alfa-Aesar. Spherical pre-alloyed Ni-rich Ti-50.6 at. %Ni powders (99.9% purity) had an average particle size of 25 μm and they were supplied from Nanoval GmbH & Co. KG, respectively (Figure 1).

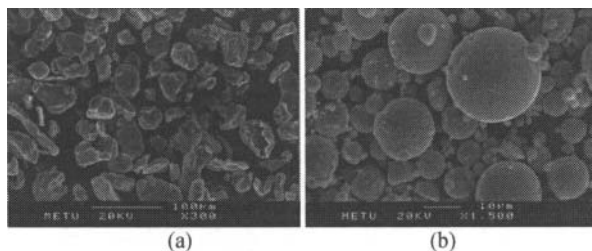


Figure 1. SEM micrographs of utilized powders, (a) angular magnesium, (b) pre-alloyed spherical Ti-50.6 at. %Ni.

Sample Preparation

Initially, Mg-TiNi powder mixtures containing 5, 10 and 15 vol. % TiNi have been prepared by mixing the powders for ½ hour. Then, 10 grams of powder mixtures were pre-shaped in a double ended steel die at around 600 MPa using a hydraulic press to break the possible oxide layers present on magnesium powders. Prior to manufacturing by rotary hot swaging, magnesium and Mg-TiNi green compacts were filled in copper tubes which had one end closed by oxy-acetylene welding and had internal diameter of 12 mm and 1 mm wall thickness. In the next step of production, the green compacts filled tubes are vacuum and argon treated for five cycles to remove the air and/or moisture that might present in copper tubes. This step was finalized by enclosing the tubes when they contained a mixture of vacuum and argon atmosphere. After that, encapsulated copper tubes were heated up to 450°C temperature at which they were kept for 20 minutes prior to feeding them to swaging machine for coaxial deformation. In the deformation stage, about 45% deformation was applied to samples in two steps at a feeding temperature of 450°C. Annealing heat treatments were also conducted at 450°C for 20 minutes between the stages and at the end of the coaxial deformation to increase the sintering degree and to homogenize the heavily deformed structures. Eventually, magnesium and its composite rod samples with 6.5 mm diameter were obtained by peeling and machining copper tubes containing pure magnesium

and Mg-TiNi composite samples after they cooled down to room temperature in furnace.

Characterization

Particle size characterization of as-received magnesium and TiNi pre-alloyed powders were done with a Malvern Mastersizer 2000 using wet method. The density and total porosities of manufactured rod samples were determined on the basis of Archimedes' principle with a Sartorius precision balance equipped with a density determination kit by dipping the samples in a xylol solution ($\text{CH}_3\text{C}_6\text{H}_4\text{CH}_3$). X-ray diffractograms of as-received powders and manufactured rod samples were taken using a Rigaku D/Max 2200/PC model X-Ray Diffractometer by continuous scanning at 40 kW between 30° to 90° 2 θ angles. Microstructures of samples were examined by optical microscope and scanning electron microscope (SEM) Jeol JSM 6400 equipped with Noran System 6 X-ray microanalysis system, on the other hand, was used for both micro structural and fracture surface analysis. Commercial software (Clemex Vision, Professional Edition, version 3.5.020) was used for quantitative analysis to determine the average grain sizes of pure magnesium and magnesium composite rod samples. Manufactured samples were examined both in polished and etched conditions using 5% Nital solution as an etchant.

Further microscopic studies were carried with a Jeol JEM 2100F Transmission Electron Microscope to investigate especially magnesium matrix-TiNi particle interfaces present in Mg-TiNi composite samples. Thin foils of specimens were prepared by ion milling after their thicknesses were reduced using a dimpler. Both dark field (DF) and bright field (BF) techniques were used to investigate the microstructure. In addition, EDS line analysis was carried out to investigate compositional change across the magnesium matrix, TiNi and magnesium-TiNi interface.

Mechanical properties of manufactured samples were determined under compression loads using machined samples having height to diameter ratio of 1.5. The compression tests were conducted under quasi-static conditions with a Shimadzu ACS-J of 10 kN capacity universal tension-compression test machine at a cross-head speed of 0.5 mm/min.

Results and Discussion

Density and Porosity

Table 1 shows the densities and porosity contents of pure magnesium and Mg-TiNi composites produced by rotary hot swaging performed at 450°C.

Table I. Density and porosity content of rotary swaged specimens.

Material	Density (gr /cm ³)	Porosity (%)
Magnesium	1.734±0.002	0.34
Mg-5 vol. %TiNi	1.97±0.002	0.4
Mg-10 vol. %TiNi	2.207±0.002	0.44
Mg-15 vol.% TiNi	2.44±0.001	0.6

As can be seen from Table I, the utilized manufacturing technique resulted in nearly dense materials. The porosity content of the manufactured materials are observed to increase as the content of TiNi reinforcement particles increase presumably due to an increase in interfacial area between matrix and TiNi particles. In addition, spherical pores with 1-5 μm diameters, which were

observed rarely, were found both in the matrix of magnesium and its composites perpendicular to swaging direction. Probably, these micro pores formed due to air entrapped in the powders during cold compaction stage which combined together to form larger pores as deformation and sintering proceeded.

X-Ray Diffraction

X-Ray diffractogrammes of both as-received and processed Mg and pre-alloyed TiNi powders are shown in Figure 2. Completely same X-ray diffractogrammes have been obtained for both as-received magnesium powders and manufactured magnesium rod samples such that mainly Mg peaks have been detected in the profile. In addition, a slight change is observed in the relative intensities of (10-10) and (0002) peaks detected at 32.24° and 34.48°, respectively, in the X-Ray diffractogrammes of processed Mg powders or rotary swaged Mg samples. The higher intensity of (10-10) peak of rotary swaged magnesium was attributed to the texture formation which is usually characterized as fiber texture. Another shallow peak characterized as MgO was also detected both in as-received magnesium powders and in swaged samples which indicates presence of MgO on the starting Mg powder particles and/or a slight oxidation during processing.

As can be seen in Figure 2, as-received TiNi powders contain only one phase which is known as B2 ordered austenite phase and exhibits super elastic behavior. On the other hand, X-Ray diffractogramme of Mg-TiNi composites labeled as (d) in Figure 2 consisted of peaks of both magnesium and B2 austenite phase. Similar to magnesium samples MgO peak was also observed in addition to Mg and B2 phase peaks.

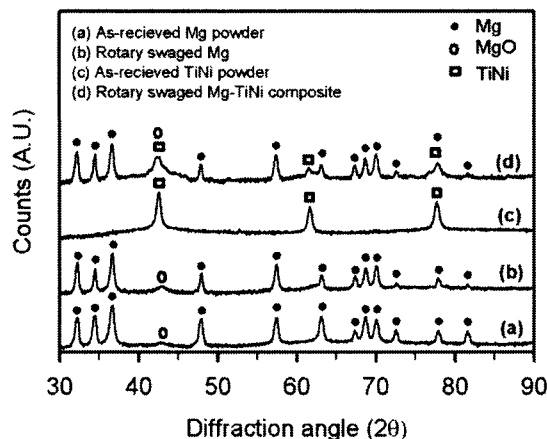


Figure 2. X-ray diffraction spectra of starting and processed Mg and TiNi pre-alloyed powders.

Microstructure

Microstructures of rotary swaged pure magnesium and magnesium composite samples are shown in Figure 3 and Figure 4. Nearly equi-axed particles, which are separated by partly continuous dark thin lines, were observed in the cross-sections of pure magnesium rods perpendicular to the rotary-swaging direction (Figure 3(a)). While the bright regions exhibited nearly 100% of magnesium as a result of EDS analysis, the dark lines which separate the starting powder particles revealed oxygen and magnesium contents about 8.19 wt. % and 91.81 wt. %, respectively, indicating the presence of MgO as previously found in X-Ray diffractogrammes. Magnesium starting powders generally contain oxide layers ranging between 1 and 2 nm [15].

EDS analysis taken from some starting powders exhibited oxygen content as high as 10 wt. %, while the oxygen content of the most of the powders couldn't be measured or observed to change around 1 wt. %. Because of that, the observed oxide layers some of which encountered inside the powder particles probably come from the starting powders and retain in the structure throughout the manufacturing stage. In addition to thin oxide layers observed in microstructure, at some regions of manufactured samples, especially at tripole junctions, nearly spherical small pores with 1-5 μm are detected. Similar to observed oxide layers, these pores appeared during the initial stage of production, namely cold pressing, as in irregular shaped ones and became spherical during rotary swaging at elevated temperatures as they cannot escape out of the structure.

The average grain sizes of pure magnesium and Mg-TiNi composite rods in the cross-sections perpendicular to rotary swaging direction were calculated as 45 μm, which is comparable to initial powder particle size. The oxide layers present on the surfaces of starting powder particles possibly hinder the grain growth and act as barriers to diffusion. The available sintering paths for this type of structures seem to be the broken oxide regions only, formed during powder consolidation stage, i.e. cold pressing and rotary hot swaging stage.

Micro structural examination of Mg-TiNi composites revealed a homogenous distribution of TiNi reinforcement particles throughout the magnesium matrix as shown in Figure 3(b). Similar to processed pure magnesium rods, MgO phase was also detected in Mg-TiNi composites as dark lines which separate the magnesium powder particles. In addition, as shown in Figure 4(a), a highly elongated structure was observed in the cross-sections of both magnesium and Mg-TiNi composite rods in directions parallel to the rotary swaging. As can be seen in Figure 4(a), the shape of the starting TiNi powders were preserved although the specimen matrix elongated in one direction upon hot swaging. The preservation of TiNi powder particle's shape was attributed to super elastic behavior of Ti-50.6 at. % Ni pre-alloyed powders as they contain only B2 austenite phase which is responsible for shape recovery. Apart from these, in the regions close to the surface of manufactured rods, each elongated structure were observed to contain nearly equi-axed grains whose dimensions are limited to the width of the elongated powder particles as shown in Figure 4(b). The equi-axed grains are believed to form as a result of dynamic recovery and recrystallization during rotary-hot swaging stage since the utilized deformation temperature during manufacturing was higher than the recrystallization temperature of the magnesium. Presence of equi-axed grain containing elongated structures, especially in the regions close to the surface, is the indication of non-homogenous deformation resulted by hot swaging operation.

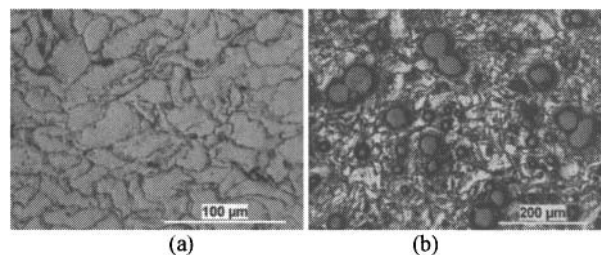


Figure 3. Optical micrographs of rotary swaged specimens in the cross section perpendicular to rotary swaging direction, (a) pure magnesium, (b) Mg-10 vol. % TiNi composite

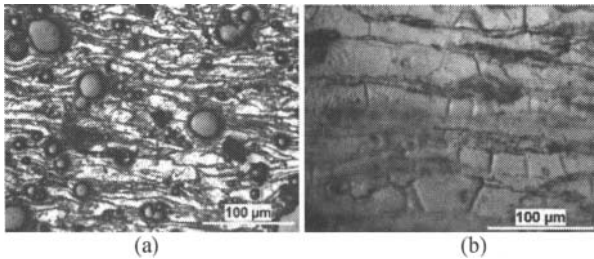


Figure 4. Optical micrographs of Mg-10 vol. % TiNi composite in the cross section parallel to the rotary swaging direction, (a) Elongated magnesium particles and spherical TiNi particles, (b) Recrystallized grains in elongated structures.

In addition, micro structural examination conducted in the interface region of Mg matrix/TiNi particles revealed neither cavities nor reactions. A non-porous continuous interface and good interfacial bonding have formed between dark TiNi particles and bright Mg matrix as shown in Figures 5(a) and 5(b).

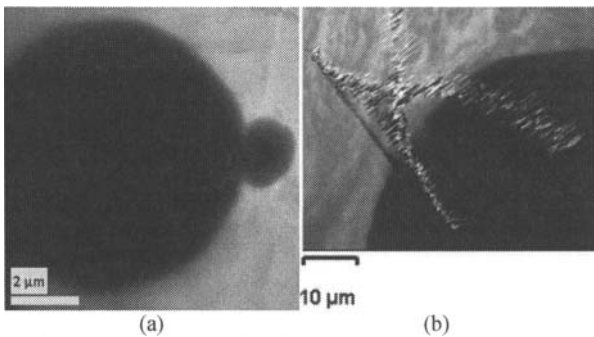


Figure 5. (a) TEM micrograph showing non-porous interface between magnesium matrix and TiNi particles in Mg-10 vol.% TiNi composite, (b) EDS spectra across a TiNi particle

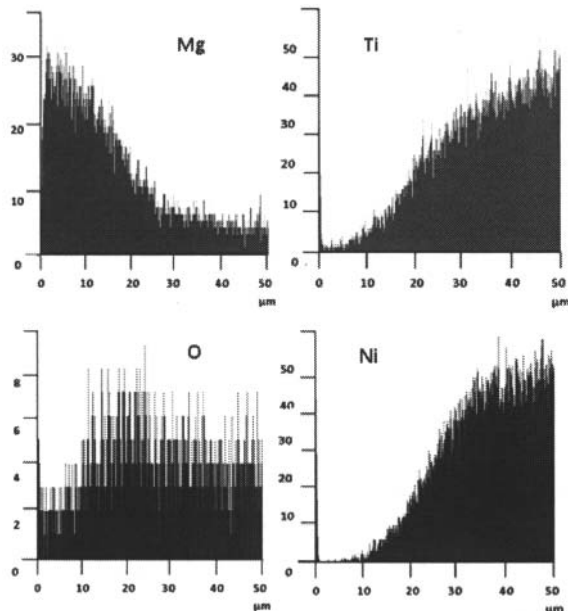


Figure 6. EDS line analysis of the elements across TiNi particle and Mg/particle interface in Mg-10 vol. % TiNi composite

Figure 5(b) and Figure 6 show EDS line spectra across a TiNi particle and magnesium matrix in Mg-10 vol. % TiNi composite. Although, both Ti and Ni peaks were observed only in TiNi particles, a slight amount of magnesium was detected in TiNi particles. On the other hand, oxygen is distributed both in magnesium matrix and TiNi particles, but with a clear enrichment in the Mg matrix-TiNi interface (Figure 6) which indicates the presence of oxides in the interfacial region. In this study, detected oxygen peaks were attributed to the oxygen mainly coming from MgO in addition to oxides formed on TiNi powder surfaces.

Compression Tests

Stress-strain diagrams of compression tested pure magnesium and Mg-TiNi composites are shown in Figure 7. Upon compression the samples under quasi-static conditions stress-strain curves similar to wrought alloys has been obtained with almost linear elastic behavior at small strains, followed by yield and strain hardening up to an ultimate stress. Subsequent to a peak stress fracture occurred after a small straining whose magnitude depends on the sample purity and content of the TiNi reinforcement particles.

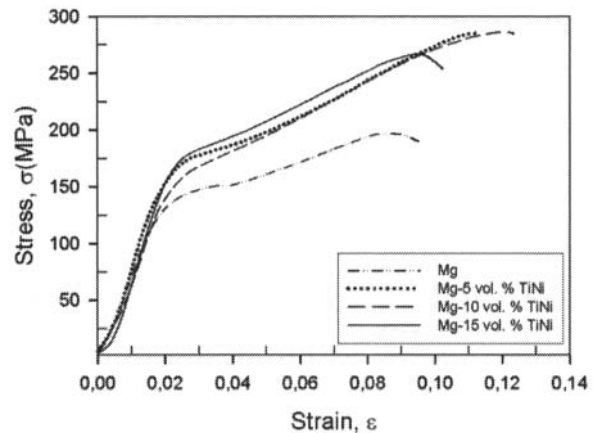


Figure 7. Stress-strain diagrams of compression tested samples.

The mechanical properties of magnesium and magnesium alloys, i.e. elastic modulus and yield strength, may be enhanced by grain refinement, alloying and subsequent heat treatment or by the composite production through the addition of stiffer particles to the matrix in solid or liquid states. In this study, only the effect of reinforcement particles on mechanical properties are discussed and the results of the compression behavior revealed a significant increment in compression yield and peak stresses of magnesium as a result of TiNi addition, which were around 130 and 210 MPa, respectively. The yield strength of the composites was observed to increase linearly with an increase in TiNi reinforcement particle content as shown in Figure 8 and about 25% increment in yield strength was detected in Mg-15 vol. % TiNi samples. Yield strength of an alloy depends on the obstacles, i.e. grain size of the alloy, and the size and the distance between precipitates, which render the motion of dislocations. TiNi particles in this study possibly serve as obstacles for dislocation motion and result in an increased $\sigma_{0.2}$ as their content in the matrix increase. In contrast to yield strength, the effect of TiNi particles on peak or maximum stress at which internal micro pores or cracks form was not linear. Although the peak stress of magnesium is enhanced by 35% as a result of adding around 5 or 10 vol. % of TiNi, presence of 15 vol.

% TiNi particles in matrix didn't exhibited an increment as high as the other composites show. As can be seen in Figure 8, the effect of reinforcement TiNi particle content on maximum stress diminishes when the reinforcement quantity reaches to 15 vol. %. The observed non-linear effect of TiNi particle content on peak stress of Mg-TiNi composites was attributed to the higher interfacial area created by increased TiNi reinforcement content, which may serve as crack initiation sites. Observed increment in peak stress of composites is generally attributed to two facts, i.e. grain refinement and load transfer from matrix to reinforcement. In addition to these two reasons it has been shown that MgO in magnesium may act as secondary reinforcement although it reduces the ductility of the alloy.

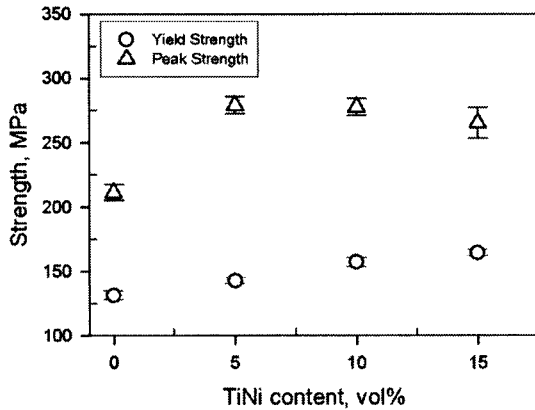


Figure 8. Yield and peak strength values of compression tested magnesium and Mg-TiNi composites.

In contrast to other magnesium composites containing various types of reinforcement particles, i.e. SiC, ductility of the manufactured Mg-TiNi composites were increased notably compared to pure magnesium samples. As shown in Figure 7, about 40% increment is observed in ductility level of pure magnesium when 10 vol. % TiNi is added. Similar effect was also seen in the study of Hassan and Gupta [1] conducted on Mg-Ti composites in which the increment in ductility level by the addition of Ti was attributed to diffusional dissolution of Ti in magnesium matrix. In the present study, the increase in ductility level is attributed to the load transfer from magnesium matrix to interface and TiNi particle. For Mg-15 vol. % TiNi composite, on the other hand, elongation value at fracture point was decreased and composite's elongation value reach to that of pure magnesium probably due to increased number defects in matrix or interfacial area due to increased quantity of reinforcement particles.

Upon testing the samples under compression loads, fracturing occurred after small straining beyond their peak stress values. Occasionally fracturing took place at an angle 45° to the loading axis in some samples, while most of the samples failed in regions parallel to the loading directions which were also parallel to the rotary swaging direction (Figure 9 (a)). Failure took place in central weakest regions of rod samples since the severity of deformation in rotary swaged samples decreases as going from sample's surface to center. Examination of fracture surfaces of pure magnesium revealed brittle type intergranular and transgranular mode of fractures which followed the MgO present between the powder particles and the magnesium matrix, respectively, as shown in Figure 9(b). Application of loads

beyond a critical value of composite materials results in fracture due to concentration of load in matrix, reinforcement particle or the interfacial area between matrix and reinforcement. As it can be seen in Figure 9(c) and 9(d), all the Mg-TiNi composites fractured by de-bonding of interface as a result of initiation of localized damage upon compression loading. During failure de-bonded TiNi particles also caused fracturing of the magnesium matrix (Figure 9(d)).

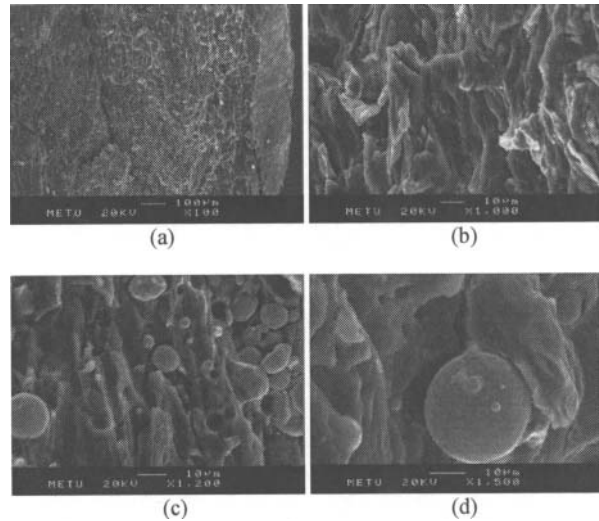


Figure 9. Fracture surfaces of samples, (a), (b) magnesium, (c), (d) Mg-10 vol. % TiNi.

Conclusions

Present study have shown the feasibility of pure magnesium and Mg-TiNi composite rods production by rotary hot swaging which uses cold pressed green compacts. Highly dense rod samples were manufactured and they were observed to contain very few amount of spherical micro pores formed due to entrapped air during cold compaction step. Recrystallized equi-axed grains containing elongated structures were produced due to combined effect of high deformation rate and high temperature. Recrystallized grains were observed especially in the surface regions since the rotary swaging deformation is more severe in the surfaces of samples. Although the grains of magnesium matrix elongated in the direction parallel to the rotary swaging direction, the shape of the spherical TiNi particles was preserved due to their super elastic behavior. EDS analysis has shown that the processed samples of pure magnesium and Mg-TiNi composites contain thin layers of MgO which is mainly due to starting powders and processing conditions. In addition, MgO was also detected in interfacial region of Mg-TiNi composites which may degrade the mechanical properties of composites. The addition of TiNi particles resulted in a linear increase in the yield strength of Mg-TiNi composites while the enhancement of maximum or peak strengths of composites was limited. As high as 280 MPa peak strength value was obtained in Mg-TiNi composites, which contain about 10% TiNi by volume. However, higher content of TiNi particles in magnesium resulted in a decrease both in peak stress and ductility of the samples presumably due to an increase in defective interface areas between TiNi particles and magnesium matrix. Fracture in magnesium and Mg-TiNi composite samples was observed to start in the central regions parallel to rotary swaging

deformation probably due to non-homogenous nature of hot-swaging in surface and central regions of samples. In magnesium and Mg-TiNi composites both intergranular and transgranular modes of failure were observed and fracture cracks passed followed either the MgO present between prior powder particles and the grains of magnesium matrix. Fracture surfaces of Mg-TiNi composites were similar to those of pure magnesium samples except the circular cup regions formed due to de-bonding of TiNi particles upon compression loading.

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