

Microstructural Characterization of Cast Magnesium Matrix Composites by Raman Microscopy

M.A. Malik^{a*}, K.N. Braszczyńska-Malik^b, K. Majchrzak^a

^a Division of Chemistry, ^b Institut of Materials Engineering, Faculty of Materials Processing Technology and Applied Physics, Częstochowa University of Technology, Al. Armii Krajowej 19, 42-200 Częstochowa, Poland

* Corresponding author. E-mail address: malik@wip.pcz.pl

Received 16.07.2012; accepted in revised form 04.09.2012

Abstract

Cast magnesium matrix composites reinforced with silicon carbide particles were investigated by using Raman microscopy. 3C, 4H and 6H polytypes of SiC particles were identified in the investigated composites. Additionally, Mg₂Si compound was detected by Raman microscopy in the composites microstructure.

Keywords: Magnesium matrix composites, SiC, Mg₂Si, Raman spectroscopy

1. Introduction

Among various metal matrix composites (MMCs), magnesium matrix composites (MgMC) reinforced with ceramic particles deserve special consideration, due to their low density, high specific stiffness and strength, high damping capacity and good dimensional stability [1-11]. The microstructure and the properties of MMCs depend on various factors, which include: (i) distribution, a size and a volume fraction of reinforcing particles, (ii) a type of matrix alloy, (iii) a fabrication method and process parameters and (iv) a bonding type between components [9, 12-15].

Silicon carbide particles are the most widely used as reinforcing phase in MMCs due to their low price and appropriate mechanical properties. In particular, the Mg-SiC system is characterised by (i) very good wettability of SiC by molten Mg, (ii) very high stability of SiC in liquid Mg, (iii) a precipitate-free SiC/Mg interface and (iv) strong adhesive bonding between SiC and the magnesium matrix [16-22]. SiC exhibits polytypism

which originates from differences in stacking faults of Si-C double layers along [111] or [0001] directions [23-25]. Different arrangements of *n* bilayers within the unit cell cause the formation of cubic (C), hexagonal (H), or rhombohedral (R) structures. The most common polytypes of SiC are: 3C (zinc blend type, β-type SiC), 4H and 6H (α-type SiC). Their structures are schematically presented in Fig.1.

SiC particles, which are commercially used for the fabrication of MMCs, very often contain considerable amounts of impurities, such as: graphite, Si, SiO₂ and metal oxides (Fe₂O₃, Al₂O₃, CaO, MnO₂). It should also be noted, that SiC particles tend to be covered with a SiO₂ film due to the oxidising processes during their manufacturing [6,8]. The silicon oxide film can remain on the SiC particles in MgMC when contact time of molten magnesium alloy with the particles is relatively short during the fabrication process. On the other hand, the sufficiently long contact time of SiC with molten alloy leads to the formation of small amounts of Mg₂Si phase in the composite matrix due to the reaction of SiO₂ and Mg.

Raman spectroscopy is a powerful technique for the identification of SiC polytypes [26-30]. This work is focused on the application of Raman microscopy for the characterization of individual SiC particles in MgMCs, which is important for better understanding of nucleation processes of the metal matrix occurring on different SiC polytypes. Since SiC polytypes differ in electrical conductivity [31], the possibility of their identification in the composite matrix is also significant for recognition of micro-galvanic corrosion mechanisms in MgMCs.

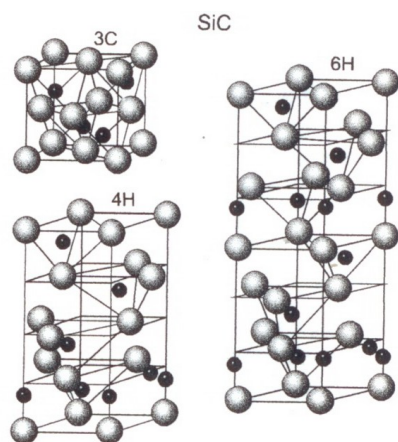


Fig. 1. Unit cells of silicon carbide polytypes [33]

2. Experimental materials and procedures

The investigated composites containing 20 wt. % of SiC particles were prepared on the basis of Mg-Si hypo-eutectoid alloy (1 wt. % Si) and commercial AM50 alloy. Silicon carbide particles with an average diameter of 40 μm (manufactured by Fabryka Materiał \acute{o} w i Wyrob \acute{o} w Ściemych KORDUN S.A.) were used as the reinforcing phase. Composites were fabricated by means of a simple and non-expensive casting method involving mechanical mixing of the molten metal with the SiC particles in a steel crucible under a protective atmosphere, and subsequent cast into a metal moulds. The fabrication process parameters, such as the time and rate of suspension stirring, a casting temperature, a mould temperature, were chosen experimentally for each composite material.

Samples for optical and Raman microscopic investigations were prepared by the conventional grinding with waterproof abrasive paper of grain size 4000 and subsequent mechanical polishing with a lubricant containing 0.1 μm diamond particles (Buehler, U.S.A.). The samples were etched in 1% solution of concentrated nitric acid in ethanol for 5 second.

An EZRaman-L spectrometer with a diode laser (excitation wavelength 785nm, power 50 mW) from Enwave Optronics, Inc. (Irvine, CA, U.S.A.) was used for recording Raman spectra. The Raman spectrometer was coupled with an optical microscope equipped with a HC PL Fluotar objective with a numerical aperture of 0.80 and a magnification of 50x (Leica Microsystems, Wetzlar, Germany). The laser spot size was equal to ca. 3 μm .

3. Results and discussion

Fig. 1 shows an optical micrograph of the Mg/SiC composite. Apart from SiC particles Mg+Mg₂Si eutectic is visible. A Raman spectrum recorded for the particle indicated by the arrow in Fig. 1 exhibits two sharp bands at 789 and 970 cm^{-1} , corresponding to the transversal optic phonon (TO) and the longitudinal optic phonon (LO) modes of 3C-SiC [26-30]. Two different patterns of Raman spectra, which were recorded from other SiC particles, are shown in Figs 3 and 4. The spectrum, presented in Fig 3, consisted of the three peaks at 771; 786 and 798 cm^{-1} (degenerated TO mode) and 970 cm^{-1} (LO mode) is characteristic for 6H-SiC [26-30]. In Fig. 4, the Raman spectrum for the third polytype of SiC, which was found in the investigated Mg/SiC composite, is presented. This spectrum is consisted of a sharp and intense band at 788 cm^{-1} with a shoulder at 798 cm^{-1} (TO) and a very weak band at 970 cm^{-1} (LO), which is characteristic for 4H-SiC polytype [26-30].

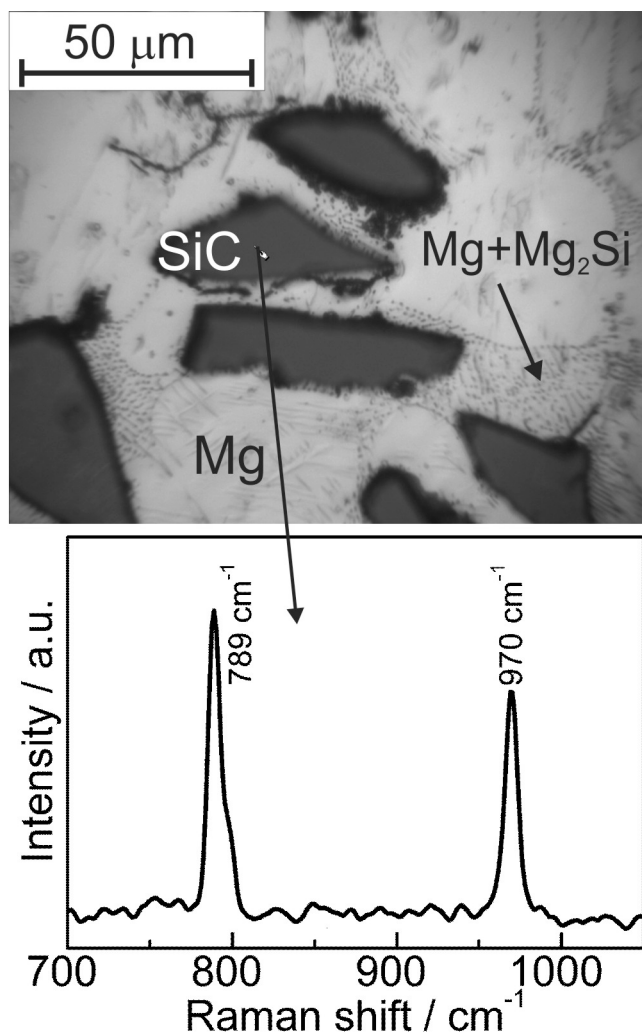


Fig. 2. Optical micrograph of the Mg/SiC composite surface and Raman spectrum recorded from the 3C-SiC particle

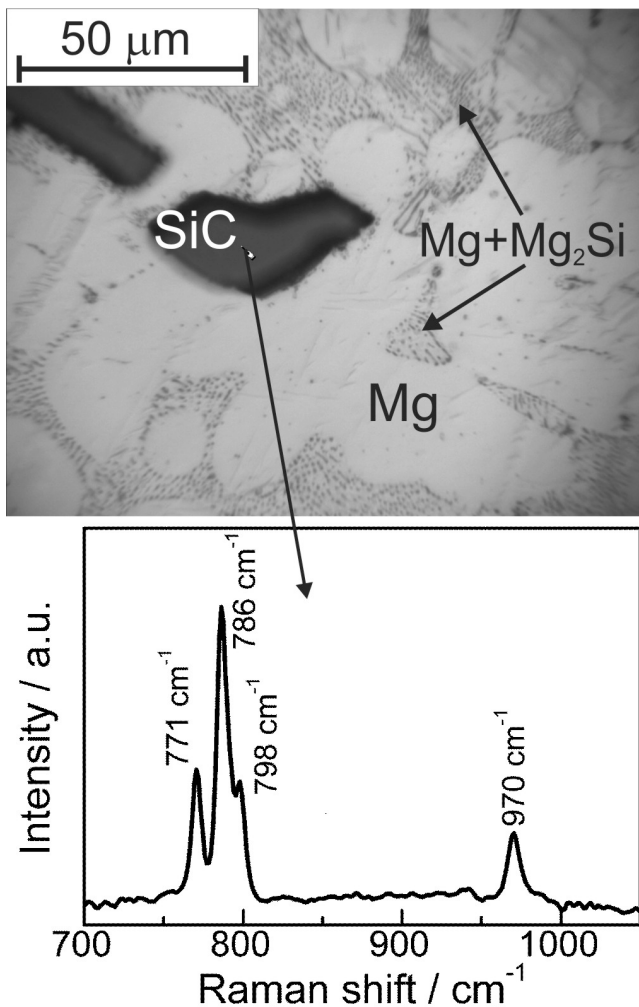


Fig. 3. Optical micrograph of the Mg/SiC composite surface and Raman spectrum recorded from the 6H-SiC particle

The results presented above indicate that three different polytypes exist in the cast Mg/Mg₂Si composite. This information can be useful for considerations on the influence of the reinforcing particles on the nucleation of the metal matrix. Lattice correlation between solid state ceramic and metal phase nucleating on it, is one of the factors influencing on nucleation processes.

Raman spectroscopy can be applied for identification of Mg₂Si compound which can be a deliberately introduced component in magnesium composites or which can form due to the reaction between liquid magnesium and SiO₂. Fig. 4 shows an optical micrograph of the AM50/SiC composite. The composite matrix is consisted of α -phase (a solid solution of Al in Mg) and $\alpha+\gamma$ eutectic (where γ is Al₁₂Mg₁₇). Apart from SiC reinforcing particles another type of particles was found in the composite microstructure. The Raman spectrum recorded from the particle of this type is presented in Fig. 5. The presence of two peaks at 259 and 340 cm⁻¹ is characteristic for Mg₂Si [32]. It is likely that Mg₂Si was formed by the reaction of SiO₂, which was present on the SiC surface, with molten magnesium. It is worth noting that

the ability of Raman microscopy to identify individual particles of Mg₂Si is very helpful in studies of multiphase composites. Detection of Mg₂Si compound is difficult by other methods (e.g. X-ray diffraction) because of its very low content in a bulk material.

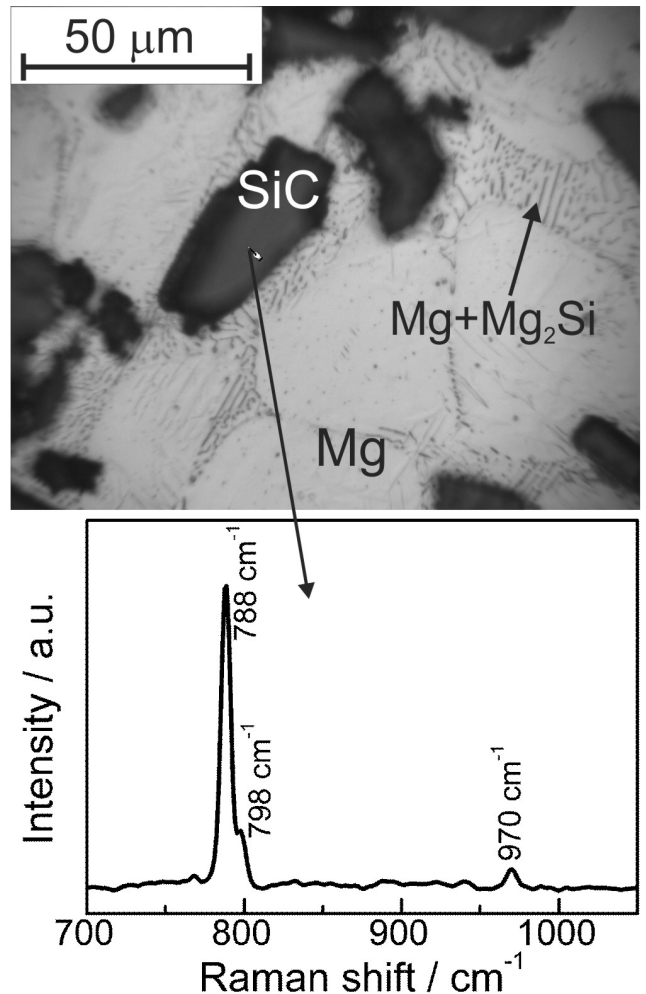


Fig. 4. Optical micrograph of the Mg/SiC composite surface and Raman spectrum recorded from the 4H-SiC particle

4. Summary

The application of Raman microscopy for microstructure characterisation of cast magnesium matrix composites provides valuable information on polytypes of SiC reinforcement. In the investigated Mg/SiC composite three SiC polytypes were detected, namely 3C, 4H and 6H. Raman microscopy can also be used to identify Mg₂Si compound, which can be present in magnesium matrix composites. This is important because due to very low mass fraction of Mg₂Si compound in the cast AM50/SiC composite, this phase is difficult to detect by other methods (e.g. X-ray diffraction analysis) commonly used for microstructural studies of magnesium matrix composite materials.

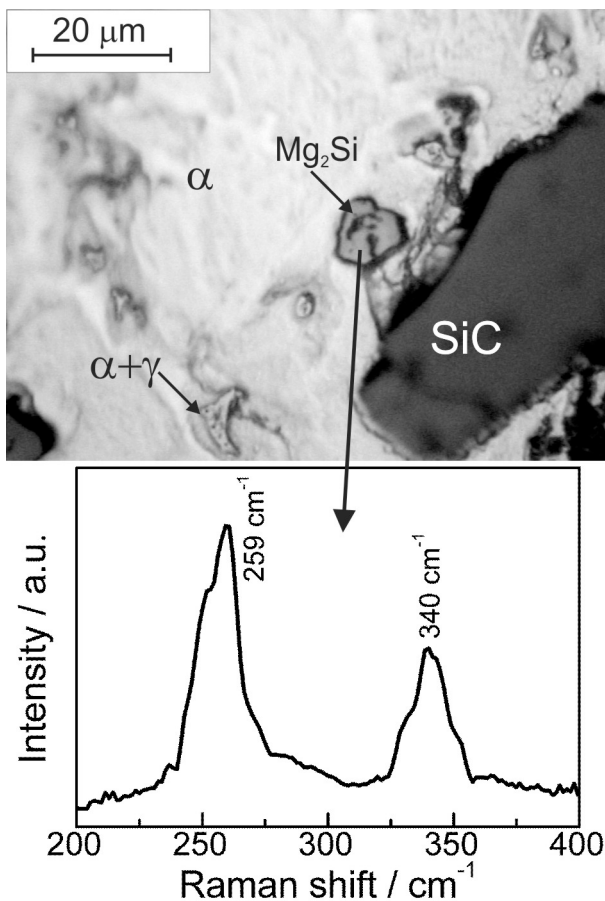


Fig. 5. Optical micrograph of the AM50/SiC composite and Raman spectrum recorded from the Mg_2Si particle

Acknowledgements

This work was supported by National Science Centre under the project no. N N204 270138.

References

- [1] Saravann, R.A. & Surappa, M.K. (2000). *Materials Science and Engineering*. 276 A, 108-116.
- [2] Cai, Y., Tan, M.J., Shen, G.J. & Su, H.Q. (2000). *Materials Science and Engineering*. 282 A, 232-239.
- [3] Cai, Y., Taplin, D., Tan, M.J. & Zhou, W. (1999). *Scripta Materialia*. 41(9), 967-971.
- [4] Ugandhar, S., Gupta, M. & Sinha, S.K. (2006). *Composite Structures*. 72, 266-272.
- [5] Cai, Y., Shen, G.J. & Su, H.Q. (1997). *Scripta Materialia*. 37(6), 737-742.
- [6] Braszczyńska, K.N., Bochenek, A. (2000). *La Revue de Metalurgie*. 1455-1462.
- [7] Chua, B.W., Lu, L. & Lai, M.O. (1999). *Composite Structures*. 47, 595-601.
- [8] Braszczyńska, K.N., Lityńska, L., Zyska, A. & Baliga, W. (2003). *Materials Chemistry and Physics*. 81, 326-328.
- [9] Bochenek, A. & Braszczyńska, K.N. (2000). *Materials Science and Engineering A*. 290 A, 122-127.
- [10] Lim, S.C.V. & Gupta, M. (2001). *Materials Research Bulletin*. 36, 2627-2636.
- [11] Hu, H. (1998). *Scripta Materialia*. 39(8), 1015-1022.
- [12] Poddar, P., Srivastava, V.C., De, P.K. & Sahoo, K.L. (2007). *Materials Science and Engineering A*. 460-461, 357-364.
- [13] Zheng, M., Wu, K., Yao, C., Sato, T., Tezuka, H., Kamio, A. & Li, D.X. (1999). *Material Letters*. 41, 57-62.
- [14] Manoharan, M., Lim, S.C.V. & Gupta, M. (2002). *Materials Science and Engineering A*. 333 A, 243-249.
- [15] Jayamathy, M., Lailas, S.V., Kumar, K., Seshan, S. & Srivatsan, T.S. (2005). *Materials Science and Engineering A*. 393 A, 27-35.
- [16] Braszczyńska, K.N. & Bochenek, A. (2000). *Science and Engineering of Composite Materials*. 9(3), 149-158.
- [17] Cao, G., Konishi, H. & Li, X. (2008). *Materials Science and Engineering A*. 486, 357-362.
- [18] Thakura, S.K. & Dhindaw, B.K., *Wear* 2001, 247, 191-201.
- [19] Rudajevova, A. & Lukac, P. (2002). *Materials Science and Engineering A*, 324 A, 118-121.
- [20] Moll, F., Chmelik, F., Lukac, P., Mordike, B.L. & Kainer, K.U. (2000). *Materials Science and Engineering A*. 291 A, 246-249.
- [21] Braszczyńska-Malik, K.N. (2007). *Kompozyty (Composites)*. 7(1), 51-55.
- [22] Braszczyńska, K.N. (2003). *Zeitschrift für Metallkunde*. 94(2), 144-148.
- [23] Zhu, Y.O., Sekine, T., Kobayashi, T. & Takazawa, E. (1998). *Journal of Materials Science*. 33, 5883-5890.
- [24] Daulton, T.L., Bernatowicz, T.J., Lewis, R.S., Messenger, S., Stadermann, F.J. & Amari, S. (2003). *Geochimica et Cosmochimica Acta*. 67(24), 4743-4767.
- [25] Fissel, A. (2001). *Inorganic Materials*. 3, 1273-1275.
- [26] Ward, Y., Yound, R.J. & Shatwell, R.A. (2004). *Journal of Materials Science*. 39, 6781-6790.
- [27] Gouadec, G. & Colomban, P. (2001). *Journal of the European Ceramic Society*. 21, 1249-1259.
- [28] Li, X-B., Chen, Z-Z. & Shi, E-W. (2010). *Physica B*. 405, 2423-2426.
- [29] Harima, H. (2006). *Microelectronic Engineering*. 83, 126-129.
- [30] Nakashima, S., Kisoda, K., Niizuma, H. & Harima, H. (1996). *Physica B*. 219-220, 371-373.
- [31] Sadow, S.E. & Agarwal, A. (Eds) (2004). *Advances in Silicon Carbide Processing and Applications*, Artech House, Boston.
- [32] Atanassov, A. & Baleva, M. (2007). *Thin Solid Films*. 515(5), 3046-3051.
- [33] Braszczyńska, K.N. Charakterystyka kompozytu na podstawie stopu magnezu umacnianego cząstkami węgla krzemu, PhD thesis (in Polish)