

Synthesis of Ti_3SiC_2 -Bicarbide Based Ceramic by Electro-Thermal Explosion

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Abstract

A polycrystalline dense Ti_3SiC_2 based ceramic material has been produced by several techniques. The effect of addition of TiC and SiC is also studied. The Ti_3SiC_2 material shows extraordinary electrical, thermal and mechanical properties. Furthermore, it shows a damage tolerance capability and oxidation resistance. In this work, we have synthesized Ti_3SiC_2 by electro-thermal explosion chemical reaction (ETE) with high current density (900 Amperes/a.u) followed by uniaxial pressure. The structural properties of the as-prepared materials are studied by x-ray diffraction (XRD), scanning electron microscope (SEM) and energy dispersive x-ray spectroscopy (EDX) techniques. The chemical cartography, imaging and electronic properties are investigated using Ultra-STEM and electron high energy loss resolution spectroscopy (EELS) techniques, respectively. The surface of Ti_3SiC_2 is characterized by means of X-ray photoelectron spectroscopy (XPS). High resolution C 1s, Si 2p, Ti 2p, Ti 3s core level spectra are explained in terms of crystallographic and electronic structure. Valence band spectrum is performed to confirm the validity of the theoretical calculations.

Keywords

Ti_3SiC_2 Based Ceramic, Intermetallic, Bicarbide, Electro-Thermal Explosion Reaction (ETE), Valence Band Spectra, Core Level Excitation

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1. Introduction

The investigations of the ternary ceramic Ti_3SiC_2 show that it has exceptional properties [1]-[8]. Similar electrical and thermal conductivity at room temperature exceeds those of Ti metal; the former possesses excellent oxidation resistance (up to 1400°C), a resistance to thermal shock; a moderately low coefficient of thermal expansion. Ti_3SiC_2 also possess an unusual array of mechanical properties (including good compressive strength and a high Young's modulus all along with low hardness and some evidences for ductility) the ceramic is readily processed by standard tools. Also, it has been shown that large, highly oriented polycrystals of Ti_3SiC_2 undergo auto-deformation plastically at room temperature by means of shear and kink band formation. The phase was first synthesized by Jeitschko and Nowotny back on 1967 [9]. Recent interest in the phase is due to the availability of improved material, which leads to high purity polycrystalline single-phase samples of near-theoretical density. The structure has been reported [10] as being hexagonal in space group P 63/mmc (194) with $a < 0.307$ nm and $c < 1.769$ nm. It shows a great resistance against oxidation, extreme hardness, and above all, it can retain its strength to temperatures that makes the best superalloys available today usable. Up to now, no other material has shown such a combination of properties like machinability, strength, and ductility at elevated temperatures and nonsusceptibility to thermal shock. It has an excellent self-lubricating property, so it can be better than graphite for rotating electrical contacts for ac motors. It is a promising candidate for ceramic engines. More details about the technological importance of Ti_3SiC_2 can be found in reference [1].

As far as the experiments of this compound are concerned, Jeitschko and Nowotny [9] have synthesized Ti_3SiC_2 via chemical reaction between TiH_2 , Si, and graphite at 2000°C . Goto and Hirai synthesized Ti_3SiC_2 through the chemical-vapor deposition technique [11]. Very little is known about this material. Panczyk *et al.* [10], shown that the melting point of Ti_3SiC_2 occurs at around 3000°C . Pampuch *et al.* [12] produced a hard Ti_3SiC_2 -based material with a high young's modulus of 326 GPa through self-propagating high-temperature synthesis and ceramic processing. Barsoum and El-Raghy [1] fabricated polycrystalline bulk samples of Ti_3SiC_2 by reactively hot-pressing a blend of Ti, graphite, and SiC powders at 40 MPa and 1600°C for 4 h. Onodera *et al.* [13] [14] at high pressure measured the value of the bulk modulus at about 20666 MPa. The study of the electronic structure and chemical bonding gave a better insight into the important Ti_3SiC_2 materials properties. In order to study the electronic structure of Ti_3SiC_2 , full-potential linear-muffin-tin-orbital methods (FPLMTO) was used [15]. The calculations were based on the local-density approximation (LDA) and the Hedin-Lundqvist [12] parameterization was used for the exchange and correlation potential. Basis functions, electron densities, and potentials were calculated without any geometrical approximation [15]. The radial basis functions within the muffin-tin spheres are linear combinations of radial wave functions and their energy derivatives which are computed at energies appropriate to their site and principal as well as orbital atomic quantum numbers, whereas outside the muffin-tin spheres the basic functions are combinations of Neuman or Hankel functions [16] [17]. For the sampling of the irreducible wedge of the Brillouin zone, the special k-point methods were used [18]. Each calculated eigenvalue was associated with a Gaussian broadening of width 20 mRy. Ti_3SiC_2 belongs to the D6h 4-P63/mmc space group of hexagonal crystalline structure [19], in which, its primitive cell contains two formula units. The observed lattice parameters were $a = 53.064\text{\AA}$ and $c = 517.650\text{\AA}$. c/a was optimized by calculating of the total energy for different c/a values at the equilibrium volume. The calculated value of c/a was around 5.77 and it was in a very good agreement with the experimental value of 5.76. The calculated value of the bulk modulus was 225 GPa and is in a good agreement with the experimental value [17]. If one compares it with TiC, which has a bulk modulus of 240 GPa [20], one find that they are comparable; the density of states (DOS) for Ti_3SiC_2 and the corresponding band structure is shown. The lowest-lying states around 10 eV are originated from the non-metal C 2s states. If one compares this with the DOS of TiC [21], there are also C 2s states which lie around 10.0 eV. Between 9.0 and 6.0 eV below the E_F , there are states which are derived from the Si 3s states. However, one can see a big gap in TiC in this energy range, due to the absence of Si. The states just below E_F are dominated by strongly hybridizing bonding states combinations of Ti 3d orbitals of eg symmetry, C and Si p-derived orbitals. The states above E_F contain antibonding Ti 3d-dominated orbitals of eg symmetry, C and Si p states. In the case of TiC, the states near E_F show the same character. E_F in TiC lies exactly in the pseudo gap, whereas in Ti_3SiC_2 it has moved outside this gap. This indicates that TiC should be harder than Ti_3SiC_2 .

In summary, it is shown that the modern electronic structure theory is sufficiently developed to give an accurate description of Ti_3SiC_2 . The calculated volume and structural parameters are in a very good agreement with the experimental values. The chemical bonding appears to be similar to that of TiC.

A polycrystalline material based on a Ti_3SiC_2 compound and produced by sintering the powders synthesized through the electro-thermal explosion (ETE) method has been shown to have the advantages of ceramic materials combined with some metal-type plastic properties. Reaction in ternary Ti-Si-C system under the ETE regime may produce either TiC, SiC, TiSi_2 , $\text{Ti}_5\text{Si}_3\text{C}$ or Ti_3SiC_2 : each in a quantity depending on the starting mixture composition [22]. However, until now, attempts to produce a Ti_3SiC_2 single phase resulted at the very most in 10% TiC amount and traces of titanium-silicon intermetallic as ascertained. Polycrystalline dense Ti_3SiC_2 based ceramic has been produced by several techniques and the effect of addition of TiC and SiC is also studied [23]-[33].

2. Experimental Procedures

Polycrystalline high-purity Ti, C, and Si powders (99.99% pure) of the nominal composition 3Ti/Si/2C, was mechanically alloyed. The powder was prepared from Goodfellow SARL powder in a high-energy Fritsch planetary ball mill with a balls/mass ratio of 13/1. The rotation speed could be varied within the range 450 - 650 rpm. In order to avoid oxidation during alloying, the ball mill was filled with high-purity argon gas. The vial was opened in 10 - 12 h to assure high homogenization and repeated fractioning. The powders were compacted into small discs (2 - 3- 13 mm) at the compacting pressure $P = 6000$ psi and then subjected to ETE in Argon or in vacuum at 900 A° during 55 seconds followed by uniaxial pressure.

The structural properties were determined by XRD using a Philips diffract-meter (Co- $K\alpha$ radiation). Further structural characterizations were carried out by energy-dispersive X-ray microanalysis (EDX), scanning electron microscopy (SEM), field emission scanning electron microscopy (FESEM), and optical microscopy (OM). SEM, FESEM micrographs were taken with a JEOL JSM-840A instrument equipped with an EDX accessory to check the elemental composition of the system [33]. XPS analysis were carried out in an ESCALAB Mk.II instrument using Al $K\alpha$ X-rays as the source of excitation, the gun being run at 13 kV and 20 mA. Survey spectra were obtained over a kinetic energy range of 80 - 600 eV, in steps of 0.5 eV (CAE mode) of 100 eV. Detailed data were obtained for selected ranges of photoelectron energies (*i.e.* Ti 2p , C 1s , O 1s and Si 2p lines) in steps of 0.2 eV and at a pass energy of 20 eV; the regions were scanned repetitively in order to improve the signal-to-noise ratio. X-ray induced AES (XAES) structures data of Si KLL were obtained from excitation with Bremsstrahlung radiation; repetitive scans were carried out at pass energy of 20 eV and for steps of 0.2 eV. The XPS data were processed by subtraction of a Shirley-type background; minor charge shifting was accounted for by setting the graphitic adventitious C 1s component to 284.6 eV and by ensuring internal self-consistency of the shifted spectra. The noisiest of the spectra (e.g. Si KLL spectra) were smoothed, but not otherwise processed. The spectra from as-mounted, specimens were characterized by surface oxide and some adventitious surface carbon. Both were substantially removed by a light ion etch (ca. 6 mA min cm^{-2} dose of 3 keV) [6].

In this work, we have produced Ti_3SiC_2 with chemical reaction by electro-thermal explosion with high current density (900 A°) followed by uniaxial pressure. The present study was examined the structural properties of materials using XRD, SEM and EDX. The chemical cartography, imaging and electronic properties will investigated using Ultra-STEM and electron high energy loss resolution spectroscopy (EELS), respectively. The Ti_3SiC_2 is studied by means of X-ray photoelectron spectroscopy. High resolution C 1s, Si 2p , Ti 2p ,Ti 3s core level spectra are inspected in terms of crystallographic and electronic structure. Valence band spectra will be performed to confirm the validity of the theoretical calculations.

3. Results and Discussion

The final product has been investigated and analyzed by means of scanning electron microscopy (SEM), XPS, X-ray diffraction (XRD) and EDX. The observations have proved that the elongation slabs with rounded corners of well fused Ti_3SiC_2 grains form a matrix within which some rounded TiC and less frequent angular SiC inclusions are present (Figure 1). Figure 1 shows the SEM images of the Ti_3SiC_2 , bulkbicide base ceramic (TiC, SiC) produced via electro-thermal-explosion reaction (ETE, Figure 2) of crystalline elemental powder of Ti, C, and Si after balls milling (13/1 (balls/mass)) has been synthesized (Figure 3).

The phase composition after ETE reaction has been studied and analysed using the X-ray diffraction technique (Figure 4).

The morphology of the as-prepared materials was observed by scanning electron microscopy (SEM) as shown in the Figure 1. Further studies have been carried out by energy-dispersive X-ray microanalysis (EDX) (Figure 5)

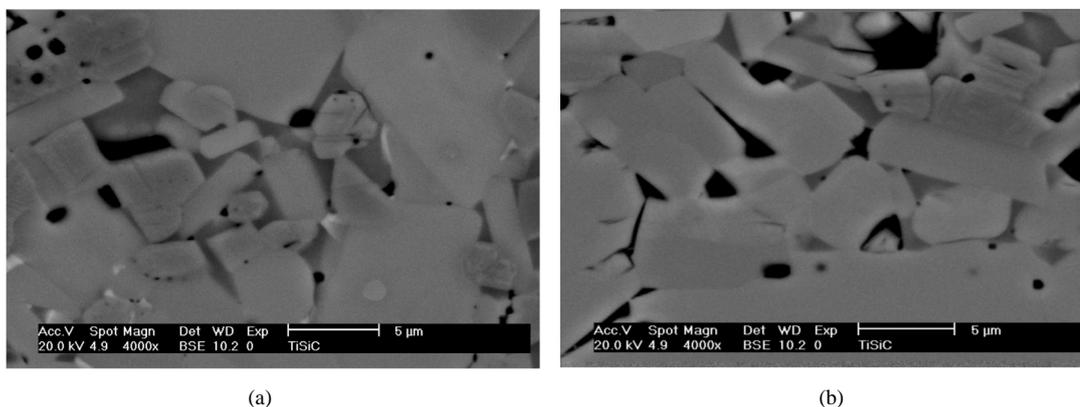


Figure 1. SEM imaging: (a, b) representation of bulk Ti_3SiC_2 corresponding to the carbides TiC, SiC and may be intermetallic TiSi_2 .

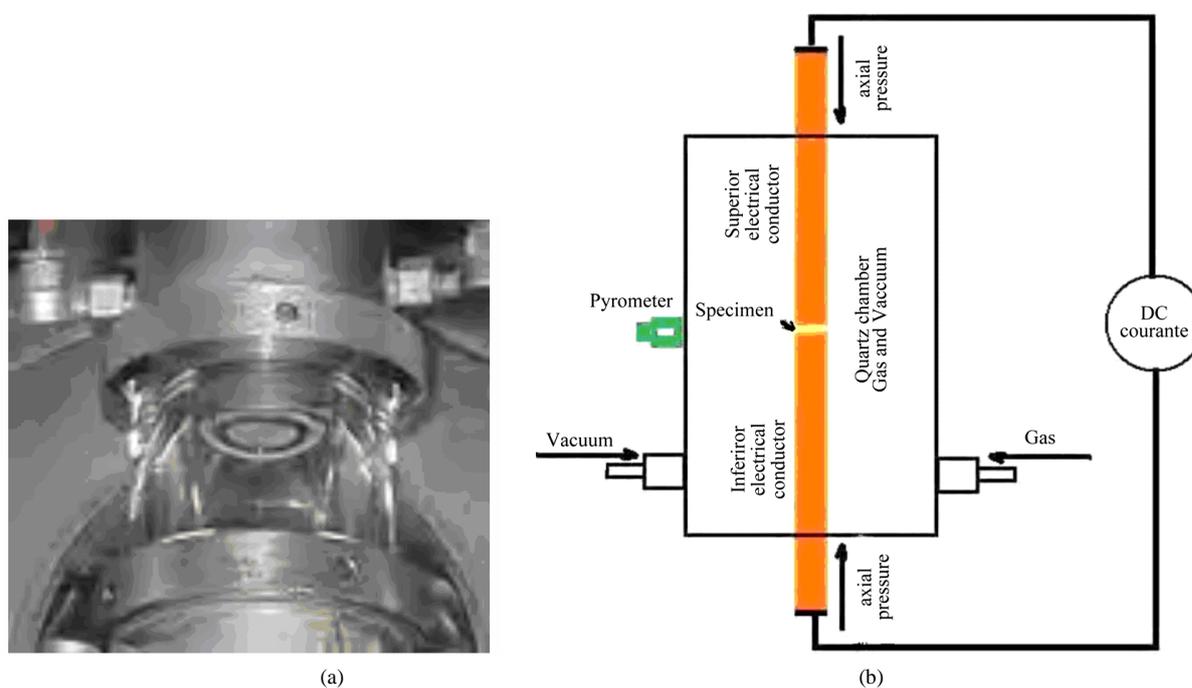


Figure 2. The electro-thermal explosion (ETE) reactor. (a) Chamber electro-thermal explosion reactor; (b) Schematic diagram of the electro-thermal explosion.

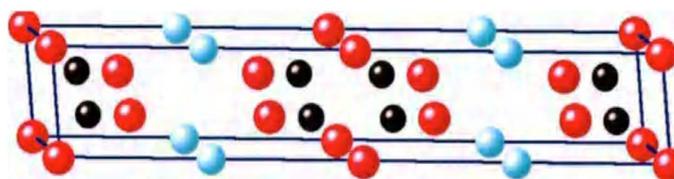


Figure 3. Crystal structure of Ti_3SiC_2 , as refined from FPLO ab-initio calculations. Red, blue, and black spheres represent Ti, Si, and C atoms, respectively.

and X-ray photoelectron spectroscopy (XPS) (Figure 6).

The diffraction pattern of the combustion product formed upon ETE of the starting mechanically alloyed powder of Ti, C, and Si confirms the presence of Ti_3SiC_2 bulk bicarbide base ceramic (TiC, SiC) and possibly

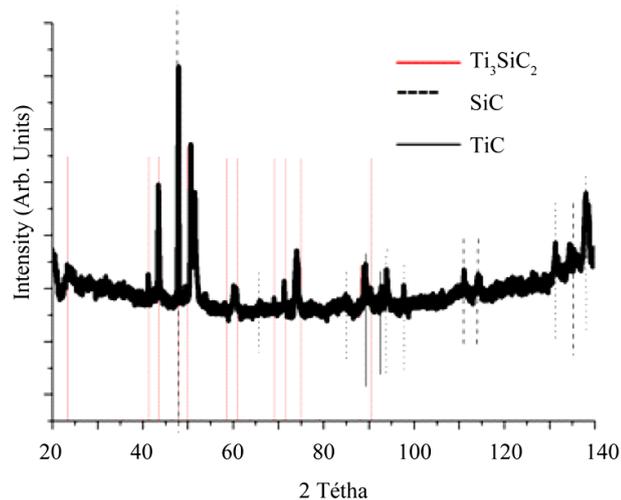


Figure 4. XRD patterns of the ETE from Ti-Si-C system product.

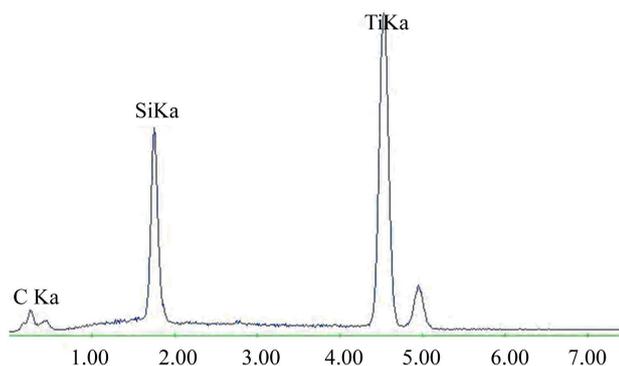


Figure 5. EDX spectra of bulk Ti_3SiC_2 bicarbide based ceramic.

TiSi_2 . The morphology and microstructure of the ETE product are shown in **Figure 1**. The EDX data (**Figure 5**) also confirm the presence of the Ti_3SiC_2 bulk bicarbide base ceramic. The formation of the Ti_3SiC_2 bulk bicarbide is a two-step process: 1) the diffusion of Si into TiC to form the Ti_3SiC_2 bulk bicarbide, and 2) the diffusion of Ti into SiC resulting in Ti_3SiC_2 bulk bicarbide. The remaining amount of TiC and SiC could be subdivided in the matrix based ceramic.

The **Figure 6** shows the XPS data. The persistence of surface oxide is shown from the intensity of the O 1s line. The spectra of the model compounds were representative of those from the specimens that arise from optimized processing routes. Detailed spectra for the relevant specimens are shown in **Figures 6(a)-(c)** for C 1s, Ti 2p and Si 2p/KLL, respectively. There were a number of significant and less intense spectra, but the latter are all well resolved. The C 1s spectra (**Figure 6(a)**) for Ti_3SiC_2 and (**Figure 6(b)**) for TiC revealed major contributions from adventitious graphitic/aromatic components at 284.6 eV, the carbide components were less intense, but well resolved at lower binding energies (BEs). The SiC spectrum, on the other hand, was dominated by a carbide peak at 283.5 eV and a small unresolved graphitic component on the high-BE shoulder of the main peak. The Ti 2p envelopes (**Figure 6(a)**) reflected the carbide contributions, as well as from the TiC surface that has the slightly higher BE. The detailed Si spectra (**Figure 6(c)**) exhibited various features. The SiC specimen resulted in envelopes (Si 2p and Si KLL) which are predominantly due to carbide, but with a small high-BE shoulder on the 2p envelope reflecting a SiOx state. The latter is matched by a resolved component in the KLL envelope at a kinetic energy (KE) of ca. 1608.5 eV. The spectra for Ti_3SiC_2 exhibited two major contributions, one being at a BE of ca. 98.5 eV with a strong Si KLL component at ca. 1617.4 eV. And the second, which is most likely due to SiOx related features, that was found at ca. 102.3 eV with a matching KLL component at ca. 1610.6 eV. The error bars on the experimental results were typically 6, 0.2 eV, with somewhat larger uncertain-

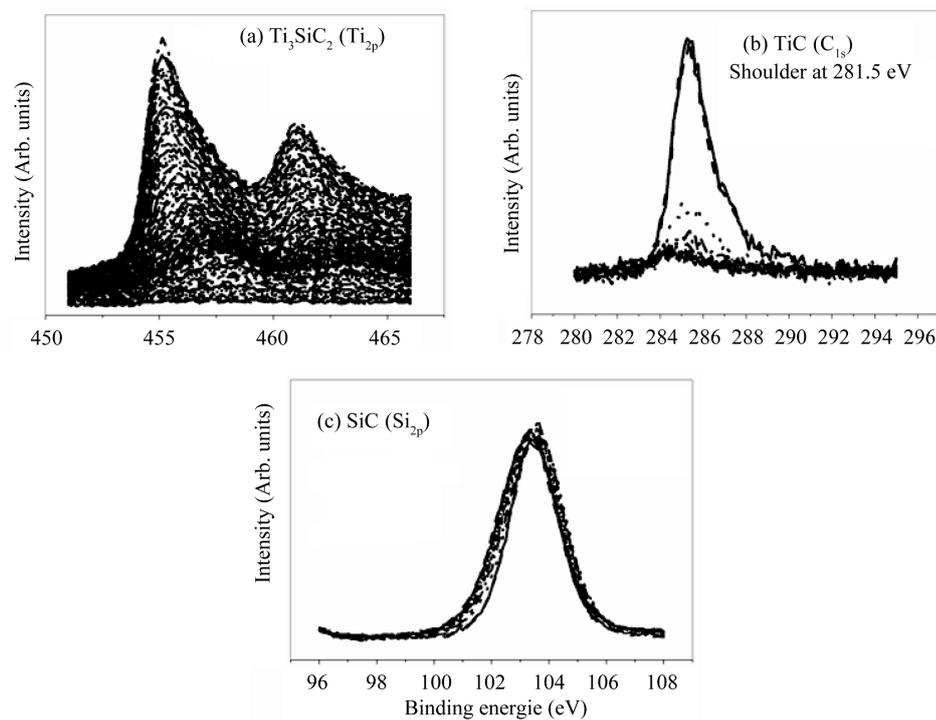


Figure 6. High resolution X-ray photoelectron spectroscopy (XPS) spectra for: (a) Ti 2p, (b) C 1s and (c) Si 2p/Si KLL. The spectra refer to Ti_3SiC_2 , TiC and SiC in order from the top to the bottom.

ties in the data for Si KLL. The present results for SiC and TiC are in excellent agreement with those from other studies. The C 1s low-BE component at 281.06, 0.2 eV for Ti_3SiC_2 is close to the lowest C 1s value ever reported. Similarly, the Ti 2p_{3/2} binding energy obtained from analysis of Ti_3SiC_2 is close to that of metallic elemental Ti, *i.e.* 454.0 eV [6] [32], and is less, by ca. 0.5 eV, than that for TiC. Finally, the Si 2p binding energy for Ti_3SiC_2 is below that of elemental Si, and some 2 eV is below those of the metal silicides [6] [32] and SiC.

The kinetic energy of the KLL transition is above that of elemental Si and is close to those for the metal silicides [6] [32]. The obvious qualitative conclusion is that the three constituent species are all exceptionally well screened (e.g., the Ti species appears to be as well screened as is in the metallic state), hence the low binding energies and the high Si KLL kinetic energy. The conclusion is in agreement with the observation that the ceramic is an excellent electrical conductor.

4. Conclusion

We have successfully produced that the Ti_3SiC_2 bulk bicarbide based ceramic (TiC, SiC) via ETE-method. Further experiment will be focused on the synthesis of the pure Ti_3SiC_2 bulk bicarbide with ETE and SHS reactions combined ETE-Sintering and ball milling-ETE. The aim of these investigations was to contribute to understanding the properties and to synthesis a single phase of these ceramic machinable materials.

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References

- [1] Barsoum, M.W. and El-Raghy, T. (1996) Synthesis and Characterization of a Remarkable Ceramic: Ti_3SiC_2 , *Journal of the American Ceramic Society*, **79**, 1953-1956. <http://dx.doi.org/10.1111/j.1151-2916.1996.tb08018.x>

- [2] El-Raghy, T., Zavaliangos, A., Barsoum, M.W. and Kalidinidi, S. (1997) Damage Mechanisms around Hardness Indentations in Ti_3SiC_2 . *Journal of the American Ceramic Society*, **80**, 1997, 513-520. <http://dx.doi.org/10.1111/j.1151-2916.1997.tb02861.x>
- [3] Barsoum, M.W., El-Raghy, T. and Ogbuji, L. (1997) Oxidation of Ti_3SiC_2 in Air. *Journal of the Electrochemical Society*, **144**, 1997, 2508-2516. <http://dx.doi.org/10.1149/1.1837846>
- [4] Barsoum, M.W. and El-Raghy, T. (1997) Diffusion Kinetics of the Carburization and Silicidation of Ti_3SiC_2 . *Journal of Materials Synthesis and Processing*, **5**, 1997, 197-216.
- [5] El-Raghy, T. and Barsoum, M.W. (1998) Diffusion Kinetics of the Carburization and Silicidation of Ti_3SiC_2 . *Journal of Applied Physics*, **83**, 1998, 112-120. <http://dx.doi.org/10.1063/1.366707>
- [6] Kisi, E.H., Crossley, J.A.A., Myhra, S. and Barsoum, M.W. (1998) Structure and Crystal Chemistry of Ti_3SiC_2 . *Journal of Physics and Chemistry of Solids*, **59**, 1437-1443. [http://dx.doi.org/10.1016/S0022-3697\(98\)00226-1](http://dx.doi.org/10.1016/S0022-3697(98)00226-1)
- [7] Low, I.M., Lee, S.K., Lawn, B. and Barsoum, M.W. (1998) Contact Damage Accumulation in Ti_3SiC_2 . *Journal of the American Ceramic Society*, **81**, 1998, 225-231. <http://dx.doi.org/10.1111/j.1151-2916.1998.tb02320.x>
- [8] Farber, L., Barsoum, M.W., Zavaliangos, A. and Levin, I. (1998) Dislocations and Stacking Faults in Ti_3SiC_2 . *Journal of the American Ceramic Society*, **81**, 1998, 1677-1681. <http://dx.doi.org/10.1111/j.1151-2916.1998.tb02532.x>
- [9] Jeitschko, W. and Nowotny, H. (1967) Die Kristallstruktur von Ti_3SiC_2 ein Neuer Komplexcarbidge Typ. *Monatshefte für Chemie/Chemical Monthly*, **98**, 329-337.
- [10] Panczyk, J., Niemyski, T., Vinogradov, L. and Sinelnikova, V. (2000) Production of Ti_3SiC_2 Material. *Applied Physics Letters*, **76**, 1972-1976.
- [11] Goto, T. and Hirai, T. (1987) chemically-Vapor Deposited Ti_3SiC_2 . *Materials Research Bulletin*, **22**, 1195-1201. [http://dx.doi.org/10.1016/0025-5408\(87\)90128-0](http://dx.doi.org/10.1016/0025-5408(87)90128-0)
- [12] Pampuch, R., Lis, J., Piekarczyk, J. and Stobierski, L. (1993) Ti_3SiC_2 -Based Materials Produced by Self-Propagating High Temperature Synthesis and Ceramic Processing. *Journal of Materials Synthesis and Processing*, **1**, 93-100.
- [13] Onodera, A., Hirano, H., Yuasa, T., Guo, N.F. and Miyamoto, Y. (1999) Static compression of Ti_3SiC_2 to 61 GPa. *Applied Physics Letters*, **74**, 3782-3796. <http://dx.doi.org/10.1063/1.124178>
- [14] Takitani, Y., Matuki, T., Li, J.-F. and Watanabe, R. (2003) Evaluation of Ti_3SiC_2 Prepared by Mechanical Alloying. *Journal of the Japan Society of Powder and Powder Metallurgy*, **50**, 880-884.
- [15] Wills, J.M., Eriksson, O., Wills, J.M. and Cooper, B.R. (1987) Synthesis of Band and Model Hamiltonian Theory for Hybridizing Cerium Systems. *Physical Review B*, **36**, 3809-3823. <http://dx.doi.org/10.1103/PhysRevB.36.3809>
- [16] Andersen, O.K. (1975) Linear Methods in Band Theory. *Physical Review B*, **12**, 3060-3083. <http://dx.doi.org/10.1103/PhysRevB.12.3060>
- [17] Stryker, H.L. (1984) The LMTO Method. Springer, Berlin. <http://dx.doi.org/10.1007/978-3-642-81844-8>
- [18] Chadi, D.J. and Cohen, M.L. (1973) Special Points in the Brillouin Zone. *Physical Review B*, **8**, 5747-5753. <http://dx.doi.org/10.1103/PhysRevB.8.5747>
- [19] Pearson, W.B. (1972) The Crystal Chemistry and Physics of Metals and Alloys. Wiley-Interscience, New York, 502-518.
- [20] Chang, R. and Graham, L.J. (1966) Low-Temperature Elastic Properties of ZrC and TiC. *Journal of Applied Physics*, **37**, 3778-3786. <http://dx.doi.org/10.1063/1.1707923>
- [21] Ahuja, R., Eriksson, O., Wills, J.M. and Johansson, B. (1996) Structural, Elastic, and High-Pressure Properties of Cubic TiC, TiN, and TiO. *Physical Review B*, **53**, 3072-3087. <http://dx.doi.org/10.1103/PhysRevB.53.3072>
- [22] Lis, J., Pampuch, R. and Stobierski, L. (1992) Reactions during SHS in a Ti-Si-C System. *This International Journal Encompasses Self-Propagating High-Temperature Synthesis*, **1**, 401-408.
- [23] Jin, S.Z., Liang, B.Y., Li, J.F. and Ren, L.Q. (2007) Effect of Al Addition on Phase Purity of $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ Synthesized by Mechanical Alloying. *Journal of Materials Processing Technology*, **182**, 445-449. <http://dx.doi.org/10.1016/j.jmatprotec.2006.09.001>
- [24] Zhang, Z.F., Sun, Z.M., Hashimoto, H. and Abe, T. (2003) Fabrication and Microstructure Characterization of Ti_3SiC_2 Synthesized from Ti/Si/2TiC Powders Using the Pulse Discharge Sintering (PDS) Technique. *Journal of the American Ceramic Society*, **86**, 431-436. <http://dx.doi.org/10.1111/j.1151-2916.2003.tb03317.x>
- [25] Liang, B.Y., Wang, M.Z., Sun, J.F., Li, X.P., Zhao, Y.C. and Han, X. (2009) Synthesis of Ti SiC in Air Using Mechanically Activated 3Ti/Si/2C Powder. *Journal of Alloys and Compounds*, **474**, L18-L21. <http://dx.doi.org/10.1016/j.jallcom.2008.06.147>
- [26] Yeh, C.L. and Shen, Y.G. (2008) Effects of SiC Addition on Formation of Ti_3SiC_2 by Self-Propagating High-Temperature Synthesis. *Journal of Alloys and Compounds*, **461**, 654-660. <http://dx.doi.org/10.1016/j.jallcom.2007.07.088>

- [27] Zakeri, M., Rahimipour, M.R. and Khanmohammadian, A. (2008) Effect of the Starting Materials on the Reaction Synthesis of Ti_3SiC_2 . *Ceramics International*, **35**, 1553-1557. <http://dx.doi.org/10.1016/j.ceramint.2008.08.011>
- [28] Liang, B.Y., Jin, S.Z. and Wang, M.Z. (2008) Low-Temperature Fabrication of High Purity Ti_3SiC_2 . *Journal of Alloys and Compounds*, **460**, 440-443. <http://dx.doi.org/10.1016/j.jallcom.2007.05.074>
- [29] Meng, F.L., Chaffron, L. and Zhou, Y.C. (2009) Synthesis of Ti_3SiC_2 by High Energy Ball Milling and Reactive Sintering from Ti, Si, and C Elements. *Journal of Nuclear Materials*, **386-388**, 647-649.
- [30] Abu, M.J., Mohamed, J.J. and Ahmad, Z.A. (2012) Effect of Excess Silicon on the Formation of Ti_3SiC_2 Using Free Ti/Si/C Powders Synthesized via Arc Melting. *International Scholarly Research Network, ISRN Ceramics*, **2012**, Article ID: 341285, 10 Pages.
- [31] El Saeed, M.A., Deorsola, F.A. and Rashad, R.M. (2012) Optimization of the Ti_3SiC_2 MAX Phase Synthesis. *International Journal of Refractory Metals and Hard Materials*, **35**, 127-131. <http://dx.doi.org/10.1016/j.ijrmhm.2012.05.001>
- [32] Briggs, D. and Beamson, G. (1992) Primary and Secondary Oxygen-Induced C1s Binding Energy Shifts in X-Ray Photoelectron Spectroscopy of Polymers. *Analytical Chemistry*, **64**, 1729-1736. <http://dx.doi.org/10.1021/ac00039a018>
- [33] Chakri, N.E., Bendjemil, B. and Baricco, M. (2013) The Mechanical Properties of the System and Training $Zr_{59}Nb_5Cu_{18}Ni_8Al_{10}$ Bulk Metallic Glasses. *Advances in Chemical Engineering and Science*, **3**, 274-277. <http://dx.doi.org/10.4236/aces.2013.34034>