Ti₃SiC₂ MAX Phase Synthesis by Plasma Basis Method

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Abstract The polycrystalline dense Ti₃SiC₂ based ceramic have been prepared by several techniques. The effects on the addition of TiC or SiC are also studied. Ti₃SiC₂ has an unusual combination of electrical, thermal and mechanical extraordinary properties. It has furthermore damage tolerant and oxidation resistance. In this work we have produced Ti₃SiC₂ by electro-thermal explosion chemical reaction started with high current density (680 amperes) followed by uniaxial pressure. The microstructures of the materials were studied using XRD, SEM and EDX. The surface of Ti₃SiC₂ was studied by means of X-ray photoelectron spectroscopy (XPS). High resolution C_{1s}, Si_{2p}, Ti_{2p}, Ti_{3s} core level spectra are inspected in terms of electronic structure. Valence band spectra will be performed to confirm the validity of the theoretical calculations.

Keywords Ti₃SiC₂ Based Ceramic, Intermetallic, Bicarbid, Electro-thermal Explosion Reaction (ETE), Core Level Excitation, XPS

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1. Introduction

The MAX phases constitute a group of ternary ceramics which has received intense attention over the last decade due to their unique combination of properties. The Ti₃SiC₂ is the most well studied MAX phase to date and it has turned out to be a promising candidate for high temperature applications. It is oxidation resistant, refractory and not susceptible to thermal shock. It can be machined with conventional tools, which is of great technological importance.

Most attempts to synthesis bulk Ti₃SiC₂ has been done starting with powders including pure titanium, but Ti powder is oxidising [1] and requires an inert atmosphere throughout the synthesis process which makes the procedures unsuitable for large scale production.

Table 1. An overview of the previous work on Ti₃SiC₂ relevant to this study

Authors	Starting powders	Synthesis Method	Intermediate Phases	Secondary Phases
Pampuch et al.[9]	Ti/Si/C	SHS	Liquid	TiC
Orthner et al.[10]	Ti/Si/C	SHS/MA	TiSi ₂ , Ti ₅ Si ₃ C _x	TiC
Zhou et al.[8]	Ti/Si/C	Hot Pressing	Liquid	TiC
Zhang et al.[7]	Ti/Si/C	PDS	Liquid	TiC, SiC, TiSi ₂
Barsoum et al.[2, 3]	Ti/SiC/C	HP/HIP	TiC _x , Ti ₅ Si ₃ C _x	---
Wu et al.[11, 12]	Ti/SiC/C	Pressureless	TiC _x , Ti ₅ Si ₃ C _x	TiC _x
Istomin et al.[13]	Ti/SiC/C	Vacuum	TiC _x , TiSi ₂ , Ti ₅ Si ₃ C _x	TiC, TiSi ₂ , Ti ₅ Si ₃
Riley et al.[14]	Ti/SiC/C	SHS	Solid solution	TiC _x
Sun et al.[4]	Ti/Si/TiC	Vacuum	TiC, Ti ₅ Si ₃ C _x	---
Li & Miyamoto[15]	Ti/Si/TiC	Pressureless	Liquid	---
Gao et al.[5]	Ti/Si/TiC	HIP	Ti ₅ Si ₃ C _x	TiC _x
Yang et al.[16]	Ti/Si/TiC	Vacuum	Ti ₅ Si ₃ , TiSi ₂	---
Radhakrishnan et al. [17, 18]	TiC/Si	Pressureless/HP	TiSi ₂	SiC, TiC
Li et al. [19]	TiC/Si	Pressureless	Ti ₅ Si ₃	SiC, TiC
Hwang et al.[20]	TiC _{0.67} /Si	Pressureless	---	TiC _x
Córdoba et al.[21]	TiC/Si	Pressureless	---	SiC, TiC

The previous work on the Ti₃SiC₂ is summarised in Table 1. There is consensus in the literature with some minor variations on the route of formation of Ti₃SiC₂ from starting powders containing pure Ti. Barsoum et al. [2, 3] suggested a series of reactions from Ti/SiC/C starting powders based on the intermediate phases TiC_x and Ti₅Si₃C_x. Their results were essentially confirmed by Wu et al. and Istomin et al. The latter added the intermediate phase TiSi₂. Sun et al. [4] and Gao et al. [5] used Ti/Si/TiC starting powders but found the same intermediate phases. Gao et al. also reported on the effect of the partial pressure of Si in the furnace atmosphere on the thermochemical stability of Ti₃SiC₂. They observed that the MAX phase tend to decompose, as initially proposed by Racault et al., [6] into TiC and gaseous Si. The reactions are often observed to take place in a silicon rich liquid [7-9] from which the Ti₃SiC₂ crystals grow. The liquid is sometimes associated with one or both of the two eutectic reactions of the Ti-Si phase diagram; Ti-Ti₅Si₃ and Si-TiSi₂.

Recent interest in the phase is due to the availability of improved material from a hot-pressing route, leading to high purity polycrystalline single-phase samples of near-theoretical density. The structure has been reported [23] as 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, its strength to temperatures that makes the best superalloys available today unusable. 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 is self-lubricating so it will be better than graphite for rotating electrical contacts for ac motors. It is a promising candidate for ceramic engines. As far as the experiments of this compound are concerned. Jeitschko and Nowotny [22] have synthesized Ti₃SiC₂ by a chemical reaction between TiH₂, Si, and graphite at 2000 °C. Goto and Hirai have synthesized Ti₃SiC₂ by a chemical-vapor deposition technique. There is very little known about this material. Panczyk *et al* [23], have shown that the melting point of Ti₃SiC₂ is around 3000 °C. Pampuch *et al* [24], have produced by self-propagating high-temperature synthesis and ceramic processing Ti₃SiC₂-based material. Their measured value of the Young's modulus is 326 GPa and higher hardness [23]. The study of the electronic structure and the chemical bonding get a better insight into this important material. In order to study the electronic structure of Ti₃SiC₂, Full-Potential Linear-Muffin-Tin-Orbital method (FPLMTO) is used [25]. The calculations were based on the Local-Density Approximation (LDA) and have been used the Hedin-Lundqvist 10 parametrization for the exchange and correlation potential. Basis functions, electron densities, and potentials were calculated without any geometrical approximation [26]. The radial basis functions within the muffin-tin spheres are linear combinations of radial wave functions and their energy derivatives, computed at energies appropriate to their site and principal as well as orbital atomic quantum numbers, whereas outside the muffin-tin

spheres the basis functions are combinations of Neuman or Hankel functions [27, 28]. For sampling the irreducible wedge of the Brillouin zone, the special *k*-point method is used [29].

Ti₃SiC₂ belongs to the D_{6h}4-P63/*mmc* space group of hexagonal crystalline structure [30]. Its primitive cell contains two formula units. The observed lattice parameters are *a*=53.064Å and *c*=517.650Å. *C/a* is optimized by calculating of the total energy for different *c/a* values at the equilibrium volume. The calculated value of *c/a* is around 5.77, which is in very good agreement with the experiment value of 5.76. The calculated value of the bulk modulus is 225 GPa and is in good agreement with the experimental value [28]. If one compares it with TiC, which has a bulk modulus of 240 GPa [31], they are comparable; the density of states (DOS) for Ti₃SiC₂ and the corresponding band structure is showed. The lowest-lying states around 10 eV are originated from the nonmetal C_{2s} states. If one compares this with the DOS of TiC [32], there are also C_{2s} states, which lay 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 is lies exactly in the pseudogap, whereas in Ti₃SiC₂ it has moved outside of this gap. This indicates that TiC should be harder than Ti₃SiC₂.

The modern electronic structure theory is sufficiently developed to give an accurate description of Ti₃SiC₂. The calculated volume and structural parameter are in very good agreement with the experiment. The chemical bonding appears to be similar to that for TiC.

A polycrystalline material based on a Ti₃SiC₂ compound produced by sintering of powders prepared using electro-thermal explosion (ETE) has been shown to have the advantages of ceramic materials combined with some metal-type plastic properties.

The reaction in ternary Ti-Si-C system under the SHS regime may produce either TiC, SiC, TiSi₂, Ti₅Si₃C or Ti₃SiC₂; each in a quantity depending on the starting mixture composition [33], however, until now attempts to produce a Ti₃SiC₂ single phase resulted at the very most in 10 % TiC, 20% of SiC and amount of titanium-silicon intermetallic as ascertained, the effect of the addition (TiC, SiC) are also studied [34, 35].

In this work, we have produced Ti₃SiC₂ with chemical reaction by electro-thermal explosion with high current density (680 A°) followed by uniaxial pressure.

The present study investigated the microstructures of materials using XRD, FESEM equipped by EDX analysis. The Ti₃SiC₂ 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 electronic structure. Valence band spectra will be performed to confirm the validity of the theoretical calculations. The aim of these studies is synthesizing a highly pure Ti_3SiC_2 ceramic machinable material with better thermal, physical and mechanical properties.

2. Experimental

Polycrystalline high-purity Ti, C, and Si powders (99.99% pure) of the nominal composition Ti_3SiC_2 were mechanically alloyed. The powder was prepared from Good fellow 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 reaction in vacuum and Argon that launched at 680°A during 360 seconds followed by uniaxial pressure. The combustion is started at the measured temperature by an infrared pyrometer at about 1800°C .

The reactions considered for the three different systems were the following:

The structural properties were determined by XRD using a Philips diffractometer (Co-K α radiation). Further structural characterizations were carried out by energy-dispersive X-ray microanalysis (EDX), field emission scanning electron microscopy (FESEM), and optical microscopy (OM). FESEM micrographs were taken with a JEOL-JSM-840A instrument equipped with an EDX accessory to check the elemental composition of the system. XPS analysis were carried out in an ESCALAB Mk. II instrument using Al K_{α} 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).

Figure 1. Crystal structure of Ti_3SiC_2 , obtained from Full Potential Local Orbital Band Structure Scheme (FPLO) ab-initio calculations code. Red, blue, and black spheres represent Ti, Si, and C atoms, respectively.

3. Results and Discussion

Ti_3SiC_2 (Fig.2) bulk bicarbide base ceramic (TiC, SiC) produced by electro-thermal explosion (ETE, Fig.1) of crystalline elemental powder of Ti, C, and Si after balls milling, 13/1 (balls/mass) has been synthesized. The phase composition after ETE reaction has been studied using the X-ray diffraction technique (Fig.3) The diffraction pattern of the combustion product formed upon ETE reaction in the starting mechanically alloyed powder Ti, C, and Si is seen to contain Ti_3SiC_2 bulk bicarbide base ceramic (TiC, SiC) and maybe TiSi_2 . The microanalysis EDX data (Fig. 4) also confirm the XRD data in the presence of the Ti_3SiC_2 bulk bicarbide base ceramic. The morphology by Field Emission Scanning Electron Microscopy (FESEM) and Optical Microscopy (OM) is reported in the Fig.5. The observation have proved that the elongation slabs with rounded corners of well fused Ti_3SiC_2 grains form a matrix within some rounded TiC and less frequent angular SiC inclusions are present in the Fig.5.

Figure 2. a. Welding resistance machine, b-Scheme of current distribution

Figure 3. XRD patterns of the ETE from Ti-Si-C system product.

Figure 4. EDX spectra of bulk Ti_3SiC_2 bicarbide based ceramic.

Figure 5. FESEM (c, d) and OM (a, b) imagings corresponding to bulk Ti_3SiC_2 ceramic bicarbides (TiC, SiC) and maybe intermetallic phase TiSi_2 .

The formation of Ti_3SiC_2 bulk bicarbide is a two-step process. The first one involves the diffusion of Si into TiC to form Ti_3SiC_2 bulk bicarbide, the second step is the diffusion of Ti into SiC resulting Ti_3SiC_2 bulk bicarbide. The remaining TiC and SiC is reported in the matrix based ceramic [33].

Further studies have been carried out by X-ray photoelectron spectroscopy (XPS) The Figure 6 shows the

XPS data. The persistence of surface oxide is apparent from the intensity of the O_{1s} line. The spectra of the model compounds were representative of those from specimens arising from optimized processing routes. Detailed spectra are shown in Fig. 6(a), (b) and (c) for C_{1s}, Ti_{2p} and Si_{2p}/KLL, respectively. The C_{1s} spectra (Fig. 6(a)) for Ti₃SiC₂ and (Fig. 6(b)) for TiC revealed major contributions from adventitious graphitic/aromatic components at 284.6 eV and less intense, but well resolved, carbide components 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 (Fig. 6(a)) reflected mainly carbide contributions, with that from the TiC surface having the slightly higher BE. The detailed Si spectra (Fig. 6(c)) exhibited various features. The SiC specimen resulted in envelopes (Si_{2p} and Si_{KLL}) predominantly due to carbide, but with a small high-BE shoulder on the 2p envelope reflecting a SiO_x state, which is matched by a small, well will to be resolved component in the KLL envelope at a kinetic energy (KE) of ca. 1608.5 eV. The spectra for Ti₃SiC₂ 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. The slightly more intense contribution, most likely due to SiO_x related features, 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 uncertainties in the data for Si KLL.

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 are referred to Ti₃SiC₂, TiC and SiC in order from the top to the bottom.

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 Ti₃SiC₂ is close to the lowest C_{1s} value ever reported. Similarly, the Ti_{2p_{3/2}} binding energy obtained from analysis of Ti₃SiC₂ is close to that of metallic elemental Ti, i.e. 454.0 eV [35], and is less, by ca. 0.5 eV, than that for TiC finally; the Si_{2p} binding energy for Ti₃SiC₂ is below that of elemental Si, and some 2 eV below those of the metal silicides [35] and SiC. The kinetic energy of the KLL transition is above that of elemental Si and close to those for the metal silicides [35]. 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 it is in the metallic state), hence the low binding energies and high Si_{KLL} kinetic energy. The conclusion is in accord with the observation that the ceramic is an excellent electrical conductor.

4. Conclusions

We have produced successfully via ETE reaction the Ti₃SiC₂ bulk bicarbide based ceramic (TiC, SiC). Further experiment will be focused in the synthesis of the highly pure Ti₃SiC₂ bulk bicarbide with Spark Plasma Sintering (SPS) and then reinforced by single, multi-walled carbon nanotubes and nanocarbon fibers to obtain better properties for specified application

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